ENVIRONMENTAL MONITORING OF ACTINIDE CONCENTRATION IN WATER*

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ABSTRACT

Measurement of actinide concentration in water is described using a new and novel process based on the fission track method. Elements of this process are delineated with emphasis on the environmental sampling/neutron irradiation cell and neutron irradiation procedures. The sensitivity of this new process is compared with methods based on nuclear (decay) metrology. Advantages of this novel process are discussed with specific elaboration on the high sensitivity, the small sample size and the elimination of laboratory procedures that would introduce natural uranium contamination. Although our method does not distinguish between different fissile nuclides, the advantages of our process act in consort to provide a practical cost effective means for site wide environmental surveys of actinide pollution in ground water.

INTRODUCTION

The environmental impact of nuclear energy is a critical world wide issue. A principal concern of nuclear energy activities is radioactive pollution, with the long-lived actinide elements representing a significant hazard. Indeed, hazards created by the leaching of actinides into water have been an ever-present problem since the inception of nuclear energy activities. Principal concerns involve the leaching of actinides into the ground water of the ecosystem to cause long-lived pollution.

The two primary fissile nuclides that exist at all sites in the nuclear fuel cycle (whether a power reactor plant, a fuel fabrication facility or a spent fuel storage facility) are U-235 and Pu-239. Consequently, these two fissile nuclides can be used as tracers, so to speak, in site wide surveys to identify actinide releases to the environment.

In measuring actinide water pollution concentrations, it is highly advantageous to use assay methods of high sensitivity for site surveys. Methods of high sensitivity can provide early warning of any actinide release problem. Since time is a crucial factor in addressing and resolving such release problems, the method of highest sensitivity provides the earliest possible warning and hence affords the greatest possible protection.

A new and novel process of the highest sensitivity for surveying actinide concentrations in water has been conceived (1). Since our new process is based on the fission track method, it is described below within the context of the more conventional use of the fission track method for actinide mass assay.

FISSION TRACK METHODS FOR ACTINIDE MASS ASSAY

Conventional Fission Track Method

The fission track method has been applied to measure low level plutonium contamination in biological materials by Larsen and Oldham. (2-5) They demonstrated that the conventional fission track method possessed higher sensitivity for low level plutonium mass assay than any other nuclear (decay) metrology method. The conventional method involves a chemical separation of the plutonium from the biological material. After the separation, electrodeposition is used to prepare a plutonium fission deposit. The fission deposit is then placed against a solid state track recorder (SSTR) and irradiated in a known thermal neutron flux for a known time duration. Larsen and Oldham used lexan SSTR, which is a polycarbonate polymer, with a typical thermal neutron flux of 2×10^{13} neutrons/($cm^2 \cdot sec$) for a 24 hour exposure duration.

The amount of plutonium present in the biological material is then determined from the observed fission track density, the measured thermal neutron flux, the known thermal neutron fission cross section of plutonium and the measured efficiencies for: (a) Extraction of plutonium from the chemical separation process. (b) Plate out of the plutonium in the electrodeposition process. (c) Fission track counting in lexan-SSTR.

In all steps of this method, great care must be exercised against contamination by naturally occurring uranium. At the levels of concentration of interest here, which are at and below the maximum permissable concentration (MPC) levels established by regulatory agencies, naturally occurring uranium is ubiquitous. Contamination by naturally occurring uranium can create a fission track background large enough to totally compromise the measurement. Sources of ubiquitous uranium contamination are the apparatus, materials, reagents, and laboratory ware that are employed, even particulate material in the laboratory atmosphere. Indeed, contamination by naturally occurring uranium is the controlling factor in the applicability of the conventional fission track method devised by Larsen and Oldham for mass assay of plutonium in biological materials.

Consequently, sources of natural uranium contamination must be identified and if significant they must be reduced to the point where fission track background does not compromise the intended measurements. Everything must be scrutinized in this way, all equipment, apparatus, materials, reagents, laboratory-ware, . . . etc., even a specialized

"clean room" laboratory facility may be required. Hence great care and expense are incurred to properly implement this conventional method.

Asymptotic Fission Track Method

For environmental site surveys, a simpler method of higher sensitivity is needed to provide early warning of any actinide release to the environment. Such an early warning method need only signal whether or not actinide pollution has occurred. When evidence for actinide pollution has been established through application of this early warning method, it may then be desirable to determine isotopic composition using either the conventional fission track method or some other separation method.

We have conceived a process employing the fission track method wherein there are no intermediate steps or stages between the act of environmental sampling and the neutron irradiation used to induce fission tracks. Consequently, introduction of natural uranium contamination by intermediate stages of the process is eliminated. Moreover, any such contamination introduced after irradiation does not contribute to fission track background and is therefore of no consequence. The process does not determine which actinide element or isotope is present; this would normally not be required of an early warning detection system. In this respect, the high sensitivity of our new asymptotic fission track method ideally compliments the selectivity for isotope identification possible with the conventional fission track method.

In our process, a very special cell is used for both environmental sampling and subsequent neutron irradiation. This cell possesses a number of novel features, perhaps the most unusual of which is the use of a liquid for an asymptotic SSTR detection configuration. The environmental water sample is the asymptotic source. Hence, it is appropriate to call our new process the asymptotic fission track method.

In an asymptotic SSTR detection configuration, the source of fission fragments (i.e. the fission deposit) is thicker than the range of fission fragments in the source. Consequently increasing the source thickness will no longer increase the observed fission track density; the SSTR sensitivity attains a maximum or limiting value called the asymptotic sensitivity. Hence in this configuration, the source is "infinitely" thick in so far as the SSTR fission fragment detector is concerned. The asymptotic sensitivity is effectively the maximum number of atoms per unit area of the medium from which fission tracks can be observed by an SSTR placed in direct contact with the medium. We have used the symbol s_∞ to denote the asymptotic sensitivity, which can be expressed in units of gm/cm^2 or atoms/cm^2.

Metallic actinide foils have been used as asymptotic media for neutron dosimetry(6) and to measure spontaneous fission half-lives(7). To conduct these experiments, special calibration irradiations were used to measure the asymptotic sensitivity. Calculations have been used to infer asymptotic sensitivity values of other fissile elements from the measured asymptotic sensitivity of metallic uranium(8). More recently the asymptotic sensitivity of uranium oxide foil has been measured to permit direct observation of fission rates in uranium oxide fuel(9).

The concept of a liquid as the source medium for an asymptotic SSTR detection configuration has heretofore never been conceived or used, let alone applied for the liquid water samples of interest in environmental surveys. An intrinsic advantage of the asymptotic fission track method is that the components of the sampling/irradiation cell can not contribute directly to the fission track background. However, a possible source of fission track background could arise through leach out of natural uranium contamination from components of the sampling/irradiation cell into the environmental water sample. One other source of background for our process must be stressed, namely the natural uranium impurities that exist in the SSTR itself(10). For a properly designed sampling/irradiation cell, these latter impurities will be the dominant source of fission track background.

For numerical estimates, the following approximate representation of the asymptotic sensitivity, s_∞, can be used(6)

$$s_\infty = \eta R/2 \qquad \text{(Eq. 1)}$$

where η is the optical efficiency of the SSTR and R is the range of fission fragments in the asymptotic medium. The asymptotic sensitivity expressed in units of atoms/cm^2, a_∞, can be obtained by multiplying s_∞ by the atom density of the asymptotic medium. One can therefore write

$$a_\infty = N_a s_\infty = N_a \eta R/2 \qquad \text{(Eq. 2)}$$

where N_a is the number of atoms per cm^3 of the asymptotic medium. Using the following numerical estimates for water, $R \cong 20u$ and $N_a \cong 10^{23}$ atoms/cm^3, one has $a_\infty \cong 10^{20}$ atoms/cm^2. Here we have assumed that $\eta = 1$, which is approximately true for most candidate SSTR materials that will be considered for environmental surveys. An American Society for Testing and Materials (ASTM) standard on SSTR techniques has been established and should be consulted for further details(11).

ENVIRONMENTAL SAMPLING/NEUTRON IRRADIATION CELL

The sampling/irradiation cell is a fundamental element of the asymptotic fission track method for environmental water monitoring. The asymptotic water configuration for SSTR detection can be obtained in a number of different

ways. The SSTR can be suspended in the water volume. Alternatively the SSTR can be used as a liner in the cell, either at the bottom or around the inside of the cell, should the cell be cylindrical. The cell can even be fabricated from an SSTR material such as glass, in which case the interior surfaces of the cell will provide the desired asymptotic water configuration. In all cases, water should cover the SSTR area of interest by a layer greater than the fission fragment range, R ≅ 20u. At the same time, the cell geometry should afford the SSTR area of interest with an unobstructed view of the asymptotic water volume. This will maintain the high efficiency corresponding to the asymptotic sensitivity of water and eliminate any possible contribution of fission track background from actinide impurities in the structural components of the cell.

Figure 1 is an illustration of a sampling/irradiation cell where the SSTR is placed at the bottom of the cell to provide the asymptotic SSTR detection configuration. In this cell, the two capillary tubes extend down inside the cell to within a distance d above the top of the cell, where $d \gtrsim R$. These capillary tubes allow collection of the environmental water sample, but are sealed off at all other times.

For environmental surveys, it is advantageous to simultaneously irradiate many cells to the same thermal neutron fluence. Since only limited irradiation volumes exist in reactors, the smaller the size of the cell the larger will be the allowable number of cells that can be simultaneously irradiated. Smaller cell size is advantageous in that lower radioactivity is produced by the neutron irradiation of the cell.

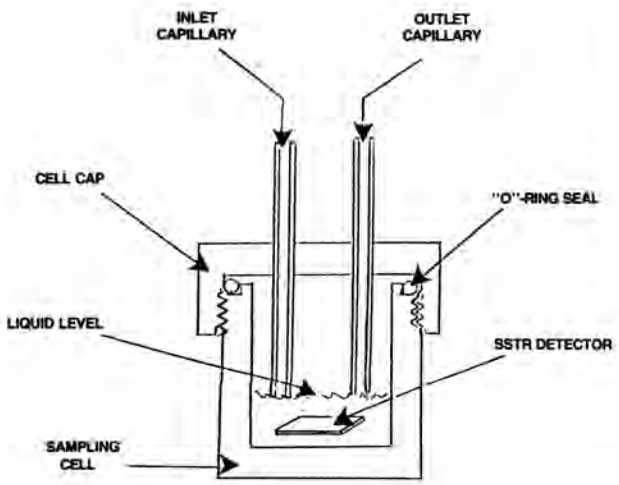

Fig. 1. SSTR Environmental Sampling/Neutron Irradiation Cell for Monitoring Actinide Pollution in Water.

Consequently, the smaller the cell the lower is any personnel exposure that arises in post-irradiation handling of the cells. To this end, the cell and any associated components should be fabricated of materials that possess the lowest neutron activation cross sections. This generally implies that metals can not be used and that metallic impurities must be kept to a minimum.

Perhaps of even greater significance to cell characteristics than the size of the cell and the subsequently induced radioactivity is the decomposition of water by exposure to the nuclear reactor radiation field. In this process of radiolytic dissociation or radiolysis, hydrogen and oxygen gases are liberated. Since our water samples are contained in a closed cell, the gas pressure will increase until a steady-state (equilibrium) value is attained. The magnitude of this pressure depends chiefly on the radiation field intensity and the concentration of impurities as well as the type of impurities in the water.

Typical values of gas pressure can be found in experiments described in the text book of Hoag(12). In these experiments, a neutron flux of 2E + 13 neutrons/($cm^2 \cdot sec$) generated a pressure of approximately 1 atmosphere (above atmospheric pressure) after an irradiation duration of about 1 hour. There was also a modest temperature rise of approximately 10°C above room temperature.

Although pressure relief can be achieved in a number of ways, a simple method exists that takes advantage of the asymptotic detection configuration, which can actually be obtained with a very thin layer of water, $d \gtrsim 20\mu$. Consider therefore the cell depicted in Fig. 1. If this cell is fabricated so that the inside length of the cell, h, is much greater than d, then the volume of the air space, V_{air}, is much greater than the volume of water, V_w, in the cell. In fact for a cylindrical cell, the ratio of the water to air volumes is simply $V_w/V_{air} \cong d/h << 1$. Because of this condition, two factors operate to limit gas pressure. The first factor is that the amount of gas generated through radiolysis is proportional to the amount of water exposed, which can be held to a minimum in this design. The second factor is the much larger volume of air, $V_{air} >> V_w$, into which the gas can escape. Both of these factors act in consort, so that the equilibrium gas pressure attained in such a sampling/irradiation cell is so low that cell integrity is assured.

NEUTRON IRRADIATION PROCEDURES

Three different procedures can be employed for neutron irradiation of environmental water samples in our sampling/irradiation cells. All three procedures use a uniform flux assembly to insure that all irradiated cells are exposed to an identical thermal neutron fluence. The original measurements of asymptotic sensitivity in uranium metal serve to illustrate one possible version of a uniform flux assembly. As shown in Fig. 2, this particular uniform flux assembly was

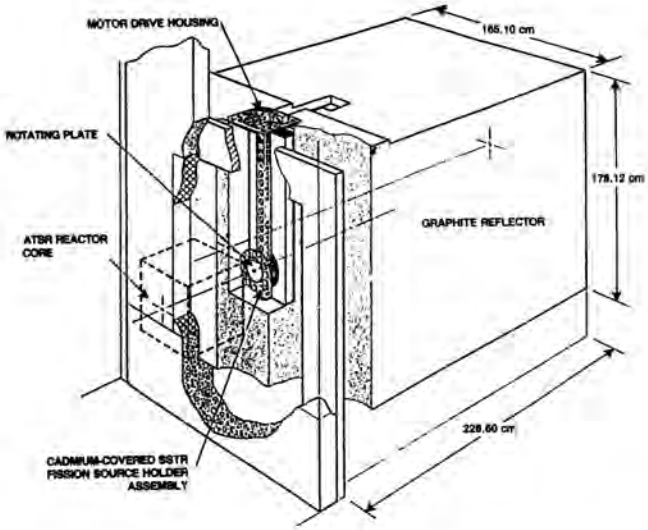

Fig. 2. Cross-Sectional View of the ATSR Uniform Flux Assembly.

a rotating plate that was mounted in the graphite reflector of the Argonne Thermal Source Reactor (ATSR). The rotating plate was located near the front face of the graphite reflector and faced the ATSR core. Approximately 12 cadmium covered SSTR could be mounted on the surface of the rotating plate.

For environmental monitoring, it is advantageous to simultan-eously irradiate many samples in a uniform flux assembly. For example, if a site survey required 50 environmental water samples from distributed locations around and under the site, it would then be desirable for the uniform flux assembly to handle 50 or more of our sampling/irradiation cells at one time. The cell capacity of a uniform flux assembly depends on the size of the uniform flux assembly as well as the size of a single cell. Since our sampling/irradiation cells can be small, such a capacity could be accommodated even by the original ASTR uniform flux assembly. Here the plate was approximately 12 inches in diameter, which provides a circumference of about 96 cm. Hence to accommodate 50 of our sampling/irradiation cells, the outer dimension of our cell would have to be approximately 1.9 cm or 0.75 in. Consequently, the original uniform flux assembly can handle 50 cells easily. Hence through careful design, it will be possible to extend this capacity many-fold.

The fission track density, ρ_∞, attained in the SSTR of a sampling/irradiation cell from a thermal neutron irradiation is given by the relation

$$\rho_\infty = \phi_{th} \cdot T \cdot \sigma_{th} \cdot a_\infty \cdot f_a \qquad \text{(Eq. 3)}$$

where ϕ_{th} is the thermal neutron flux in units of neutrons/(cm^2 · sec), T is the duration of the irradiation in seconds, ϕ_{th} is the thermal neutron fission cross section of the fissile nuclide in cm^2 and f_a is the atom fraction of the fissile nuclide in the water. Using these units, the track density, ρ_∞, will possess units of tracks/cm^2.

In the first neutron irradiation procedure, one employs a uniform flux assembly in a manner similar to the original asymptotic sensitivity measurements in uranium metal(8). Solving Eq. (3) for the atom fraction f_a, one has

$$f_a = \rho_\infty \Big/ \left(\phi_{th} \cdot T \cdot \sigma_{th} \cdot a_\infty\right) \qquad \text{(Eq. 4)}$$

Consequently f_a can be determined by measuring the track density, ρ_∞, provided one knows the factors in the denominator of Eq. (4). These factors can be determined as follows: (a) The thermal neutron flux, ϕ_{th}, can be determined using conventional neutron dosimetry methods. (b) The time duration T can be measured by using available monitoring of the reactor power/time history for the irradiation. Many power monitoring charts in the reactor control room are available for this purpose. (c) The value of the thermal neutron fission cross section, ϕ_{th}, can be found in the extensive nuclear data literature that exists for neutron cross sections. (d) The asymptotic sensitivity of water, a_∞, can be measured by following the procedures originally used to determine the asymptotic sensitivity of uranium metal(8). Since all of these factors arise in neutron dosimetry, one can see why procedure 1 is so closely related to our original asymptotic sensitivity measurements, which were also carried our for neutron dosimetry applications.

In the second procedure, at least one SSTR neutron dosimeter, using an accurately quantified actinide fission deposit, is mounted on the uniform flux assembly. The mass density (atoms/cm^2) of this actinide deposit, d_a, must be accurately known and should be small, so that fission fragment self-absorption within the thickness of the deposit is negligible. The track density for this SSTR neutron dosimeter ρ_d, is given by

whereas the track density, ρ_∞, attained in the asymptotic water configuration of one of our sampling/irradiation cells is given as before by Eq. (3).

$$\rho_d = \phi_{th} \cdot T \cdot \sigma_{th} \cdot d_a \qquad \text{(Eq. 5)}$$

Dividing Eq. (3) by Eq. (5) and solving for f_a, one finds

$$f_a = \left(d_a / a_\infty\right) \cdot \left(\rho_\infty / \rho_d\right) \qquad \text{(Eq. 6)}$$

Hence the atom fraction, f_a, is determined by the ratio of the measured track densities (ρ_∞/ρ_d) in terms of the

known ratio of atom densities (d_a/a_∞). Here the atom fraction is the equivalent atom fraction of the actinide deposit. In other words if the deposit were natural uranium, the atom fraction f_a given by Eq. (6) is the equivalent natural uranium atom fraction, i.e. the atom fraction that would exist in the water sample if only natural uranium was present.

Procedure 2 has an advantage over procedure 1 in that three neutron dosimetry factors, namely φ_{th}, T and σ_{th}, need no longer be determined. Beyond measuring the track densities ρ_d and ρ_∞ , one only has to know the actinide deposit mass density d_a and the asymptotic sensitivity of water a_∞.

Procedure 3 is even simpler, since it eliminates the need to determine the fourth factor, namely the asymptotic sensitivity a_∞. In procedure 3, one prepares water solutions of known actinide concentrations. Actinide solution concentrations can be accurately prepared by dissolving a known mass of the actinide, say natural uranium, in a known volume of water. Very low concentration levels can be achieved by accurate dilution with water. We shall define solutions so prepared as standard water solutions of known atom fraction f_s.

Water samples from the standard solution should be acquired in our sampling/irradiation cells using the identical procedure for taking environmental water samples. In the neutron irradiation, at least one of the sampling/irradiation cells should contain the standard solution. Under these conditions, Eq. (3) still represents the track density, ρ_∞, attained in the environmental water samples. The track density attained in the standard solution, ρ_s , can be written as

$$\rho_s = \phi_{th} \cdot T \cdot \sigma_{th} \cdot a_\infty \cdot f_s \qquad \text{(Eq. 7)}$$

Dividing Eq. (3) by Eq. (7) and solving for f_a, one has

$$f_a = f_s \cdot \left(\rho_\infty / \rho_s\right) \qquad \text{(Eq. 8)}$$

Thus in procedure 3, the sought atom fraction f_a is simply the standard atom fraction f_s multiplied by the ratio of the observed track densities (ρ_∞/ρ_s). Just as in procedure 2, procedure 3 provides an equivalent atom fraction, i.e. the atom fraction f_a in Eq. 8 is the equivalent atom fraction in the environmental water sample of those actinides that comprise the standard solution. The beauty of procedure 3 is that all neutron dosimetry factors are eliminated.

Although accuracy is not a particularly significant requisite for an early warning system, it should be noted that the accuracy of procedure 3 is limited essentially by the uncertainty of f_s. Because of the high sensitivity of our fission track process, the track densities ρ_∞ and ρ_s are so large that they can be measured, in principle, to high statistical precision. As a conse-quence,the accuracy of procedure 3 depends chiefly on how accurately the standard solution can be prepared.

There is also less likelihood that systematic uncertainties will arise in procedure 3. This conclusion follows from the fact that both environmental and standard solution water samples use the same: (a) Water sampling procedure. (b) Sampling/irradiation cell. (c) Uniform flux assembly neutron irradiation. In addition, track densities ρ_∞ and ρ_s both arise from an asymptotic water configuration in the same SSTR material. Moreover, the same scanning procedures, manual or automated, can be used to measure both track densities.

It must be emphasized that our process does not distinguish between different fissile nuclides that can exist in environmental water samples. For this reason, it has already been explained that neither neutron irradiation procedure 2 nor procedure 3 can provide isotopic atom fractions, but only furnish equivalent atom fractions. Nevertheless it can be shown that an equivalent atom fraction is an upper bound on the corresponding isotopic atom fraction. As a consequence, our process can be implemented in terms of equivalent atom fractions and thereby provide an early warning method possessing significant practical and cost effective advantages. For environmental surveys, one would establish baseline values of track density with the asymptotic fission track method that are representative of the indigenous environment. These baseline values provide background levels for comparison with follow-on observations. An increased track density in follow-on observations would signal that an actinide release to the environment has occurred.

Consequently the asymptotic fission track method works in a manner that is analogous to the use of an alpha particle survey meter for health physics or environmental monitoring. More specifically, an alpha survey meter alerts the user to the existence of some trans-uranic deposit or contamination. It can not, however, provide information on the isotopic composition of the deposit or contamination. Such data must be determined by the application of complimentary detection methods.

ADVANTAGES

The sensitivities that can be attained from the asymptotic fission track method for MPC concentration levels of different actinide isotopes in water have been calculated from Eq. (3) and are shown in Table I. These track density estimates have been obtained assuming a neutron irradiation with a thermal flux of 2×10^{12} neutrons/ (cm^2 · sec) for a duration of one hour. Even with these conservative assumptions, it is apparent that adequate track densities are generated to detect concentrations consider-ably lower than MPC values.

TABLE I
Estimated Track Densities for MPC Levels in Water

Actinide Isotope	MPC (curies/cm^3)	Atom Fraction	σ_{th}[a] (cm^2)	Track Density (tracks/cm^2)	Number of Decays[c]
Natural Uranium	3 E-11	8.09 E-09[b]	577 E-24[b]	3.4 E+06	8.0
U-235	3 E-11	3.55 E-07	577 E-24	1.5 E+08	8.0
Pu-239	5 E-12	2.03 E-12	714 E-24	1.0 E+03	1.3

a. σ_{th} is the thermal neutron fission cross section.

b. Contribution from U-235 at a presence of 0.72 weight percent in natural uranium.

c. Number of alpha-particle decays in one hour in an asymptotic volume of 1 cm^2 x 20 μ = 2.0 E-03 cm^3.

The last column in Table I provides a general basis for comparing the asymptotic fission track method with nuclear metrology methods. Here it can be seen that the sensitivity of the asymptotic fission track method is orders of magnitude greater than the sensitivity of any method based on nuclear (decay) metrology. While the intrinsic elements and factors comprising different metrology methods can vary widely, making direct comparisons difficult without introducing some set of assumptions, it is clear that our process possesses a signifi-cant sensitivity advantage. It is this high sensitivity that furnishes the early warning capability of the asymptotic fission track method. Moreover, the asymptotic fission track method can be applied in a complimentary way with any separation method should follow-on isotope identification be desirable. In contrast with our new and novel process, current practice invariably utilizes separation methods for effluent and environmental radiation surveillance of actinides in ground water(13).

Separation methods, which lack the sensitivity of the asymptotic fission track method, can require liters of water for a single sample and some separation methods use up to 10 liters of water or more per sample. Despite the need for such large samples, these separation methods still fall far short of the sensitivity of our process. For site wide surveys involving many samples, of say 50 or more samples, it can be impractical to implement these separation methods. On the other hand, high sensitivity as well as small size of the sampling/irradiation cells make the asymp-totic fission track method ideal for site wide surveys.

Since our process does not possess any intermediate steps or stages between sampling and irradiation, measurements can not be compromised by ubiquitous natural uranium contamination introduced through laboratory procedures. In contrast with other methods, the ability to apply a combined environmental sampling/neutron irradiation cell virtually eliminates the problem of contamination by natural uranium.

Finally, incorporation of standard (actinide concentration) solutions in uniform flux assembly irradiations: (1)Eliminates dependence on all neutron dosimetry factors, (2) Reduces the process to one of relative measurements, i. e. dependence on only relative track density ratios and (3) Improves accuracy by reduction of systematic uncertainties.

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