Designation: E 854 – 98

Standard Test Method for Application and Analysis of Solid State Track Recorder (SSTR) Monitors for Reactor Surveillance, E706(IIIB)[1]

This standard is issued under the fixed designation E 854; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes the use of solid-state track recorders (SSTRs) for neutron dosimetry in light-water reactor (LWR) applications. These applications extend from low neutron fluence to high neutron fluence, including high power pressure vessel surveillance and test reactor irradiations as well as low power benchmark field measurement. **(1)** This test method replaces Method E 418. This test method is more detailed and special attention is given to the use of state-of-the-art manual and automated track counting methods to attain high absolute accuracies. In-situ dosimetry in actual high fluence-high temperature LWR applications is emphasized.

1.2 This test method includes SSTR analysis by both manual and automated methods. To attain a desired accuracy, the track scanning method selected places limits on the allowable track density. Typically good results are obtained in the range of 5 to 800 000 tracks/cm^2 and accurate results at higher track densities have been demonstrated for some cases. **(2)** Track density and other factors place limits on the applicability of the SSTR method at high fluences. Special care must be exerted when measuring neutron fluences (E>1MeV) above 10^{16} n/cm^2. **(3)**

1.3 High fluence limitations exist. These limitations are discussed in detail in Section 13 and in references **(3-5)**.

1.4 SSTR observations provide time-integrated reaction rates. Therefore, SSTR are truly passive-fluence detectors. They provide permanent records of dosimetry experiments without the need for time-dependent corrections, such as decay factors that arise with radiometric monitors.

1.5 Since SSTR provide a spatial record of the time-integrated reaction rate at a microscopic level, they can be used for “fine-structure” measurements. For example, spatial distributions of isotopic fission rates can be obtained at very high resolution with SSTR.

1.6 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 418 Method for Fast-Neutron Measurements by Track-Etch Techniques[2]

E 844 Guide for Sensor Set Design and Irradiation for Reactor Surveillance, E706 (IIC)[3]

3. Summary of Test Method

3.1 SSTR are usually placed in firm surface contact with a fissionable nuclide that has been deposited on a pure nonfissionable metal substrate (backing). This typical SSTR geometry is depicted in Fig. 1. Neutron-induced fission produces latent fission-fragment tracks in the SSTR. These tracks may be developed by chemical etching to a size that is observable with an optical microscope. Microphotographs of etched fission tracks in mica, quartz glass, and natural quartz crystals can be seen in Fig. 2.

3.1.1 While the conventional SSTR geometry depicted in Fig. 1 is not mandatory, it does possess distinct advantages for dosimetry applications. In particular, it provides the highest efficiency and sensitivity while maintaining a fixed and easily reproducible geometry.

3.1.2 The track density (that is, the number of tracks per unit area) is proportional to the fission density (that is, the number of fissions per unit area). The fission density is, in turn, proportional to the exposure fluence experienced by the SSTR. The existence of nonuniformity in the fission deposit or the presence of neutron flux gradients can produce non-uniform track density. Conversely, with fission deposits of proven uniformity, gradients of the neutron field can be investigated with very high spatial resolution.

3.2 The total uncertainty of SSTR fission rates is comprised of two independent sources. These two error components arise from track counting uncertainties and fission-deposit mass uncertainties. For work at the highest accuracy levels, fission-deposit mass assay should be performed both before and after the SSTR irradiation. In this way, it can be ascertained that no significant removal of fission deposit material arose in the

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[1] This test method is under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applicationsand is the direct responsibility of Subcommittee E10.05on Nuclear Radiation Metrology.

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[2] Discontinued; see 1983 *Annual Book of ASTM Standards*, Vol 12.02.

[3] *Annual Book of ASTM Standards*, Vol 12.02.

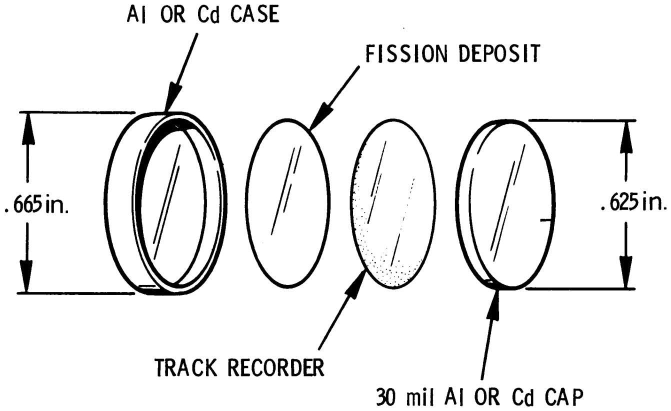


FIG. 1 Typical Geometrical Configuration Used for SSTR Neutron Dosimetry

course of the experiment.

4. Significance and Use

4.1 The SSTR method provides for the measurement of absolute-fission density per unit mass. Absolute-neutron fluence can then be inferred from these SSTR-based absolute fission rate observations if an appropriate neutron spectrum average fission cross section is known. This method is highly discriminatory against other components of the in-core radiation field. Gamma rays, beta rays, and other lightly ionizing particles do not produce observable tracks in appropriate LWR SSTR candidate materials. However, photofission can contribute to the observed fission track density and should therefore be accounted for when nonnegligible. For a more detailed discussion of photofission effects, see 13.4.

4.2 In this test method, SSTR are placed in surface contact with fissionable deposits and record neutron-induced fission fragments. By variation of the surface mass density (µg/cm^2) of the fissionable deposit as well as employing the allowable range of track densities (from roughly 1 event/cm^2 up to 10^5 events/cm^2 for manual scanning), a range of total fluence sensitivity covering at least 16 orders of magnitude is possible, from roughly 10^2 n/cm ^{2} up to 5×10^{18} n/cm^2. The allowable range of fission track densities is broader than the track density range for high accuracy manual scanning work with optical microscopy cited in 1.2. In particular, automated and semi-automated methods exist that broaden the customary track density range available with manual optical microscopy. In this broader track density region, effects of reduced counting statistics at very low track densities and track pile-up corrections at very high track densities can present inherent limitations for work of high accuracy. Automated scanning techniques are described in Section 11.

4.3 For dosimetry applications, different energy regions of the neutron spectrum can be selectively emphasized by changing the nuclide used for the fission deposit.

4.4 It is possible to use SSTR directly for neutron dosimetry as described in 4.1 or to obtain a composite neutron detection efficiency by exposure in a benchmark neutron field. The fluence and spectrum-averaged cross section in this benchmark field must be known. Furthermore, application in other neutron fields may require adjustments due to spectral deviation from the benchmark field spectrum used for calibration. In any event, it must be stressed that the SSTR-fission density measurements can be carried out completely independent of any cross-section standards **(6).** Therefore, for certain applications, the independent nature of this test method should not be compromised. On the other hand, many practical applications exist wherein this factor is of no consequence so that benchmark field calibration would be entirely appropriate.

5. Apparatus

5.1 *Optical Microscopes*, with a magnification of 200 × or higher, employing a graduated mechanical stage with position readout to the nearest 1 µm and similar repositioning accuracy. A calibrated stage micrometer and eyepiece scanning grids are also required.

5.2 *Constant-Temperature Baths*, for etching, with temperature control to 0.1°C.

5.3 *Analytical Weighing Balance*, for preparation of etching bath solutions, with a capacity of at least 1000 g and an accuracy of at least 1 mg.

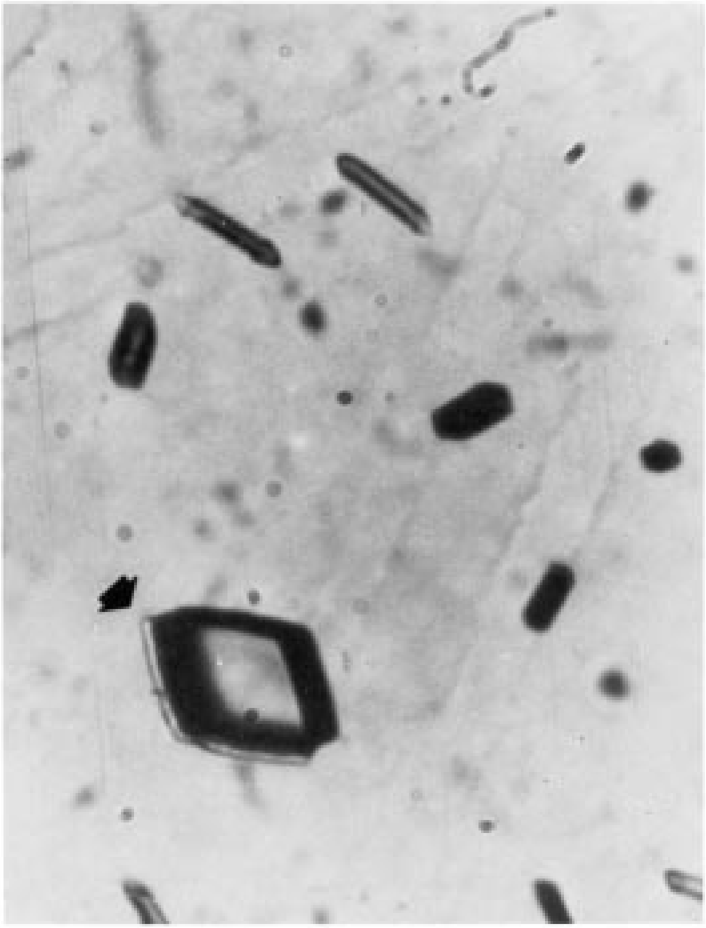
6. Reagents and Materials

6.1 *Purity of Reagents*—Distilled or demineralized water and analytical grade reagents should be used at all times. For high fluence measurements, quartz-distilled water and ultrapure reagents are necessary in order to reduce background fission tracks from natural uranium and thorium impurities. This is particularly important if any pre-irradiation etching is performed (see 8.2).

6.2 *Reagents*:
6.2.1 *Hydrofluoric Acid (HF)*, weight 49 %.
6.2.2 *Sodium Hydroxide Solution (NaOH)*, 6.2 N.
6.2.3 *Distilled or Demineralized Water*.
6.2.4 *Potassium Hydroxide Solution (KOH)*, 6.2 N.
6.2.5 *Sodium Hydroxide Solution (NaOH)*, weight 65 %.
6.3 *Materials*:
6.3.1 *Glass Microscope Slides*.
6.3.2 *Slide Cover Glasses*.

7. SSTR Materials for Reactor Applications

7.1 *Required Properties*—SSTR materials for reactor applications should be transparent dielectrics with a relatively high ionization threshold, so as to discriminate against lightly ionizing particles. The materials that meet these prerequisites most closely are the minerals mica, quartz glass, and quartz crystals. Selected characteristics for these SSTR are summarized in Table 1. Other minerals such as apatite, sphene, and zircon are also suitable, but are not used due to inferior etching properties compared to mica and quartz. These alternative SSTR candidates often possess either higher imperfection density or poorer contrast and clarity for scanning by optical microscopy. Mica and particularly quartz can be found with the additional advantageous property of low natural uranium and thorium content. These heavy elements are undesirable in neutron-dosimetry work, since such impurities lead to background track densities when SSTR are exposed to high neutron fluence. In the case of older mineral samples, a background of fossil fission track arises due mainly to the spontaneous fission decay of ^{238}U. Glasses (and particularly phosphate glasses) are



NOTE 1—The track designated by the arrow in the mica SSTR is a fossil fission track that has been enlarged by suitable preirradiation etching.

FIG. 2 Microphotograph of Fission Fragment Tracks in Mica

less suitable than mica and quartz due to higher uranium and thorium content. Also, the track-etching characteristics of many glasses are inferior, in that these glasses possess higher bulk etch rate and lower registration efficiency. Other SSTR materials, such as Lexan[4] and Makrofol[5] are also used, but are less convenient in many reactor applications due to the presence of neutron-induced recoil tracks from elements such as carbon and oxygen present in the SSTR. These detectors are also more sensitive (in the form of increased bulk etch rate) to the β and γ components of the reactor radiation field **(13).** Also, they are more sensitive to high temperatures, since the onset of track annealing occurs at a much lower temperature for plastic SSTR materials.

7.2 *Limitations of SSTR in LWR Environments*:

7.2.1 *Thermal Annealing*—High temperatures result in the erasure of tracks due to thermal annealing. Natural quartz crystal is least affected by high temperatures, followed by mica. Lexan and Makrofol are subject to annealing at much lower temperatures. An example of the use of natural quartz crystal SSTRs for high-temperature neutron dosimetry measurements is the work described in reference **(14)**.

7.2.2 *Radiation Damage*—Lexan and Makrofol are highly sensitive to other components of the radiation field. As mentioned in 7.1, the bulk-etch rates of plastic SSTR are increased by exposure to β and γ radiation. Quartz has been observed to have a higher bulk etch rate after irradiation with a fluence of 4×10^{21} neutrons/cm^2, but both quartz and mica are very insensitive to radiation damage at lower fluences ($<10^{21}$ neutrons/cm^2).

7.2.3 *Background Tracks*—Plastic track detectors will register recoil carbon and oxygen ions resulting from neutron scattering on carbon and oxygen atoms in the plastic. These fast neutron-induced recoils can produce a background of short tracks. Quartz and mica will not register such light ions and are not subject to such background tracks.

8. SSTR Pre- and Post-Irradiation Processing

8.1 *Pre-Irradiation Annealing*:

8.1.1 In the case of mica SSTR, a pre-annealing procedure designed to remove fossil track damage is advisable for work at low neutron fluences. The standard procedure is annealing

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[4] Lexan is a registered trademark of the General Electric Co., Pittsfield, MA.

[5] Makrofol is a registered trademark of Farbenfabriken Bayer AG, U. S. representative Naftone, Inc., New York, NY.

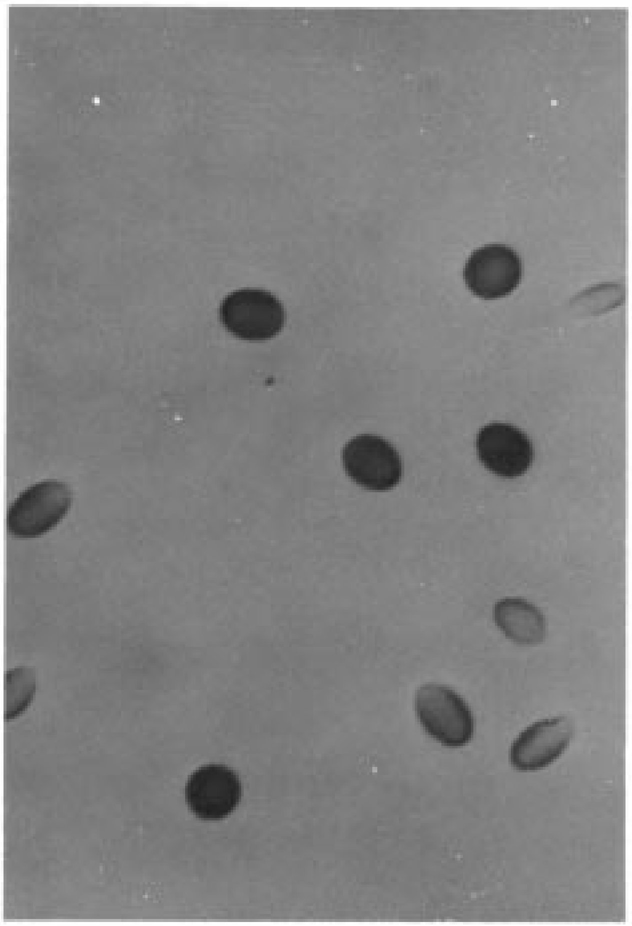

FIG. 2 Quartz Glass *(continued)*

FIG. 2 Quartz Crystal (001 Plane) *(continued)*

for 6 h at 600°C (longer time periods may result in dehydration). Fossil track densities are so low in good Brazilian quartz crystals that pre-annealing is not generally necessary. Annealing is not advised for plastic SSTR because of the possibility of thermal degradation of the polymer or altered composition, both of which could effect track registration properties of the plastic.

8.2 *Pre-Irradiation Etching*:

8.2.1 *Mica*—Unannealed fossil tracks in mica are easily distinguished from induced tracks by pre-etching for a time that is long compared to the post-etching conditions. In the case of mica, a 6-h etch in 48 % HF at room temperature results in large diamond-shaped tracks that are easily distinguished from the much smaller induced tracks revealed by a 90-min post-etch (see Fig. 2)).

8.2.2 *Quartz Crystals*—Pre-etching is needed to chemically polish the surface. Polish a crystal mechanically on the 001 or 100 plane so that it appears smooth under microscopical examination, etch for 10 min in 49 % HF at room temperature, then boil in 65 % NaOH solution for 25 min. Examine the crystal surface microscopically. If it is sufficiently free of pits, select it for use as an SSTR.

8.2.3 *Quartz Glass*—If the glass has been polished mechanically, or has a smooth surface, then pre-etch in 49 % HF for 5 min at room temperature. Upon microscopical examination a few etch pits may be present even in good-quality quartz glass. If so, they will be larger than tracks due to fission fragments revealed in the post-etch, and readily distinguished from them.

8.2.4 *Plastic-Track Recorders*—If handled properly, background from natural sources, such as radon, will be negligible. Consequently, both preannealing and pre-etching should be unnecessary.

8.3 *Post-Irradiation Etching*:

8.3.1 *Mica*—Customary etching is for 90 min in 49 % HF at room temperature. Both the etch time and temperature may be varied to give optimum track sizes for the particular type of mica used. Except for work at the highest accuracy levels, precise control of the temperature is not necessary due to the zero bulk etch rate of the mica perpendicular to the cleavage planes. In the event that precise etching control is necessary, a technique has been demonstrated for mica that permits highly reproducible and standardized track size distributions **(10).**

8.3.2 *Quartz Crystals*—Etch for 25 min in boiling 65 % NaOH solution. Minimize evaporation by covering the nickel or platinum crucible in which the solution is heated. If left open, condense evaporated water and return to the solution. The value of the optical efficiency is dependent on the etching conditions (since the bulk etch rate is not zero), so both the concentration of the NaOH solution and the etching temperature must be controlled.

TABLE 1 Characteristics of SSTR Candidates for LWR Reactor Applications

SSTR	Optical Efficiency, %	Asymptotic[A] Sensitivity	Conditions Under Which Accurate Annealing Corrections Can Be Made	Track Reduction, %
Muscovite mica	0.9875 ± 0.0085[B]	(1.114 ± 0.018) × 10^{19} ^{238}U atoms/cm^{2}[B]	501°C, 146.5 h[C]	0[C]
Makrofol N	95.2 ± 0.53[D]	...	...	...
Quartz glass	~70[E]	...	402°C, 8 h[C]	73[C]
Natural quartz Crystal	~80[E]	...	857°C, 1 h[F]	20[F]

[A] Needs to be known only if used with asymptotically thick sources.
[B] Etched 90 min in 49 % HF **(6, 7, 8).**
[C] Data from Ref **(9).**
[D] Etched ~20 h in 6.2 *N* KOH solution at room temperature **(6)**.
[E] Quartz glass etched 5 min in 48 % HF at room temperature. Quartz crystal etched in boiling 65 % NaOH solution for 25 min **(10, 11)**.
[F] Data from Ref **(12)**.

8.3.3 *Quartz Glass*—Etch for 5 min in 48 % HF at room temperature. Temperature control is essential because of the high bulk etch rate.

8.3.4 *Lexan,*[4] *or Makrofol*[5], *N*—Various time temperature combinations in 6.2 *N* NaOH or KOH solution have proved satisfactory, depending upon the desired purpose. Examples of appropriate conditions are: (*1*) 50 h in 6.2 *N* NaOH solution at 20°C, (*2*) 24 h in 6.2 *N* KOH solution at 20°C, and (*3*) 30 min in 6.2 *N* KOH solution at 50°C.

9. SSTR Fissionable Deposits

9.1 *Properties*:

9.1.1 *Fission Deposit Characteristics*— Perhaps the most critical factor in attaining high accuracy in SSTR neutron dosimetry is the quality of the fission deposit. High quality SSTR fission deposits possess the following characteristics: **(6-17)**

9.1.1.1 Accurately known total mass and mass density. The overall accuracy of the mass calibration must be consistent with the desired overall accuracy of the measurement.

9.1.1.2 Accurately known isotopic composition. Possible interfering isotopes must be minimized and the overall fission rate must be corrected for contributions from interfering isotopes.

9.1.1.3 *Negligible Impurities*—Impurities that contribute to the measured fission rate must be minimized (<1 % contribution) and the overall fission rate must be corrected for contributions from impurities.

9.1.1.4 High uniformity is recommended. An independent measurement is required which verifies the uniformity of the deposit to an uncertainty commensurate with the desired accuracy of subsequent measurements using the deposit. Conversely, use of nonuniform deposits entails scanning of the entire SSTR surface to attain accurate results.

9.1.2 As has already been stated in 3.2, the accuracy of fission deposit characterization provides a fundamental limitation for the accuracy of the SSTR method. Fission-deposit mass assay as well as uniformity are important. Dosimetry goal accuracies provide bounds for the acceptable quality of SSTR fission deposits. For work at the highest accuracy levels, fission deposits can be prepared at close to or better than 1 % mass assay. Less accurate SSTR dosimetry can, however, be performed at a lower cost with less stringent requirements for fission deposit characterization. The deposit backing should contribute negligible background and the deposit should be flat, rigid, and capable of maintaining good contact with the SSTR. The deposit should be firmly adherent to the backing. The appropriate mass density for a particular LWR application may be calculated from:

$$\phi t \times W = \frac{\rho M}{\eta N_o \bar{\sigma} I} \tag{1}$$

where:

ϕt = the expected fluence,
W = the mass density of the deposit, g/cm^{2},
ρ = the track density (the optimum track density for most manual scanning is about 5 × 10^{4} tracks/cm^{2}),
I = the isotopic abundance (atomic fraction),
η = the optical efficiency of the SSTR,
σ = the spectral average fission cross section,
M = the average atomic weight of the isotopic mixture used, and
N_o = Avogadro's number (6.022 × 10^{23}).

9.1.3 In Eq 1, the assumption is made that the thickness (mass density) of the deposit is much less than the range of a fission fragment in the deposit material. Under these conditions, self-absorption is negligible and sensitivity depends linearly on *W*. For deposit thicknesses greater than about 100 μ/cm^{2}, self-absorption of fission fragments by the deposit becomes increasingly important. For deposit thicknesses greater than twice the range of a fission fragment in the deposit material, the effective thickness may be represented by a constant value. This constant value is referred to as the asymptotic sensitivity, s_∞. It can be analytically shown **(6)** for a uniform deposit with no flux depression that the asymptotic sensitivity is approximately given by:

$$s_\infty \simeq \eta \frac{(R)}{2} \tag{2}$$

where:

$\langle R \rangle$ = the mean fission fragment range in the deposit.

In the case of uranium metal, an asymptotic sensitivity of 4.522 ± 0.070 mg/cm^{2} has been measured **(6,8).** Thicknesses in the approximate range from 0.1 to 30 mg/cm^{2}should be avoided due to problems arising from self-absorption of fission fragments in the source. While it is possible to work in this range, additional error will be incurred due to the need to correct for self-absorption. In the region beyond 30 mg/cm^{2}, one should use the asymptotic sensitivity.

9.2 *Isotypes Required*—In general, when performing reaction rate measurements for a particular isotope, contributions to the fission rate from other isotopes must be either negligible or corrected with sufficient accuracy. For example, use of the threshold reaction ^{238}U (n,f) in a neutron field where the thermal flux is appreciable requires highly depleted uranium in order to minimize contributions from ^{235}U (n,f). Similarly chemical purity must be taken into account. When measuring the reaction rate for an even-even nuclide such as ^{240}Pu, the abundance of the fissionable even-odd isotopic neighbors ^{239}Pu and ^{241}Pu must be minimized. For low-flux measurements, contributions from spontaneously fissioning nuclides must be minimized and if necessary spontaneous fission track contributions must be subtracted.

9.3 *Source Preparation*:

9.3.1 Electrodeposition and vacuum deposition are the most frequently used and the most effective techniques. The latter method normally results in more uniform deposits, but economy of material and convenience may favor the former. In both cases, actinide deposits are produced more easily in the oxide than in the metallic form. Adherence of the deposit to the backing material can often be accomplished by heating the deposit to red heat in an inert atmosphere. Uniformity can be demonstrated by α-autoradiography using an α-sensitive SSTR such as cellulose nitrate or by fission track radiography with uniform neutron field irradiations.

9.3.2 Metallic backing for the fission deposit should be chosen to meet a number of requirements. For dosimetry purposes the backing should only be thick enough to ensure firm contact between the track recorder and the deposit (see Fig. 1). Furthermore, since it is preferable that no foreign elements be introduced into the radiation environment, backing materials should be chosen wherever possible from constituent elements that already exist in the radiation environment. Neutron field perturbations due to the backing are considered in Section 12. For high-fluence measurements, extremely pure-backing materials are required in order to reduce background fission tracks from natural uranium and thorium impurities. The surface of the backing material must be smooth and preferably possess a mirror finish.

9.4 *Mass Assay*:

9.4.1 *Absolute Disintegration Rate*—Mass assay may be accomplished by absolute α-counting using a low geometry α-counter **(6).** In many cases, the alpha decay constant is known to an accuracy of better than 1 %. In fact, the uncertainty of the alpha decay constant provides a fundamental limitation in this mass-assay method. Relative masses of several sources of the same isotope may be established to better than 1 % by α-counting in a 2π proportional counter. (See Table 2 for a summary of alpha decay constants of the actinide elements **(15).)**

9.4.2 *Mass Spectrometry*—Mass spectrometry combined with isotopic dilution techniques is a potentially useful method for mass assay of deposits. Mass spectrometry is particularly useful for low specific activity isotopes or isotopes with decay constants that have not been measured to an accuracy of 1 %. While mass spectrometry can provide accuracies of better than 1 %, it suffers from an inherent disadvantage, namely the need for destructive analysis.

9.4.3 *Isotopic Spikes*—High specific activity isotopes may be used as a tracer to indicate target mass. Alpha active isotopes such as ^{230}Th, ^{236}Pu, and ^{238}Pu as well as γ-emitting isotopes such as ^{237}U and ^{239}Np are useful for relative mass determinations. When using isotopic spikes, care must be taken to ensure that the source isotope and the spike are chemically equivalent. Also, the fission rate of the isotopic spike and its daughter products should be kept negligible compared to the fission rate of the isotope of interest. The use of isotopic spikes that feed complex decay chains (such as ^{228}Th and ^{232}U) should be avoided.

9.4.4 *Less Frequently Used Methods*—Ion, X-ray, and Auger microprobe analysis, X-ray fluorescence, neutron activation analysis, and wet chemical analysis methods may be useful for specific applications, but rarely attain an accuracy comparable to previously mentioned methods.

9.5 *Ultra Low-mass Deposits*—Methods for producing and calibrating ultra low-mass fissionable deposits are described in reference **(3)**. Because of the low masses involved, typically 10^{-14} to 10^{-9} grams, care must be taken to avoid contamination of the deposits. Therefore, the deposits must be made under

TABLE 2 Decay Constants and Associated Uncertainties Used in Actinide Mass Quantification

Nuclide	$t_{1/2}$(years)	$\lambda(s^{-1})$	Uncertainty, %	Reference **(15)**		
				Vol	No.	Date
^{230}Th	$(7.538 \pm 0.030) \times 10^{4}$	2.914×10^{-13}	0.40	40	3	1983
^{232}Th	$(1.405 \pm 0.006) \times 10^{10}$	1.563×10^{-18}	0.43	36	3	1982
^{233}U	$(1.592 \pm 0.020) \times 10^{5}$	1.380×10^{-13}	1.26	24	2	1978
^{234}U	$(2.45 \pm 0.02) \times 10^{5}$	8.965×10^{-14}	1.38	40	4	1983
^{235}U	$(7.038 \pm 0.005) \times 10^{8}$	3.121×10^{-17}	0.071	40	1	1983
^{237}U[A]	6.75 ± 0.01 days	1.189×10^{-6}	0.15	23	1	1978
^{238}U	$(4.468 \pm 0.003) \times 10^{9}$	4.916×10^{-18}	0.067	38	2	1983
^{237}Np	$(2.14 \pm 0.01) \times 10^{6}$	1.026×10^{-14}	0.47	23	1	1978
^{239}Np[A]	2.355 ± 0.004 days	3.407×10^{-6}	0.17	40	1	1983
^{236}Pu[A]	2.851 ± 0.008	7.704×10^{-9}	0.28	36	3	1982
^{238}Pu	$(8.774 \pm 0.004) \times 10^{1}$	2.503×10^{-10}	0.046	38	2	1983
^{239}Pu	$(2.4119 \pm 0.026) \times 10^{4}$	9.107×10^{-13}	0.11	40	1	1983
^{240}Pu	$(6.569 \pm 0.006) \times 10^{3}$	3.344×10^{-12}	0.091	43	2	1984
^{241}Pu[B]	$(1.4355 \pm 0.0007) \times 10^{1}$	1.530×10^{-9}	0.049	44	2	1984
^{242}Pu	$(3.733 \pm 0.012) \times 10^{5}$	5.884×10^{-14}	0.32	45	3	1985

[A] Tracer materials used for quantification of low mass primary deposits (may be α or β/γ emitters, or both).

[B] The branching ratio for alpha emission is $(2.46 \pm 0.01) \times 10^{-3}$ %. The partial half-life for alpha decay is 5.79×10^{5} years (±3.2 %).

clean conditions using high-purity materials and chemical reagents.

9.5.1 *Mass Calibration*—Isotopic spiking methods (see 9.4.3) are used, and often the limitation on the amount of spike isotope that can be added is the extent of the contribution of either impurity isotopes or daughter isotopes to the overall fission rate of the deposit. For the case when short-lived ^{239}Np is used as a tracer for ^{237}Np, the eventual decay of the spike to ^{239}Pu must be considered as it will contribute to the overall fission rate of the deposit. Therefore, the ^{239}Np/^{237}Np ratio must be kept small enough to ensure that the resultant ^{239}Pu/^{237}Np fission rate ratio in the measured neutron spectrum will be small (typically less than 0.5 %). After the fission rate measurements are performed, the spike contribution to the fission rate must be confirmed to be small by calculating the fission rate due to the known amount of ^{239}Pu from the spike using the measured fission rate from a ^{239}Pu deposit exposed in the same dosimetry location.

9.5.2 *Ultra Low-Mass Deposit Calibration Uncertainties*—Additional uncertainties exist in the calibration of ultra low-mass deposits because of the additional steps necessary in the overall calibration. When isotopic spiking methods are used to determine the relative mass scale for a set of fissionable deposits, the uncertainty in the measurement of the relative radioactivity must be taken into account. For example, when short-lived ^{237}U is used as a tracer for either ^{235}U or ^{238}U, all of the uncertainties inherent in the measurements of the relative ^{237}U gamma decay rates must be taken into account. Among these uncertainties are the precision of the source to detector geometry and the Poisson statistics of the number of gamma ray counts recorded for each deposit. In order to determine an absolute mass scale, a measurement of gamma decay rate to absolute mass must be performed. Often this measurement corresponds to a relative gamma decay rate to absolute alpha decay rate measurement for a sample where both rates can be measured with sufficient accuracy. When an alpha emitting spike is used, such as ^{236}Pu to measure relative 239Pu masses, only the relative alpha peak intensities need be measured. However, the uncertainties in the alpha decay constants (half lives) of both the spike isotope and the fissionable deposit isotope contribute to the overall uncertainty. For short-lived spikes such as ^{237}U (6.75 d) or ^{239}Np (2.34 d), decay corrections must be made. An alternative method **(3** which eliminates the uncertainties contributed by the decay corrections is to use multiple detectors which are operated in parallel. Relative gamma decay rates for ^{237}U can be determined with a set of ten thin-window proportional counters setting aside one counter for a standard which is also a fissionable deposit. In each set of ten counts, the decay rate of nine deposits is measured relative to the standard which is following the same radioactive half life. However, corrections must be made for small efficiency differences in a set of ten "identical" detectors as well as for detector cross-link and detector background, and the uncertainties in these corrections all contribute to the overall uncertainty. A useful strategy in ultra low-mass deposit calibration is to ensure that the additional uncertainties added by the addition of the spiking step are kept smaller than 0.5 % by the design of the spiking procedures.

9.5.3 *Independent Mass Calibration Verification*—Because of the added complexities of the production and calibration of the ultra low-mass deposits used in reactor cavity neutron dosimetry **(2-5)**, deposits made for this application have been subjected to independent mass calibration accuracy verification through irradiations in standard reference neutron fields at NIST and elsewhere **(16)**. Typically, one deposit from each ultra low-mass electroplating series is subjected to a benchmark irradiation, although, in some cases, multiple deposits from a series have been irradiated. These irradiations and NIST comparisons are consistent with the expected uncertainty of 2 % for the spike measurement mass scales and show that the absolute mass scales are consistent to 5 %. Because ultra low-mass deposits are made by electroplating methods, uniformity is more difficult to control than for vacuum-evaporated or sputtered deposits, but the uncertainty contribution of this non-uniformity is less than 2 %. The overall uniformity does contribute to the fluence limit that can be obtained as discussed subsequently in Section 11.4.2.1.

10. Manual Track-Scanning Procedures

10.1 *Equipment and Calibration*:

10.1.1 For manual scanning, a good research quality binocular microscope is required, having a stage equipped with two dials or micrometers that make it possible to estimate the *x* and *y* position of the stage to the nearest micrometer. One eyepiece should contain a square grid (one with 36 squares has been found to be highly satisfactory). The grid should cover a large fraction of the field of view. Take care to adjust the microscopes so that good Kohler illumination and adequate image contrast is obtained. This is especially true when asymptotically thick deposits are used (since many of the tracks are short and posses lower optical contrasts).

10.1.2 Calibrate the width of the grid for each lens combination with a stage micrometer and estimate to the nearest 1 µm. The linearity and accuracy of the dials or micrometers must also be checked and calibrated with the stage micrometer.

10.1.3 It is important that the instructions in the microscope manual be studied and followed to optimize contrast and resolution. If transmitted bright field illumination is used (highly satisfactory for mica and Makrofol N[5] or Lexan[4]), contrast and resolution may be improved by using oblique instead of axial illumination, if available. Especially good contrast is obtained in quartz glass when reflected light is used.

10.2 *Manual Track Counting Procedure*:

10.2.1 Two situations need to be considered: (*1*) When it is essential to count all of the fission tracks in the SSTR, which can arise when the fission deposit is not sufficiently uniform for the desired accuracy, and (*2*) when only a fraction of the tracks need be counted to obtain the desired statistical accuracy.

10.2.2 For case (*1*), the scanner should find one edge of the region containing tracks and systematically cover the total area. A proven method **(6)** is to align the grid carefully so that the vertical lines are parallel to the *y* motion—a track or surface blemish should move on a grid line as the stage is moved along the *y*-axis. Do not count tracks touching or crossing the left and top grid lines, count those touching or crossing the right and bottom grid lines. When all the tracks in a given field are counted from left to right and from top to bottom as in reading, a track or blemish crossing or touching the top line is moved in the *y* direction until it is in the corresponding position on the bottom line. After the tracks moved into the field are counted as before, repeat the process until all the tracks in the given *y* swath have been counted. If tracks on the right edge of the region containing the tracks have been counted, move a track or surface blemish on the left line to the corresponding position on the right grid line, and count all of the tracks in a new *y* swath. Repeat this procedure over the entire area containing tracks; count all tracks. If track densities are sufficiently small, tracks may be counted as they cross a horizontal grid line as the SSTR is moved continuously in the *y* direction, instead of counting tracks field by field.

10.2.3 In case (*2*), the procedure is the same, except that a region removed from the edges of the track distribution is selected for counting. The area scanned is determined by observing the initial and final readings of the calibrated dial for the *y*-axis, and multiplying the difference by the width of the grid as measured by a stage micrometer. This may be repeated for more scanning swaths which need not be adjacent. This case offers the advantage to the scanner of selecting the best counting region if surface blemishes mar certain regions of the SSTR.

10.2.4 Count tracks with a tally counter; the scanner should be free to work the fine focus control while tracks are being counted so that tracks will be kept in sharp focus.

10.2.5 When scanners are first trained, they should *not* be told what to count. Rather, they should be asked to examine regions of the SSTR that do not contain tracks, so that they teach themselves to distinguish surface blemishes from fission tracks. In this way, careful scanners generally converge quickly to good agreement. If difficulties persist, different scanners may be asked to count tracks in the same field in order to remove small discrepancies. By using this procedure, observer biases are generally minimized and objectivity is established.

10.2.6 It is important that the SSTR surface be clean when scanned. Accomplish this by putting a cover glass over the surface of a clean SSTR ready for counting. If this is not feasible the SSTR should be cleaned, if necessary, before the tracks are counted.

11. Automated Track Counting

11.1 *Introduction*:

11.1.1 A major inconvenience of detection methods using tracks is the necessity for manual, visual measurement of tracks, a task that requires care, patience, and dedication. This drawback is especially significant for precision measurements, where inherent statistical limitations require the observation of large numbers of tracks, making the task time consuming and expensive. As a consequence, worldwide expertise in precision applications of SSTR methods is quite limited. A more detailed discussion of these requirements can be found in a critical review of the SSTR method **(17).**

11.1.2 Elimination of the human element is highly desirable for precise track measurements, since it allows the observation of larger numbers of tracks and permits the introduction of more quantitative standards of track identification and background subtraction. Such standards would obviate problems of personal bias in manual track measurements, which can otherwise compromise experimental accuracy. In order to attain high accuracy, such biases must constantly be guarded against in manual track scanning. Therefore, a considerable interest has existed, and continues to exist, in the automation of this scanning task. A perhaps tacit, but certainly reasonable assumption is that any such automated system must provide at least comparable accuracy to manual scanning techniques. Only under such a condition can the high accuracy goals of current SSTR applications be maintained.

11.2 *Background*:

11.2.1 Since the late 1960s, considerable effort has been expended by many groups in attempts to automate track scanning. Spark scanning methods have been developed **(18-21)** but have not been widely used due to limitations in accuracy (10-20 %) and track density (less than $10^3/\text{cm}^3$). More sophisticated systems employed an optical microscope under computer control **(22-30)** The availability of inexpensive minicomputers and microprocessors has afforded considerable progress in automated scanning capability **(31-33)**. Of equal significance has been the development high-quality video camera image analysis systems. In addition to scarcely compromising microscopic resolution and contrast, modern CCD camera systems provide fast digital signals that afford dramatic improvements for automated pattern recognition. In view of this rapid evolution, it is best to consult the most recent literature for details on these highly-specialized techniques.

11.3 *Automated Track Counting System*:

11.3.1 *Equipment and Calibration*:

11.3.1.1 A good research-quality microscope is required, equipped with a motor-driven stage that can be controlled by a computer and can be repositioned with an accuracy of ±1 μm.

11.3.1.2 A computer input corresponding to the visual image from the microscope must be obtained. One method is to view the microscope image with a video camera and digitize the video image for input to a suitable image analysis computer.

11.3.1.3 A computer with sufficient speed and capacity to carry out the necessary steps for identification and correlation of track data is required.

11.3.2 *Automated Track Counting Procedure*:

11.3.2.1 A consistent and verifiable procedure (software or hardware, or both) must be developed for the identification and counting of tracks. This procedure may include gray level discrimination, image-enhancement, pattern recognition or other procedures that aid in track identification, or combination thereof.

11.3.2.2 Following optimization of the automated track counter parameters, counting of a series of track standards is required to verify the operation of the scanner within the desired accuracy. Whenever the scanning parameters are changed, recalibration with standards using the new parameters is required.

11.3.2.3 It is important that the SSTR surface be clean when scanned. Accomplish this by putting a cover glass over the surface of a clean SSTR ready for counting. For automated scanning, the quality of the SSTR can be particularly important. Care should be taken to ensure that the SSTR surface is as free as possible of cracks, scratches, dust, or other sources of visual interference.

11.4 *High Precision Applications*:

11.4.1 *Low and Medium Track Density Analysis*—Analysis of SSTR with low track densities can be done by counting tracks taking each contiguous area as one track. Corrections for pile-up are small and may be made by a variety of methods. It is also necessary to correct for background arising from imperfections in the track recorder, which the automated system may identify as tracks. Other methods normally applied for high track densities can also be used for low track densities, if the background can be handled accurately.

11.4.2 *High Track Density Analysis*:

11.4.2.1 At extremely high track densities, overlap of tracks can become so great that individual tracks can no longer be distinguished. An analysis of track density uncertainty as a function of track density appears in reference 34. The uncertainty attained in track density measurements will likely be a different function of track density for different automated scanning systems. In recent efforts **(34)**, track density uncertainties less than 2 % were found to be generally unattainable for track densities greater than 8×10^5 tracks/cm^2. The high track density limit will also depend on the degree of uniformity of the fissionable deposits, and the highest track densities will be possible with the most uniform deposits where problems associated with local regions of high track pileup will be avoided. However, in most applications it is impractical to perform detailed uniformity measurements to high accuracy on each deposit to be used. For track densities lower than 8×10^5 tracks/cm^2, 2 % uncertainties were shown to be generally attainable using fissionable deposits made with ultra law-mass electroplating techniques **(4,34)** and having uniformities typical of deposits made with these methods. It has been shown that this track pile-up limitation is allayed by using the Buffon Needle Method **(31)** of track scanning which may provide a method to obtain acceptable results at higher track densities. The Buffon Needle method is, in turn, particularly well suited for automated scanning systems. More recently, it has been demonstrated that the random sampling procedure of the Buffon Needle method can be replaced by sampling on a fixed network or grid of points on the SSTR surface **(32, 33).**

11.4.2.2 In these efforts, the probability distribution for fixed grid sampling has been rigorously derived and this result has been proven through comparison with experiment down to the level of approximately 1 % (1σ). Moreover, fixed grid sampling provides significantly more alleviation from pile-up effects than even the Buffon Needle method. Using such techniques, automation promises to render practical many key experiments for power reactor environments that were heretofore not feasible.

11.4.2.3 Track counting methods used for low track densities can also be extended to the higher track regime. This involves using pattern recognition and statistical analysis to decode patterns of touching and overlapping tracks and to correct for overlapping tracks that are not observed. Empirical approaches can be used to establish system calibrations. Another method that may be applied to minimize pile-up is to underdevelop the tracks.

11.4.2.4 Each of the above methods has limitations that increase the uncertainty. It is therefore important for each laboratory to rigorously assess the accuracy of the method chosen to analyze automated track data.

11.5 *Automated System Calibration*:

11.5.1 Precision automated analysis of SSTR requires detailed calibration of the system to ensure accurate results over the range of track recorders analyzed. Calibration methods include:

11.5.1.1 Comparison with manual scanning results,

11.5.1.2 Analysis of standards, and

11.5.1.3 Comparison between automated methods.

11.5.2 In addition to initial calibration of the system, the experimenter must be aware of the various parameters affecting the result (including, for example, track size, light level, SSTR quality, background, and track density uniformity). A program for periodic analysis of standards is therefore necessary to preclude system changes. In addition, each batch of track recorders should be checked to ensure that no unexpected differences are affecting the results.

12. Neutron Field Perturbations

12.1 Introduction of a passive dosimetry monitor into a radiation environment creates a perturbation in the radiation field of interest. Neutron perturbations that are introduced by SSTR monitors are entirely similar to those created by passive radiometric monitors. The analysis that is used to generate correction factors for radiation field perturbations due to radiometric monitors is also applicable for SSTR monitors. Of the number of treatments of such correction factors, Guide E 844 is perhaps the most relevant.

12.2 Self-shielding effects of passive monitors can be characterized by the product $\Sigma_a \times \mu$, where Σ_a^{-1} is the absorption mean free path for neutrons and μ is the monitor thickness. Only when $\Sigma_a \times \mu << 1$ will self-shielding be negligible. However, this is a general rule that must be utilized with care and depends intimately upon the desired accuracy goals of the specific dosimetry application. For example, if very high accuracy goals, for example, close to 1 %, were desired, a value of $\Sigma_a \times \mu \approx 0.01$ would satisfy this general rule but would still not be negligible. For this case, a systematic perturbation exists of the order of the desired accuracy goal, so that correction of this effect becomes mandatory.

12.3 Clearly $\Sigma_a \times \mu$ should be kept as small as possible (within the other experimental constraints) for passive monitors. SSTR monitors generally possess much higher sensitivity than radiometric monitors, so that $\Sigma_a \times \mu$ is usually much smaller for SSTR. As a consequence, radiation field perturbations created by SSTR are generally much smaller than those created by radiometric monitors.

12.4 Table 3 presents values of Σ_a and μ for representative SSTR monitors in a thermal neutron field. The fission deposit has been chosen as ^{235}U with a thickness of 100 µg/cm^2, is the upper bound beyond which self-absorption of fission fragments is no longer negligible (see 9.1). It can be seen in Table 3 that both the deposit and track recorder values of $\Sigma_a \times \mu$ are negligible in comparison with the backing values, with the

TABLE 3 Representative SSTR Perturbation Parameters for Thermal Neutrons

Component	µ, cm	Σ_a, cm^{-1}	$\Sigma_a \mu$
Fission deposit (^{235}U − 100 µg/cm^2)	5.3×10^{-6}	31	1.6×10^{-4}
Track recorder:			
Mica	1.3×10^{-2}	0.015	2.0×10^{-4}
Quartz crystal	5.0×10^{-2}	0.0043	2.0×10^{-4}
Backing materials:			
Al	2.5×10^{-2}	0.015	3.8×10^{-4}
Stainless steel	2.5×10^{-2}	0.24	6.0×10^{-3}
Pt	2.5×10^{-2}	0.58	1.5×10^{-2}
Au	2.5×10^{-2}	5.8	1.5×10^{-1}

exception of aluminum. Clearly, care must be exercised in the choice of the backing material. Not only are the heavy element Pt and Au backings undesirable from a neutron field perturbation standpoint, but they also produce considerably more backscattering of both alpha particles and fission fragments than do backings of lower atomic number such as stainless steel or aluminum. Moreover, stainless steel and aluminum are often already present in reactor environments, whereas gold and platinum are rarely, if ever, used in reactors.

12.5 For many asymptotically thick SSTR applications, neutron field perturbations will not be negligible. In fact, perturbations of a few percent were observed in the very first experiments used to determine the asymptotic sensitivity **(6).** Correction factors for such perturbations due to asymptotically thick SSTR deposits should be generated from the same analysis that is used for radiometric monitors.

13. High Fluence Limitations

13.1 Beyond the limitations that can be introduced by deposits, high power irradiations can create limitations by directly affecting the properties of the track recorder material. At very high fluence the lattice of the track recorder medium can become disordered and track registration properties can thereby be altered. Thermal annealing of tracks can also occur in high power environments. These high temperature-fluence limits for mica and natural quartz crystal SSTR have not as yet been rigorously established.

13.2 *SSTR Effects*:

13.2.1 Four limitations of SSTR applicability have already been mentioned in 7.2 and 9.1.1, namely (*1*) fission deposit characteristics, (*2*) annealing, (*3*) radiation damage, and (*4*) background fission track production. It has also been noted that the existence of track pile-up produces accuracy limitations at higher track densities. Each of these effects can play a significant role in defining the high fluence limit of applicability of a given SSTR. It must be stressed that these different effects can act in consort rather than independently to produce a high fluence limit. For example, radiation damage of the crystal lattice produced by a high fluence of fast neutrons in a given SSTR can alter SSTR annealing characteristics dramatically. Hence, track fading due to annealing can be considerably enhanced due to the damage of the SSTR crystal lattice that is produced at high fluence.

13.2.2 Other important examples of high fluence limitations can arise when fission deposits are not of high quality. In particular, fission deposits that are highly nonuniform can produce nonuniform track density that adversely affects accuracy, especially at the high track densities generated in high fluence applications. Under such conditions, localized regions of extremely high track density can be produced on the SSTR surface where it is not possible to quantify the track density. Consequently, care must be taken in the design of measurements to ensure that local track densities produced by the irradiation do not exceed track scanning capabilities. Furthermore, the mass of the fissionable deposit must be sufficiently large to produce a number of tracks that is large compared to the number produced by fission of trace impurities in the deposit backing and the SSTR. At a fluence of 10^{20} n/cm^2, the purest available SSTR and deposit backing materials (~0.3 ppb natural uranium) result in a track density of 10^4 to 10^5 tracks/cm^2 (see section 13.5). In order for meaningful measurements to be carried out at such high fluences, deposit masses large enough to produce at least 10^6 to 10^7 tracks/cm^2 must be used. This necessitates the development of higher track density scanning methods as well as the extension of quality ultra low mass deposit fabrication methods to even lower masses. Until such methods are developed and tested, it is better when feasible to reduce the exposure time in high flux situations, so that deposits with higher mass can be used. A method has been devised to produce distributed track densities **(34)** which could be useful in reactor surveillance dosimetry application.

13.3 *Burn-In Effect*—In high fluence applications, ingrowth of certain actinides through neutron capture in the fission deposit can create a non-negligible contribution to the observed fission track density. This so-called "burn-in" effect is not limited to SSTR neutron fission dosimeters, but can also exist in radiometric (RM) fission monitors. A possible significant burn-in effect for ^{238}U fission neutron monitors has already been recognized through the in-growth of ^{239}Pu. In fact, corrections of up to approximately 30 % have been calculated for the burn-in effect in ^{238}U fission neutron monitors in selected LWR environments **(36).** Experimental methods that correct for burn-in effect have recently been described **(37).**

13.4 *Gamma-Ray Effects*:

13.4.1 Direct effects of gamma radiation on the mica component of the SSTR are completely negligible. It has been shown that gamma-ray exposures in excess of 10^9 *R* have no subsequent effect on either the recording or etching properties of mica. A background from the gamma-ray component of the reactor radiation field can be produced by photofission. For broad-based monitors that possess high neutron-induced fission rates, such as ^{235}U or ^{239}Pu, photofission is negligible. However, for threshold monitors, such as ^{232}Th, ^{238}U, or ^{237}Np, photofission may not be negligible compared with other sources of experimental error.

13.4.2 Estimating the photofission contribution for these threshold monitors requires a knowledge of the gamma-ray spectral intensity, especially above roughly 5 MeV where the photofission cross section first becomes significant. Unfortunately, efforts to define the gamma-ray component of the mixed radiation field in reactors have seriously lagged in contrast to the vigorous activity that has been applied to define the neutron component of the mixed radiation field. It is also well-known that neutrons are attenuated more rapidly than gamma-rays in the large water gaps that exist in LWR-PV environments. Consequently, the photofission background component that would arise for threshold monitors in LWR-PV environments may be non-negligible. For example, it has been calculated **(38)** that at the ¼-T location, which is approximately two in. from the inner PV surface, that the relative photofission background component is roughly as follows:

^{238}U 2 to 4 %
^{237}Np |Lq 1 %
^{232}Th 5 to 8 %

13.5 *Cleaning Requirements*—For high fluence applications, all SSTR components must be scrupulously clean. This includes not only the mica or quartz track recorder, but the fission deposit backing as well. All chemicals and components, whether used in the fabrication of the fission deposits or in the cleaning procedures, must be of the highest purity to guard against the inadvertent introduction of naturally occurring actinides, that is, uranium and thorium, which could compromise the deposit. At the level of extremely low masses required in high fluence applications, naturally occurring actinides are apparently ubiquitous and therefore represent a fundamental background limitation, whether introduced through the fission deposit or the track recorder.

14. Calculation

14.1 Eq 1 may be used to calculate the neutron fluence (ϕt) corresponding to a given track density, if W, I, η, and M are known, and if sufficient knowledge of the neutron spectrum is available to determine σ. If the fission rates from different fissionable isotopes are to be used to obtain spectral information, the data can be analyzed by using available unfolding codes **(39).**

15. Precision and Bias [6]

15.1 *Sources of Experimental Error*:

15.1.1 Uncertainties in reaction rate measurements with SSTR fall into four general categories: (*A*) mass and uniformity of the deposit, (*B*) time and position of irradiation exposure, (*C*) track etching, and (*D*) track scanning. Table 4 gives a summary of the limiting uncertainties that exist for these different categories. These categories do not include statistical uncertainty that arises in track scanning. It has been demonstrated **(6)** that Poisson statistics are applicable for manual track scanning.

15.1.1.1 *Mass Assay*—Mass assay has been discussed in 9.4. In most cases the mass and isotopic composition of sources can be determined to better than 1 %. For most deposits that are vacuum-evaporated, the uniformity is also within the 1 % uncertainty. For electroplated deposits the lack of uniformity is somewhat higher; when good precision is required it is often necessary to count all tracks over the entire area of the deposit.

15.1.1.2 *Exposure*—If an exposure is made by placing the SSTR in direct contact with a radioactive deposit, care must be taken to have good physical contact between the deposit and the SSTR. If this is done, errors from this cause should be negligible. Uncertainties in exposure time can be made negligibly small if the total exposure time is made large relative to a few seconds. Greater uncertainties concerning exposures to neutrons in a reactor arise if it is necessary to bring the reactor up to a desired power level and then shut it down after the appropriate exposure time. This problem results from the fact that tracks are being recorded during the total neutron exposure. Uncertainties of this type decrease as the exposure time required to obtain desired track density increases.

TABLE 4 Sources of Experimental Error

Source	% Limiting Accuracy (1σ)
A. Mass Assay	≤1 %
B. Type and Position of Irradiation	
(*1*) Exposure to spontaneous fission sources	<0.1 %
(*2*) Exposure to neutron fields	0.1 % or greater, depending upon conditions
C. Chemical Etching	0.1 % for mica; 1.0 % for quartz crystals
D. Manual Scanning	
(*1*) Area measurements	<0.1 %
(*2*) Stage movements	<0.1 %
(*3*) Track identification (isotropic incidence)	≤0.5 % for pre-selected mica and quartz crystal surfaces

15.1.1.3 *Etching*—Care also needs to be taken to control the chemical composition and the temperature of the etching bath. Use constant-temperature baths providing control to at least ± 0.1°C. The concentration of the etchant should be known and kept constant during the etching procedure. Care should be exercised to verify that bubbles are not present and that the surfaces to be etched are properly wet by the etchant to ensure complete and proper etching of all of the tracks. Errors from etching uncertainties can be kept negligible if proper care is taken. (See further instructions in 12.2.)

15.1.1.4 *Scanning*—Scanning uncertainties are more complicated to measure because of the human element in track counting. Since the human element enters into how a scanner perceives tracks in relation to background artifacts, it is difficult to quantify this problem except by experience. To obtain objectivity in manual scanning, a pool of at least three scanners should be available in each laboratory. Practice shows that for isotropic fission tracks in mica from thin sources, experienced scanners generally agree at the ≤ 0.5 % level. Preliminary results indicate that similar reproducibility is practical with quartz crystals.

15.1.2 In addition to the perception problem, the precision level in track scanning actually depends upon a number of factors that can produce uncertainty in the area scanned. These other factors are: (*1*) measurement of the area of the grid used to define the counting area, (*2*) nonlinearity in the dials used to measure the length of the scanning swaths, (*3*) uncertainty in the movement of the microscope stage from swath-to-swath, and (*4*) decision of the scanner as to whether a track is touching a grid line defining the edge of the field. The length of the grid defining the counting area can be measured at about the 0.1 % level with a suitable stage micrometer. Each person must make

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[6] Measurement uncertainty is described in terms of precision and bias in this standard. Another acceptable approach is to use Type A and B uncertainty components **(40, 41)**. This Type A/B uncertainty specification is now used in International Organization for Standardization (ISO) standards, and this approach can be expected to play a more prominent role in future uncertainty analyses.

his or her own calibration, however, since it depends upon the interocular spacing used on the microscope for the type of microscopes commonly in use. Determine non-linearities in the dial gages by comparison with the stage micrometer and these can be corrected for if significant.

15.1.3 Scanning errors can be reduced if the SSTR is preselected for surface quality. By proper selection, uncertainties can be kept below the 0.5 % level. In the case of mica SSTRs it is important to choose a surface that is smooth and free of cleavage plane discontinuities, defects, and scratches. Care should be taken to ensure that the mica is not bent, cracked, or mishandled before, during, or after the exposure to neutrons. In the case of quartz crystals, it is important that the surface have a good mechanical polish that is maintained prior to and after the exposure if a high level of precision is to be maintained.

15.2 *Optical Efficiency*:

15.2.1 If a SSTR is placed in direct contact with a fission deposit thin relative to the range of the fission fragments (<100 µg/cm ^{2}), the "optical efficiency," η, for the given SSTR is the ratio of tracks counted over a given area to fission events in that same area. In order to determine η, a thin spontaneous fission source such as ^{244}Cm or ^{252}Cf or ^{244}Pu (for which the absolute fission rate has been measured in a low geometry counter) is placed in good contact with the SSTR for a measured length of time. After suitable etching, the fission tracks are then counted. The accuracy of η thus measured, apart from counting statistics, depends upon the following:

15.2.1.1 The accuracy for the absolute fission rate of the source, which depends upon the uncertainties in the alpha half-life, the alpha to fission branching ratio, and the solid angle for the low geometry counter. The statistical accuracy of the counting is also a factor. For ^{244}Cm or ^{252}Cf or ^{244}Pu sources, all of these factors together can be less than 0.5 % at the (1σ) level.

15.2.1.2 For manual counting, the reproducibility of the track count must be demonstrated by at least two and preferably more observers. For mica SSTR this reproducibility can be as good as 0.5 % (1σ), apart from counting statistics. Further study is needed to establish the comparable accuracy for quartz crystals, but preliminary results indicate it is at least as good as mica.

15.2.1.3 The reproducibility in the bulk etch rate of a given SSTR. For Muscovite mica, the bulk etch rate perpendicular to the cleavage planes is small enough to be neglected. In other directions, the rate can be controlled by adjusting temperature and the etching time is used to standardize the track size. For Muscovite mica, this is accomplished by keeping the length *l* of the larger diagonal of normally incident fission fragments within acceptable limits. If *l* is about 6 µm, the change in η/µm is <0.5 %. Since *l* grows linearly with etching time at a given temperature, this parameter can be accurately controlled.

15.2.2 In the case of plastics and quartz, the bulk etch rate perpendicular to the surface is not zero, and η depends critically upon the ratio of the track etch rate to the bulk etch rate. In plastics, both the track etch rates and the bulk etch rates depend upon the "batch" and its environmental history. Therefore, when high precision is required, η must be measured for the given SSTR "batch" material at the time it is to be used. Since the etch rates for quartz glass will depend upon the purity and manufacturing process, η must also be measured for each" batch" of quartz glass.

15.2.3 In the case of natural quartz crystals used as SSTR, the etch rates vary in different directions in this highly anisotropic crystal. The effective η must therefore be determined for the specific crystallographic plane along which the crystal is cut. The accuracy that can be obtained for η in natural quartz crystals has yet to be established, but careful control of the etching conditions is a critical factor.

15.3 *Asymptotic Sensitivity*:

15.3.1 The effective mass per unit area from which all fission fragments are observed from a source thick relative to the range of fission fragments is called the asymptotic sensitivity, s_∞. A detailed discussion of how s_∞ was measured is found in Ref **(6).**

15.3.2 Asymptotically thick uranium foils are placed in good contact with mica about 0.1 mm thick. On the other side of the mica, a thin source (<100 µg/cm^2) of highly enriched ^{235}U is placed. The isotopic composition of the sources must be precisely known from mass spectrograph measurements. The mass density of the thin source is determined from α counting.

15.3.3 These SSTR-fission deposit combinations are placed in cadmium containers and exposed in a slow neutron flux. The value of s_∞ for mica (Ref **7**) is 4522 ± 70µg ^{238}U/cm ^{2}, or $(1.144 \pm 0.018) \times 10^{19}$ ^{238}U atoms/cm^2, that is, to an accuracy of ~ 1.6 %.

16. Keywords

16.1 dosimetry; fission deposit; monitor; PWR; SSTR; surveillance; track counting

APPENDIX

(Nonmandatory Information)

X1. QUALITY ASSURANCE REQUIREMENTS

X1.1 Introduction

X1.1.1 Reactor surveillance dosimetry measurements must generally adhere to established accuracy requirements. All facets of the measurement must be documented to ensure that the overall results and related uncertainties of the measurements accurately reflect the conditions under which the measurements are carried out. Furthermore, due to the long-term nature of reactor surveillance dosimetry measurements, documentation of the measurements must be maintained over the entire period of time of relevance to a particular operating reactor. Often this time period can be 20 to 40 years or possibly longer.

X1.1.2 In order to ensure accuracy and retracibility of reactor surveillance dosimetry measurements, a set of Quality Assurance (QA) procedures must be adhered to that:

X1.1.2.1 Document the origin and purity of the materials used for a dosimetry set,

X1.1.2.2 Document procedures used to prepare dosimetry sets,

X1.1.2.3 Document dosimeter loading configurations and encapsulation,

X1.1.2.4 Document the irradiation parameters for each dosimetry set,

X1.1.2.5 Document the procedures used to analyze dosimetry sets to obtain experimental data,

X1.1.2.6 Document final dosimetry results and associated uncertainties, and

X1.1.2.7 Define procedures for long-term maintenance of records.

X1.1.3 Each of these requirements will be discussed in the content of SSTR reactor surveillance dosimetry in this appendix.

X1.2 Materials Selection and Certification

X1.2.1 *Deposit Backings, SSTRs, and Chemical Reagents*:

X1.2.1.1 In order to fulfill the accuracy requirements of SSTR reactor surveillance dosimetry, high-purity chemicals and materials must be used to prepare the dosimetry sets. High-purity SSTR materials must be obtained from suppliers and subjected to QA overchecks to ensure that purity requirements are met. Deposit backing materials must be obtained and verified in a similar manner. Typically, materials of the highest purity attainable (less than 1 ppb uranium and thorium) must be obtained. A materials QA file should be kept to document suppliers of materials, analyses, and the use of these materials in dosimetry sets.

X1.2.1.2 In order to maintain the purity requirements for ultra low-mass fissionable deposits, high purity reagents and laboratory equipment must be used whenever the potential for contamination of the deposit with naturally occurring uranium and thorium impurities exists. In the case of reactor surveillance measurements using electro-deposited ultra low-mass fissionable deposits, ultra high purity chemical reagents must comprise the electroplating solution. The supplier and analyses of these materials as well as how these materials/reagents were used must be documented in the QA file.

X1.2.2 *Actinide Materials and Spikes*:

X1.2.2.1 Origin, purity, and isotopic analyses must be documented for all actinide materials used to produce fissionable deposits.

X1.2.2.2 Whenever isotopic spike materials are used, the purity (both chemical and isotopic) of the spike material must be documented.

X1.3 Dosimetry Set Preparation Procedures

X1.3.1 A detailed set of procedures must be established that govern the methods used to clean SSTRs and fissionable deposit backings, define the steps required to produce fissionable deposits, and provide for documentation of the contents and configurations of assembled dosimetry sets. Any significant departures from these procedures should be documented.

X1.3.2 Methods used to fabricate SSTR fissionable deposits must be documented. Typically, a procedure must be established for each type of fissionable deposit and this procedure must be adhered to during manufacture. When isotopic spikes are used, steps that ensure chemical equilibration of the spike isotope and major fissionable deposit isotope must be included in the procedure. If high uniformity is a requirement, measurements must be performed to document that the required degree of uniformity has been obtained.

X1.3.3 Methods and equipment used to calibrate the masses of the SSTR fissionable deposits must be documented. Calibrations of radiometric analysis equipment, procedures used, calibrations of standards used, and all calibration data relevant to the calculation of the deposit mass (and its mass uncertainty) must be documented. Periodic verifications of the calibrations of all equipment used should be required by the QA procedures.

X1.3.4 Final deposit masses, mass uncertainties, deposit and SSTR labels, and deposit-SSTR configurations must be documented for all SSTR dosimetry sets prior to emplacement in a reactor. This information is usually documented in the form of an as-built letter.

X1.4 Documentation of Loading Configuration and Encapsulation

X1.4.1 Documentation of dosimetry set details is required to ensure accurate analysis after irradiation. Documentation should consist of drawings or sketches detailing the dosimetry holder and orientation in the holder, and "as-built" loading sets detailing the dosimetry itself. The loading set should include the following:

X1.4.1.1 Identification of each dosimeter,

X1.4.1.2 Location of each dosimeter in the set,

X1.4.1.3 Mass of each fission deposit,

X1.4.1.4 Data for traceability of each dosimeter (lot number, purity, etc.), and

X1.4.1.5 Mass of dosimetry, holder, and other materials.

X1.4.2 In addition, if the dosimetry is sealed, details of the atmosphere inside the container and sealing technique should be recorded.

X1.5 Documentation of Irradiation Parameters

X1.5.1 The actual deployment and irradiation of dosimetry sets must be documented. This documentation should contain information on location of each dosimetry set, type of dosimetry holder used, and time of insertion and retrieval from the reactor. The details of dosimetry irradiations are often beyond the control of personnel responsible for assembly or analyses of dosimetry sets, or both, but must be documented nonetheless to ensure proper interpretation of dosimetry results. Relevant environmental conditions during the irradiation (temperature, γ -field, possible sources of perturbations, etc.) should be recorded. Diagrams or photographs, or both, of the deployed dosimetry set can form a part of this record.

X1.6 Dosimetry Set Analyses

X1.6.1 Procedures used and data obtained during disassembly and analyses of SSTR reactor surveillance dosimetry sets must be documented. Care should be taken to verify the as-built loading diagram during disassembly and to note any possible damage or unusual condition of dosimeters.

X1.6.2 *Fissionable Deposits*—Where possible, fissionable deposits should be subjected to an independent mass calibration after the dosimetry exposure in order to ensure that no appreciable loss of deposit mass (due to rub-off or damage) has occurred in the time period between assembly and disassembly of the dosimetry set. Typically, mass recalibration is only possible for a fraction of the deposits irradiated, but can be used as a (nonrandom) spot check on all of the deposits. Additionally, all deposits should be physically examined for possible damage. Plots of SSTR track density versus position can also be used to detect possible deposit damage or contamination, or both, of the dosimetry set. If damage or contamination is detected, the effect on the accuracy of the measurement must be evaluated. Further use of such deposits in subsequent exposures is normally precluded.

X1.6.3 *SSTRs*—Procedures used for SSTR post-exposures, etching, and track counting must be documented. Scanner calibrations (both automated and manual) must be documented. Procedures used for determination of the absolute scale (optical efficiency) must be documented for all scanners. Verifiable track standards must be used for periodic checks on scanner accuracies. The SSTRs themselves should be stored as a permanent record of the dosimetry exposure.

X1.7 Documentation of Final Dosimetry Results

X1.7.1 All data relevant to the calculation of fission rates (and associated uncertainties) must be entered into a quality analysis (QA) file that is maintained for the period of time relevant to the safe operation of the reactor. Hard bound notebooks and computer disk data files can comprise this QA file. It is advisable to keep a duplicate set of records in a different location to ensure against loss of data. QA procedures should ensure long-term traceability of all parts of an SSTR reactor surveillance dosimetry measurement.

X1.8 Procedure for Long-Term Maintenance of Records

X1.8.1 Since it may be necessary to maintain dosimeter records for time periods of decades, a storage and indexing system should be established to allow secure and safe storage and easy retrieval.

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