Designation: E321 – 96 (Reapproved 2012)

Standard Test Method for
Atom Percent Fission in Uranium and Plutonium Fuel (Neodymium-148 Method)[1]

This standard is issued under the fixed designation E321; 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 covers the determination of stable fission product ^{148}Nd in irradiated uranium (U) fuel (with initial plutonium (Pu) content from 0 to 50 %) as a measure of fuel burnup (**1-3**).[2]

1.2 It is possible to obtain additional information about the uranium and plutonium concentrations and isotopic abundances on the same sample taken for burnup analysis. If this additional information is desired, it can be obtained by precisely measuring the spike and sample volumes and following the instructions in Test Method E267.

1.3 *This standard does not purport to address all of the safety concerns, if any, 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:*[3]

D1193 Specification for Reagent Water

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[4]

E244 Test Method for Atom Percent Fission in Uranium and Plutonium Fuel (Mass Spectrometric Method) (Withdrawn 2001)[4]

E267 Test Method for Uranium and Plutonium Concentrations and Isotopic Abundances

3. Summary of Test Method

3.1 Fission product neodymium (Nd) is chemically separated from irradiated fuel and determined by isotopic dilution mass spectrometry. Enriched ^{150}Nd is selected as the Nd isotope diluent, and the mass-142 position is used to monitor for natural Nd contamination. The two rare earths immediately adjacent to Nd do not interfere. Interference from other rare earths, such as natural or fission product ^{142}Ce or natural ^{148}Sm and ^{150}Sm is avoided by removing them in the chemical purification (**4** and **5**).

3.2 After addition of a blended ^{150}Nd, ^{233}U, and ^{242}Pu spike to the sample, the Nd, U, and Pu fractions are separated from each other by ion exchange. Each fraction is further purified for mass analysis. Two alternative separation procedures are provided.

3.3 The gross alpha, beta, and gamma decontamination factors are in excess of 10^3 and are normally limited to that value by traces of ^{242}Cm, ^{147}Pm, and ^{241} Am, respectively (and sometimes ^{106}Ru), none of which interferes in the analysis. The 70 ng ^{148}Nd minimum sample size recommended in the procedure is large enough to exceed by 100-fold a typical natural Nd blank of 0.7 ± 0.7 ng ^{148}Nd (for which a correction is made) without exceeding radiation dose rates of 20 μ Sv/h (20 mR/h) at 1 m. Since a constant amount of fission products is taken for each analysis, the radiation dose from each sample is similar for all burnup values and depends principally upon cooling time. Gamma dose rates vary from 200 μ Sv/h (20 mR/h) at 1 m for 60-day cooled fuel to 20 μ Sv/h (2 mR/h) at 1 m for 1-year cooled fuel. Beta dose rates are an order of magnitude greater, but can be shielded out with a ½-in. (12.7-mm) thick plastic sheet. By use of such simple local shielding, dilute solutions of irradiated nuclear fuel dissolver solutions can be analyzed for burnup without an elaborate shielded analytical facility. The decontaminated Nd fraction is mounted on a rhenium (Re) filament for mass analysis. Samples from 20 ng to 20 μg run well in the mass spectrometer with both NdO$^+$ and Nd$^+$ ion beams present. The metal ion is enhanced by deposition of carbonaceous material on the filament as oxygen getter. (Double and triple filament designs do not require an oxygen getter.)

---

[1] This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

Current edition approved June 1, 2012. Published June 2012. Originally approved in 1967 . Last previous edition approved in 2005 as E321 – 96(2005). DOI: 10.1520/E0321-96R12.

[2] The boldface numbers in parentheses refer to the list of references appended to this test method.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] The last approved version of this historical standard is referenced on www.astm.org.

4. Significance and Use

4.1 The burnup of an irradiated nuclear fuel can be determined from the amount of a fission product formed during irradiation. Among the fission products, ^{148}Nd has the following properties to recommend it as an ideal burnup indicator: (*1*) It is not volatile, does not migrate in solid fuels below their recrystallization temperature, and has no volatile precursors. (*2*) It is nonradioactive and requires no decay corrections. (*3*) It has a low destruction cross section and formation from adjacent mass chains can be corrected for. (*4*) It has good emission characteristics for mass analysis. (*5*) Its fission yield is nearly the same for ^{235}U and ^{239}Pu and is essentially independent of neutron energy **(6).** (*6*) It has a shielded isotope, ^{142}Nd, which can be used for correcting natural Nd contamination. (*7*) It is not a normal constituent of unirradiated fuel.

4.2 The analysis of ^{148}Nd in irradiated fuel does not depend on the availability of preirradiation sample data or irradiation history. Atom percent fission is directly proportional to the ^{148}Nd-to-fuel ratio in irradiated fuel. However, the production of ^{148}Nd from ^{147}Nd by neutron capture will introduce a systematic error whose contribution must be corrected for. In power reactor fuels, this correction is relatively small. In test reactor irradiations where fluxes can be very high, this correction can be substantial (see Table 1).

4.3 The test method can be applied directly to U fuel containing less than 0.5 % initial Pu with 1 to 100 GW days/metric ton burnup. For fuel containing 5 to 50 % initial Pu, increase the Pu content by a factor of 10 to 100, respectively in both reagents 5.3 and 5.4.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined in Specification D1193.

5.3 *Blended ^{148}Nd, ^{239}Pu, and ^{238}U Calibration Standard*—Prepare a solution containing about 0.0400 mg ^{148}Nd/litre, 50 mg ^{238}U/litre, and 2.5 mg ^{239}Pu/litre, in nitric acid (HNO_3, 1 + 1) with 0.01 *M* hydrofluoric acid (HF) as follows. With a new calibrated, clean, Kirk-type micropipet, add 0.500 mL of ^{239}Pu known solution (see 5.11) to a calibrated 1-litre volumetric flask. Rinse the micropipet into the flask three times with HNO_3 (1 + 1). In a similar manner, add 0.500 mL of ^{238}U known solution (see 5.12) and 1.000 mL of ^{148}Nd known solution (see 5.9). Add 10 drops of concentrated HF and dilute exactly to the 1-litre mark with HNO_3 (1 + 1) and mix thoroughly.

5.3.1 From K_{148} (see 5.9), calculate the atoms of ^{148}Nd/mL of calibration standard, C_{148}, as follows:

$$C_{148} = \frac{\text{mL } {}^{148}\text{Nd known solution}}{1000 \text{ mL calibration standard}} \times K_{148} \tag{1}$$

5.3.2 From K_{238} (see 5.12), calculate the atoms of ^{238}U/mL of calibration standard, C_{238}, as follows:

$$C_{238} = \frac{\text{mL } {}^{238}\text{U known solution}}{1000 \text{ mL calibration standard}} \times K_{238} \tag{2}$$

5.3.3 From K_{239} (see 5.11), calculate the atoms of ^{239}Pu/mL of calibration standard, C_{239}, as follows:

$$C_{239} = \frac{\text{mL } {}^{239}\text{Pu known solution}}{1000 \text{ mL calibration standard}} \times K_{239} \tag{3}$$

5.3.4 Flame seal 3 to 5-mL portions in glass ampoules to prevent evaporation after preparation until time of use. For use, break off the tip of an ampoule, pipet promptly the amount required, and discard any unused solution. If more convenient, calibration solution can be flame-sealed in pre-measured 1000-µL portions for quantitative transfer when needed.

5.4 *Blended ^{150}Nd, ^{233}U, and ^{242}Pu Spike Solution*—Prepare a solution containing about 0.4 mg ^{150}Nd/litre, 50 mg ^{233}U/litre, and 2.5 mg ^{242}Pu/litre in HNO_3 (1 + 1) with 0.01 *M* HF. These isotopes are obtained in greater than 95, 99, and 99 % isotopic purity, respectively, from the Isotopes Sales Department of Oak Ridge National Laboratory. Standardize the spike solution as follows:

5.4.1 In a 5-mL beaker, place about 0.1 mL of ferrous solution, exactly 500 µL of calibration standard (see 5.3) and exactly 500 µL of spike solution (see 5.4). In a second beaker,

TABLE 1 K Factors to Correct ^{148}Nd for ^{147}Nd Thermal Neutron Capture[A]

Total Neutron Flux, φ (neutrons/cm^2/s)	Total Neutron Exposure, φl (neutrons/cm^2)				
	1×10^{20}	3×10^{20}	1×10^{21}	2×10^{21}	3×10^{21}
3×10^{12}	0.9985	0.9985	0.9985	0.9985	0.9985
1×10^{13}	0.9956	0.9952	0.9950	0.9950	0.9950
3×10^{13}	0.9906	0.9870	0.9856	0.9853	0.9852
1×10^{14}	0.9858	0.9716	0.9598	0.9569	0.9559
3×10^{14}	0.9835	0.9592	0.9187	0.9008	0.8941
1×10^{15}	0.9826	0.9526	0.8816	0.8284	0.8006

[A] Assuming continuous reactor operation and a 274 ± 91 barn ^{147}Nd effective neutron absorption cross section for a thermal neutron power reactor. This cross section was obtained by adjusting the 440 ± 150 barn ^{147}Nd cross section **(7)** measured at 20°C to a Maxwellian spectrum at a neutron temperature of 300°C.

---

[5] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

place about 0.1 mL of ferrous solution and 1 mL of calibration standard without any spike. In a third beaker, place about 0.1 mL of ferrous solution and 1 mL of spike solution without standard. Mix well and allow to stand for 5 min to reduce Pu (VI) to Pu (III) or Pu (IV).

5.4.2 Follow the procedure described in 7.2.4 – 7.5.8 or 7.6.2 – 7.7.11. Measure the Pu, U, and Nd isotopes by surface ionization mass spectrometry following the procedure described in 7.8.1 – 7.8.3.2 . On the Pu fractions, record the atom ratios of ^{242}Pu to ^{239}Pu in the calibration standard, $C_{2/9}$; in the spike solution, $S_{2/9}$; and in the standard-plus-spike mixture, $M_{2/9}$. On the U fractions record the corresponding ^{233}U-to-^{238}U ratios, $C_{3/8}$, $S_{3/8}$, and $M_{3/8}$. On the Nd fractions, record the corresponding ^{150}Nd-to-^{148}Nd ratios, $C_{50/48}$, $S_{50/48}$, and $M_{50/48}$. Correct all average measured ratios for mass discrimination bias (see 6.2).

5.4.3 Calculate the number of atoms of ^{150}Nd/mL of Spike, A_{50}, as follows:

$$A_{50} = C_{148}\left[\left(M_{50/48} - C_{50/48}\right)/\left(1 - M_{50/48}/S_{50/48}\right)\right] \tag{4}$$

5.4.4 Calculate the number of atoms of ^{233}U/mL of spike, A_{33}, as follows:

$$A_{33} = C_{238}\left[\left(M_{3/8} - C_{3/8}\right)/\left(1 - M_{3/8}/S_{3/8}\right)\right] \tag{5}$$

5.4.5 Calculate the number of atoms of ^{242}Pu/mL spike, A_{42}, as follows:

$$A_{42} = C_{239}\left[\left(M_{2/9} - C_{2/9}\right)/\left(1 - M_{2/9}/S_{2/9}\right)\right] \tag{6}$$

5.4.6 Store in the same manner as the calibration standard (see 5.3), that is, flame seal 3 to 5-mL portions in glass ampoules. For use, break off the tip of an ampoule, pipet promptly the amount required, and discard any unused solution. If more convenient, spike solution can be flame sealed in a premeasured 1000-µL portions for quantitative transfer to individual samples.

5.5 *Ferrous Solution (0.001 M)*—Add 40 mg of reagent grade ferrous ammonium sulfate ($Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$) and 1 drop of concentrated H_2SO_4 to 5 mL of redistilled water. Dilute to 100 mL with water and mix. This solution does not keep well. Prepare fresh daily.

5.6 *Filament Mounting Solution*—Dissolve 70 mg of sucrose in 100 mL of water (single filament only).

5.7 *Hydrofluoric Acid*—Reagent grade concentrated HF (28 *M*).

5.8 *Methanol,* absolute.

5.9 *^{148}Nd Known Solution*—Heat natural Nd_2O_3 (>99.9 % pure) in an open crucible at 900°C for 1 h to destroy any carbonates present and cool in a dessicator. Weigh 0.4071 g of Nd_2O_3 and place it in a calibrated 500-mL volumetric flask. Dissolve the oxide in HNO_3 (1 + 1) and dilute to the 500-mL mark with HNO_3 (1 + 1) and mix thoroughly. By using the weight of Nd_2O_3 in grams, and the purity, calculate the atoms of ^{148}Nd/mL of known solution, K_{148}, as follows:

$$K_{148} = \text{g Nd}_2\text{O}_3/500\text{ mL} \times \%\text{ purity}/100 \tag{7}$$

$$\times 50.38\text{mg }^{148}\text{Nd}/1\text{ g Nd}_2\text{O}_3 \times (6.025$$

$$\times 10^{20}\text{ atoms})/147.92\text{ molecular weight}$$

5.10 *Perchloric Acid*—70 % $HClO_4$.

5.11 *^{239}Pu Known Solution*—Add 10 mL of HCl (1 + 1) to a clean calibrated 100-mL flask. Cool the flask in an ice water bath. Allow time for the acid to reach approximately 0°C and place the flask in a glove box. Displace the air in the flask with inert gas (Ar, He, or N_2). Within the glove box, open the U.S. National Institute of Standards and Technology Plutonium Metal Standard Sample 949, containing about 0.5 g of Pu (actual weight individually certified), and add the metal to the cooled HCl. After dissolution of the metal is complete, add 1 drop of concentrated HF and 40 mL of HNO_3 (1 + 1) and swirl. Place the flask in a stainless-steel beaker for protection and invert a 50-mL beaker over the top and let it stand for at least 8 days to allow any gaseous oxidation products to escape. Dilute to the mark with HNO_3 (1 + 1) and mix thoroughly. By using the individual weight of Pu in grams, the purity, and the molecular weight of the Pu given on the NIST certificate, with the atom fraction, A_9, determined as in 8.8, calculate the atoms of ^{239}Pu/mL of ^{239}Pu known solution, K_{239} , as follows:

$$K_{239} = [(\text{mg Pu}/100\text{ mL solution}) \times \%\text{ purity}/100) \tag{8}$$

$$\times (6.025 \times 10^{20}\text{ atoms/Pu molecular weight}) \times A_9]$$

5.12 *^{238}U Known Solution*—Heat U_3O_8 from the National Institute of Standards and Technology Natural Uranium Oxide Standard Sample 950 in an open crucible at 900°C for 1 h and cool in a dessicator in accordance with the certificate accompanying the standard sample. Weigh about 12.0 g of U_3O_8 accurately to 0.1 mg and place it in a calibrated 100-mL volumetric flask. Dissolve the oxide in HNO_3 (1 + 1). Dilute to the 100-mL mark with HNO_3 (1 + 1) and mix thoroughly. By using the measured weight of U_3O_8 in grams, the purity given on the NIST certificate, and the atom fraction ^{238}U, A_8, determined as in 8.5, calculate the atoms ^{238}U/mL of ^{238}U solution, K_{238}, as follows:

$$K_{238} = [(\text{ g } U_3O_8/100\text{ mL solution}) \times (\%\text{ purity}/100 \tag{9}$$

$$\times 848.0\text{ mg } U/1\text{ g } U_3O_8) \times (6.025$$

$$\times 10^{20}\text{ atoms}/238.03\text{ molecular weight}) \times A_8$$

5.13 *Reagents and Materials for Procedure A:*

5.13.1 *Dowex AGMP-1 Resin*—Convert Dowex AGMP-1 (200 to 400 mesh) chloride form resin[6] to nitrate form by washing 200 mL of resin in a suitable column (for example, a 250-mL buret) with HNO_3 (1 + 1) until a drop of effluent falling into an $AgNO_3$ solution remains clear. Finally, rinse with water, and dry overnight in a vacuum dessicator. Store the resin in an airtight container. Since the elution characteristics of ion exchange resins depend upon their actual percentage cross linkage and particle size (surface-to-volume ratio), which may vary from one lot to the next, it is most convenient to set aside a bottle of resin to be used solely for this procedure. Before use on actual samples, a small amount of tracer ^{147}Nd should be taken through the procedure. Collect each consecutive 80 mm fraction of eluant and count for γ radioactivity. If

---

[6] Dowex resin (AGMP-1 or AG1-X4, 200–400 mesh) obtained from Bio-Rad Laboratories, 3300 Regatta Blvd., Richmond, CA, has been found satisfactory.

over 80 % of the ^{147}Nd appears in the Nd fraction, the resin can be used as directed; if not, small adjustments can be made in the elution volumes collected.

5.13.2 *Hydrochloric Acid*[7]—Prepare reagent low in U and dissolved solids by saturating redistilled water in a polyethylene container to 12 *M* with HCl gas which has passed through a quartz-wool filter. Dilute 1 + 1 and 1 + 24 with redistilled water. Store each solution in a polyethylene container. One drop of acid, when evaporated on a clean microscope slide cover glass, must leave no visible residue. Test monthly. Commercial HCl (cp grade) in glass containers has been found to contain excessive residue (dissolved glass) which inhibits ion emission.

5.13.3 *Dowex 1 Resin*—Dowex 1-X4 (200 to 400 mesh) chloride form resin.[6]

5.13.4 *Ion Exchange Column (Type I)*—Type I ion exchange columns are used whenever Dowex AG 1-X4 columns are specified in the procedure. These columns are prepared from 230-mm disposable glass capillary (Pasteur) pipets that have a glass wool plug inserted to contain the resin beads. Filling this column to the top is considered a 2-mL addition of reagent solution.

5.13.5 *Ion Exchange Column (Type II)*—Type II ion exchange columns are used whenever AGMP-1 columns are specified in the procedure. These columns are prepared from 4-mm (inside diameter) glass tubing that has been heated and drawn, forming a long, fine tip. A coating of paraffin wax melted on the long tip keeps the methanol from climbing the outside surface. A small plug of glass wool is inserted to contain the resin beads. The length of the column above the glass wool plug should be a little more than 22 cm. The columns are carefully marked every 4 cm above the top of the resin bed (4 cm = 0.5 mL of solution).

5.13.6 *Methanolic HNO_3 Eluant*—Pipet 10 mL of HNO_3 (1 + 500) into a 100-mL volumetric flask and dilute to the mark with absolute methanol. Protect this reagent against preferential evaporation of methanol by keeping it in a polyethylene wash bottle. Prepare fresh daily.

5.13.7 *Methanolic HNO_3 Loading Solution*—Pipet 1 mL of HNO_3 (1 + 1) into a 10-mL volumetric flask and dilute to the mark with absolute methanol. Store as 5.13.6. Prepare fresh daily. High nitrate loading solution is used to ensure absorption of Nd in a tight band and to overcome interference from sulfate and fluoride ions.

5.13.8 *Methanolic HNO_3 Wash Solution*—Pipet 10 mL of HNO_3 (1 + 100) into a 100-mL volumetric flask and dilute to the mark with absolute methanol. Store as 5.13.6. Prepare fresh daily.

5.13.9 *Nitric Acid* (8 *M*, 4 *M*, 3 *M*)[8]—Prepare by diluting Ultrapure[7] concentrated HNO_3 (15.6 *M*) with deionized water.

5.13.10 *Sodium Nitrite Stock Solution*(2 *M*)—Dissolve 3 g of reagent grade sodium nitrite ($NaNO_2$) in 20 mL of 0.1 *M* NaOH.

5.13.11 *Sodium Nitrite Working Solution*—Dilute 100 µL of stock solution from 5.13.10 to 10 mL with 8 *M* HNO_3. Prepare fresh daily.

5.14 *Reagents and Materials for Alternative Procedure B:*

5.14.1 *Eluting Solution (0.094 M HNO_3 in 80 % CH_3OH)*—Prepare 100 mL of 0.47 *M* HNO_3 by diluting 3.00 mL of 15.6 *M* HNO_3 to 100 mL with water in a volumetric flask. Prepare the eluting solution just before use by pipetting 20.0 mL of the 0.47 *M* HNO_3 into a 100-mL volumetric flask and diluting to the mark with anhydrous methanol. The methanol must be free of aldehydes. Absence of a characteristic aldehyde odor is an adequate criterion.

5.14.2 *First Column Resin*—Transfer a water slurry of analytical grade macroporous anion resin (AGMP-1)[8], 50 to 100 mesh, chloride-form resin to a column until the settled height is just below the reservoir. Pass 4 mL of water through, then 6 mL of 12 *M* HCl. Keep the resin wet with 12 *M* HCl until use.

5.14.3 *Hydrochloric Acid (12 M, 0.1 M)*[7]—Using plastic apparatus and an ice bath, bubble filtered HCl gas through quartz-distilled acid until it is saturated. Verify 12 *M* concentration by titration with standard base. Prepare the 0.1 *M* by dilution with quartz-distilled water.

5.14.4 *Hydrofluoric Acid (1 M)*—Dilute 1 mL of concentrated analytical reagent grade HF to 30 mL with quartz-distilled water.

5.14.5 *Hydroiodic Acid-Hydrochloric Acid Mixture (0.1 M HI-12 M HCl)*—Dilute 1 mL of distilled 57 % HI to 74 mL with 12 *M* HCl. Prepare fresh for each use. Store distilled HI in flame-sealed bottles to prevent air oxidation.

5.14.6 *Hydrogen Peroxide (30 %)*—Refrigerate when not in use.

5.14.7 *Ion Exchange Column*—Use commercial disposable polyethylene droppers, 6 mm inside diameter and 60 mm long, with a 2-mL reservoir. Cut off the top of the dropper to form a reservoir and place a glass wool plug in the tip to support the resin bed. The reservoir of the second column can be made cylindrical to accommodate the feeder by inserting as a mold a 1-dram glass vial and heating with a hot air gun. Cool and remove the glass vial mold.

5.14.8 *Loading Solution (1.56 M HNO_3 in 80 % CH_3OH)*—Prepare 100 mL of 7.8 *M* HNO_3 by diluting 50 mL of quartz-distilled HNO_3 to 100 mL with water. Prepare the loading solution by diluting 20 mL of 7.8 *M* HNO_3 to 100 mL with anhydrous methanol. The methanol must be free of aldehydes. The absence of a characteristic aldehyde odor is an adequate criterion.

5.14.9 *Nitric Acid (15.6 M, 2 M, 1 M)*[8]—Dilute quartz-distilled 15.6 *M* HNO_3 with distilled water to prepare the 2 *M* HNO_3 and 1 *M* HNO_3.

5.14.10 *Perchloric Acid (6 M)*—Dilute 12 *M* $HClO_4$ with water.

5.14.11 *Second Column Feeder*—Use polyethylene dispensing bottles (coaxial tip) of about 30-mL capacity.[9] Cut off the delivery tip to a length of about 15 mm.

5.14.12 *Second Column Resin*—Convert AGMP-1[8], 200 to 400 mesh, chloride-form resin to nitrate form. One satisfactory

---

[7] Ultrex and Ultrex II, or equivalent, ultrapure reagent obtained from J. T. Baker Chemical Co., 222 Red School Lane, Phillipsburg, NJ, has been found satisfactory.

[8] Analytical Grade Macroporous Anion Resin, AG MP-1, obtained from Bio-Rad Laboratories, 3300 Regatta Blvd., Richmond, CA, has been found satisfactory.

[9] VWR scientific apparatus Catalog No. 16354-421 or its equivalent has been found satisfactory.

method is to fill a 30-mm diameter by 120-mm high glass column with a water slurry of the resin, then pass 160 mL of HNO_3 (1 + 1) and 160 mL of loading solution (5.2.8) through the column. Verify the absence of chloride by $AgNO_3$ test on the final effluent. Store the resin in loading solution in a closed container until ready for use. When a sample is ready, transfer the resin to a column to a settled height just below the reservoir and keep wet with loading solution until use. Nitrate-form resin is not as chemically stable as chloride-form resin. For this reason it is best not to store nitrate-form resin for longer than a few months.

6. Instrument Calibration

6.1 In the calibration of the mass spectrometer for the analysis of Nd, U, and Pu, the measurement and correction of mass discrimination bias is an important factor in obtaining accurate and consistent results. The mass discrimination bias can be readily measured on natural Nd where the ^{142}Nd-to-^{150}Nd ratio spans over a 5 % spread in mass. The mass discrimination bias factor, B, is constant for Nd, U, and Pu analysis for a given method of scanning (for example, by varying either acceleration voltage or magnetic field) and for a given method of detection (for example, by pulse counting or current integration) on a given detector (for example, electron multiplier, scintillation detector, or d-c collector plate). Calculate B as follows:

$$B = (1/c)\left[\left(\bar{R}_{i/j}/R_s\right) - 1\right] \qquad (10)$$

where:

$\bar{R}_{i/j}$ = average measured atom ratio of isotope i to isotope j. For the most accurate determination of B, let $\bar{R}_{i/j}$ be the average measured atom ratio of ^{142}Nd to ^{150}Nd,

R_s = known value of the measured atom ratio. For the ratio of ^{142}Nd to ^{150}Nd in natural neodymium, $R_s = 4.824$, and

c = Δ mass/mass. The value of c for various ratios and ion species include

Ratio	Nd^+, U^+, or Pu^+	NdO^+, UO_2, or PuO_{2+}
^{148}ND/^{150}Nd	+2/150	+2/166
^{150}Nd/^{148}Nd	−2/148	−2/164
^{142}Nd/^{150}Nd	+8/150	+8/166
^{234}U/^{238}U	+4/238	+4/270
^{235}U/^{238}U	+3/238	+3/270
^{236}U/^{238}U	+2/238	+2/270
^{238}U/^{233}U	−5/233	−5/265
^{233}U/^{238}U	+5/238	+5/270
^{240}Pu/^{239}Pu	−1/239	−1/271
^{241}Pu/^{239}Pu	−2/239	−2/271
^{242}Pu/^{239}Pu	−3/239	−3/271

6.2 Correct every measured average ratio, $\bar{R}_{i/j}$, for mass discrimination as follows:

$$\bar{R}_{i/j} = \bar{R}_{i/j}/(1+cB) \qquad (11)$$

where:

$\bar{R}_{i/j}$ = the corrected average atom ratio of isotope i to isotope j.

7. Procedure

7.1 *Preparation of a Working Dilution of Dissolver Solution:*

7.1.1 Prepare a dilution of fuel dissolver solution with HNO_3 (1 + 1) to obtain a concentration of 100 to 1000 mg of U plus Pu/litre.

7.2 *Separation Procedure A:*

7.2.1 In a 10-mL beaker, place 1000 µl of spike solution (see 5.4) and an aliquot of sample containing about 70 ng of fission product ^{148}Nd. In a second beaker, place a similar aliquot of sample without any spike solution. If the approximate burnup in gigawatt days per metric ton (tonne) is known, the number of milligrams of U plus Pu required for the analysis can be read from Fig. 1. Follow the remaining procedure on each solution.

7.2.2 Add one drop of 1 *M* HF and 2 to 3 drops of concentrated $HClO_4$ to each sample and fume to dryness on a hotplate. Redissolve in 250 µL of 8 *M* HNO_3.

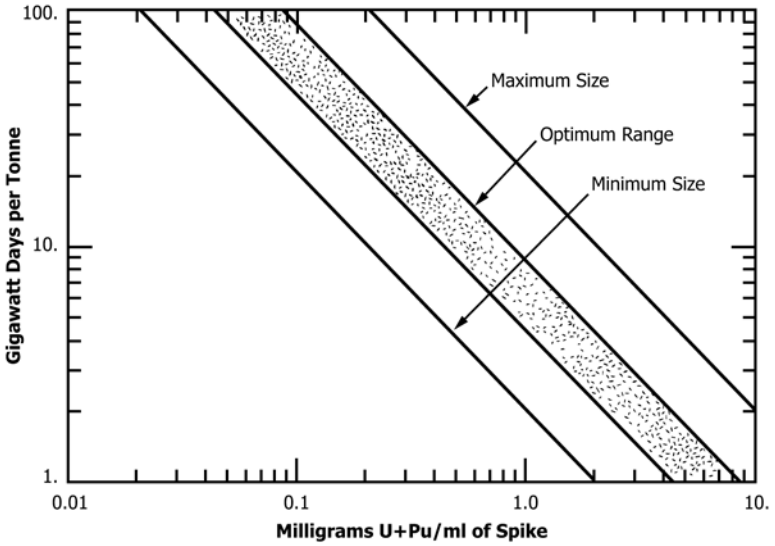

FIG. 1 Sample Size Required for ^{148}Nd Analysis

7.2.3 Add about 0.1 mL of ferrous solution (see 5.5). Mix well and allow to stand at 50 to 60°C for a minimum of 5 min to reduce Pu(VI) to Pu(III) or Pu(IV).

7.2.4 Add 0.5 mL of sodium nitrite working solution (see 5.13.11) to oxidize all Pu to Pu(IV). Evaporate to dryness, then redissolve in 250 µL of 8 *M* HNO_3, taking care to ensure complete dissolution.

7.2.5 Prepare a 1-cm long anion exchange column (see 5.13.4) using Dowex AG 1-X4 resin. Wash the column with 2 mL of 1 *M* HCl followed by 4 mL of 8 *M* HNO_3. Place a clean 10-mL beaker under the column.

7.2.6 Transfer the sample solution from 7.2.4 onto the column with a disposable glass transfer pipet. Rinse the beaker and pipet with 250 µL of 8 *M* HNO_3 and add to the column.

7.2.7 Complete the elution of the neodymium fraction using two 500 µL additions of 8 *M* HNO_3 to the column. Purify the Nd by the procedure given in 7.3.

7.2.8 Place a clean 10-mL beaker under the column. Elute the U using 2 mL of 3 *M* HNO_3. Purify this U solution by the procedure given in 7.4.

7.2.9 Wash the anion exchange column with 10 mL of 3 *M* HNO_3. Discard this wash.

7.2.10 Place a clean 10-mL beaker under the column. Elute Pu with 4 mL of 1 *M* HCl. Purify this Pu solution by the procedure given in 7.5.

7.3 *Nd Purification:*

7.3.1 Evaporate the Nd solution from 7.2.7 to near dryness.

7.3.2 Redissolve sample in 500 µL of 6 *M* HCl.

7.3.3 Prepare a 1-cm long anion exchange column (see 5.13.4) using Dowex AG1-X4 resin. Prepare the column by washing with 2 mL of 6 *M* HCl. Discard wash.

7.3.4 Place a clean 10-mL beaker under the column. Transfer the solution from 7.3.2 onto the column with a disposable glass transfer pipet.

7.3.5 Complete the elution of the Nd using two 500 µL portions of 6 *M* HCl.

7.3.6 Evaporate the solution from 7.3.5 to dryness. Add 250 µL of 8 *M* HNO_3 and again evaporate to dryness. Redissolve the residue in 2 to 3 drops of 8 *M* HNO_3.

7.3.7 Prepare a 2-cm long (Type II) column using nitrated AGMP-1 resin suspended in methanol. (The amount of resin required for column preparation should be equilibrated with methanol overnight before use.)

7.3.8 Wash the 2-cm column with 2 mL of methanolic HNO_3 eluant (1 + 500 HNO_3) followed by 4 mL of methanolic loading solution (1 + 1 HNO_3). (See 5.13.6 and 5.13.7 for preparation of eluant and loading solutions.)

7.3.9 Complete the dissolution of the sample from 7.3.6 using 500 µL of methanolic loading solution (1 + 1 HNO_3). Transfer the sample solution to the column. Rinse the sample beaker with 250 µL of methanolic loading solution and transfer to column. Finally, after all of the sample solution has contacted the resin, rinse down the walls of the column with a few drops of methanolic loading solution.

7.3.10 Add 1 mL of methanolic eluting solution (1 + 500 HNO_3) to elute off rare earths above Eu. Discard effluent.

7.3.11 Place a clean 10-mL beaker under the column. Elute the Nd containing fraction using 4 mL of 4 *M* HNO_3 (non-methanolic).

7.3.12 Evaporate the solution to dryness. Add 250 µL of 8 *M* HNO_3 and again evaporate to dryness. Redissolve in 2 to 3 drops of 8 *M* HNO_3.

7.3.13 Prepare a 6-cm long (Type II) column using nitrated AGMP-1 resin suspended in methanol. (The amount of resin required for column preparation should be equilibrated with methanol overnight before use.)

7.3.14 Wash the 6-cm column with 2 mL of methanolic HNO_3 eluant (1 + 500 HNO_3) followed by 4 mL of methanolic loading solution (1 + 1 HNO_3). (See 5.13.6 and 5.13.7 for preparation of eluant and loading solutions.)

7.3.15 Complete the dissolution of the sample from 7.3.12 using 500 µL of methanolic loading solution (1 + 1 HNO_3). Transfer the sample solution to the column. Rinse the sample beaker with 250 µL of methanolic loading solution and transfer to column. Finally, after all of the sample solution has contacted the resin, rinse down the walls of the column with a few drops of methanolic loading solution.

7.3.16 Wash the 6-cm column with 9 mL of methanolic wash solution (1 + 100 HNO_3). (See 5.13.8 for the preparation of the methanolic wash solution.)

7.3.17 After the 9 mL of methanolic wash solution has passed through the column, add 1.75 mL of methanolic eluting solution (1 + 500 HNO_3) to the column and allow to drain.

7.3.18 Place a clean 10-mL beaker under the column. Elute the purified Nd using 3.25 mL of methanolic eluting solution (1 + 500 HNO_3).

7.3.19 Evaporate the solution to dryness. Add a few drops of 8 *M* HNO_3 and again heat to dryness.

7.3.20 Dissolve the purified Nd in 50 µL of deionized water. Reserve for mass spectrometry.

7.4 *U Purification:*

7.4.1 Evaporate the solution from 7.2.8 to dryness. Add 100 µL of 6 *M* HCl and again evaporate to dryness. Redissolve the sample in 500 µL 6 *M* HCl.

7.4.2 Prepare a 0.5-cm long anion exchange column (see 5.13.4) using Dowex AG1-X4 resin. Prepare the column by washing with 2 mL 0.5 *M* HCl followed by 2 mL 6 *M* HCl.

7.4.3 Transfer sample solution from 7.4.1 onto the column.

7.4.4 Wash the column with 6 mL of 6 *M* HCl. Discard wash solution.

7.4.5 Place a clean 10-mL beaker under the column. Elute the U with 1 to 1.5 mL of 0.5 *M* HCl.

7.4.6 Evaporate the sample solution to dryness. Add several drops of 8 *M* HNO_3 and again evaporate to dryness. Finally, dissolve purified U in enough 0.8 *M* HNO_3 to produce a solution that is 1–2 µg/µL in uranium. Reserve for mass spectrometry.

7.5 *Pu Purification:*

7.5.1 Evaporate the solution from 7.2.10 almost to dryness. Add 500 µL of 8 *M* HNO_3 and again evaporate almost to dryness. Redissolve the sample in 250 µL 8 *M* HNO_3.

7.5.2 Prepare a 0.5-cm long anion exchange column (see 5.13.4) using Dowex AG1-X4 resin. Prepare the column by washing with 2 mL of 1 *M* HCl followed by 4 mL of 8 *M* HNO_3.

7.5.3 Transfer the sample solution from 7.5.1 onto the column.

7.5.4 Rinse the beaker with 250 µL of 8 *M* HNO_3 and transfer rinse to the column.

7.5.5 Wash the column with 10 mL of 3 *M* HNO_3.

7.5.6 Place a clean 10-mL beaker under the column. Elute the purified Pu using 2 mL of 1 *M* HCl.

7.5.7 Evaporate the purified Pu solution to dryness. Add a few drops of 8 *M* HNO_3 and again evaporate to dryness.

7.5.8 Dissolve the purified Pu in enough 0.8 *M* HNO_3 to produce a solution that is 1 to 2 nanograms of Pu/µL (1 to 2 micrograms Pu/mL). Reserve for mass spectrometry.

7.5.9 Proceed to 7.8 (Mass Spectrometry).

7.6 *Alternative Separation Procedure B:*

7.6.1 *Initial Treatment*—In a 10-mL tetrafluoroethylene (TFE) beaker, place 1000 µL of spike solution (see 5.4) and an aliquot of sample containing about 70 ng of fission product ^{148}Nd. In a second beaker, place a similar aliquot of sample without any spike solution. If the approximate burnup in gigawatt days per metric ton (tonne) is known, the number of milligrams of U plus Pu required for the analysis can be read from Fig. 1. Add 10 drops of 1 *M* HF and 10 drops of 6 *M* $HClO_4$ and evaporate just to dryness. Avoid baking the residue, which may make its dissolution difficult.

7.6.2 *Initial Separation of Fission Products, Uranium and Plutonium:*

7.6.2.1 Mark a TFE beaker with the sample identification plus "F.P." (fission product), and place under a first ion exchange column (5.14.2). Glassware may be used, but it is not recommended because of the possibility of contamination of the sample with neodymium from the glassware.

7.6.2.2 Add 0.5 mL of 12 *M* HCl to the residue (7.6.1), agitate to dissolve, and transfer to the column with a new plastic dropper. Add another 0.5 mL of 12 *M* HCl as a rinse, agitate, and transfer to the column with the same dropper. Discard the dropper.

7.6.2.3 Using a new plastic dropper, add 4 mL of 12 *M* HCl to the column reservoir in 1-mL increments, rinsing the reservoir walls with each increment, and wait for the reservoir to empty between increments.

7.6.2.4 Evaporate the F.P. effluent carefully to dryness and reserve for the second column separation of Nd from other fission products (7.7). The strong HCl solution will degas vigorously if overheated at the beginning.

7.6.2.5 Place a clean glass vial, marked with the sample identification plus "Pu fraction" under the column.

7.6.2.6 Pass 2 mL of 0.1 *M* HI-12 *M* HCl through the column. Wait 10 min for the complete reduction to Pu^{+3}, then elute the plutonium with an additional 2 mL of the HI-HCl solution.

7.6.2.7 Reserve the plutonium fraction for mass spectrometry (see 7.8.3).

7.6.2.8 Place a waste container under the column and pass 6 mL of the HI-HCl solution through the column, followed by 0.5 mL of 0.1 *M* HCl. Discard the solution.

7.6.2.9 Place a clean glass vial, marked with the sample identification plus "U fraction," under the column.

7.6.2.10 Elute the uranium with 4 mL of 0.1 *M* HCl. Collect the effluent and discard the column.

7.6.2.11 Reserve the uranium fraction for mass spectrometry (see 7.8.2).

7.7 *Neodymium Separation:*

7.7.1 Add 0.5 mL of 15.6 *M* HNO_3 to the dried fission product residue (see 7.6.2.4) and evaporate to dryness.

7.7.2 Add 3 drops of 30 % H_2O_2 and 1 mL of 1 *M* HNO_3 and again evaporate to dryness.

7.7.3 Add 0.5 mL of loading solution (5.14.8) and agitate the vial to dissolve the residue.

7.7.4 The following operations (7.7.5 – 7.7.10) should be done in a plastic enclosure containing an open vessel of eluting solution to minimize the evaporation effect on the water/methanol ratio of the eluting solution in the column reservoirs.[10]

7.7.5 Place a waste receptacle under a second ion exchange column (5.14.12) and transfer the dissolved residue (7.7.3) to the column with a new plastic dropper.

7.7.6 Repeat 7.7.3 and transfer to the column.

7.7.7 Pass another 2 mL of loading solution through the column.

7.7.8 Rinse the inner walls of the column reservoir with two successive 1-mL portions of eluting solution (5.14.1), waiting for the reservoir to empty before each addition.

7.7.9 Deliver 15 mL of eluting solution from a polyethylene bottle (5.14.11) inverted into the column reservoir. This step elutes most of the americium and rare earths heavier than neodymium. Because of the variations among resin lots, it is advisable to verify the collection of Nd (7.7.10) by using 11-day ^{147}Nd tracer. Adjustment of the eluting volume used in this step is usually sufficient for compensating for lot-to-lot differences. In extreme cases, it may be necessary to change the acidity or methanol/water ratio (**5**).

7.7.10 Place a clean beaker, marked with the sample identification plus "Nd fraction," under the column. Elute the neodymium with 7 mL of eluting solution using the polyethylene bottle feeder.

7.7.11 Evaporate the Nd fraction to dryness and reserve for mass spectrometry (7.8.1).

7.8 *Mass Spectrometry:*

7.8.1 Dissolve the Nd fraction (see 7.7.11) in a small drop of filament mounting solution (see 5.6) transfer and evaporate it onto a single rhenium filament by passing a small electrical current through the filament. Increase the current briefly to char the sucrose from the filament loading solution.

7.8.1.1 For Nd fractions from 7.3.20, load 8 to 10 µL of sample solution and evaporate it onto a double rhenium filament by passing a 1.3 *A* current through the filament. Increase the current to 2.4 *A* over 2 min, then turn off current.

---

[10] Commercial polyethylene glove bag, VWR Scientific Catalog No. 32980-000 has been found to be a satisfactory enclosure.

7.8.1.2 Measure the ^{148}Nd-to-^{150}Nd and the ^{142}Nd-to-^{150}Nd atom ratios for each prepared filament by means of a surface ionization mass spectrometer. Correct each average measured ratio for mass discrimination bias (see 6.2).

7.8.2 Dissolve the U fraction (from 7.6.14 only) in one drop of filament mounting solution (see 5.7) transfer and evaporate it onto a single rhenium filament and char as in 7.8.1.

7.8.2.1 For U fractions from 7.4.6, load 6 µL of sample solution and evaporate it onto a double rhenium filament by passing a 1.3 *A* current through the filament. Increase the current to 2.7 *A* over 3 min, then turn off current.

7.8.2.2 Measure the ^{234}U, ^{235}U, and ^{236}U-to-^{238}U atom ratios ($R_{4/8}$, $R_{5/8}$, and $R_{8/8}$), on each unspiked uranium sample and the ^{238}U-to-^{233}U atom ratio, $M_{8/3}$, on each spiked U sample by means of a surface ionization mass spectrometer. Correct each average measured ratio for mass discrimination (see 6.2).

7.8.3 Dissolve the Pu fraction (from 7.6.2.7) in one drop of HCl (1 + 24) transfer and evaporate it onto a single rhenium filament. Evaporate one drop of filament mounting solution (see 5.7) over the sample and char as in 7.8.1.

7.8.3.1 For Pu fractions from 7.5.8, load 4 to 6 µL of sample solution and evaporate it onto a double rhenium filament by passing a 1.3 *A* current through the filament. Increase the current to 2.6 *A* over 3 min, then turn off current.

7.8.3.2 Measure the ^{240}Pu, ^{241}Pu, and ^{242}Pu-to-^{239}Pu atom ratio ($R_{0/9}$, $R_{1/9}$, and $R_{2/9}$) on each unspiked Pu sample and the ^{239}Pu-to-^{242}Pu atom ratio, $M_{9/2}$, on each spiked Pu sample by means of a surface ionization spectrometer. Correct each average measurement ratio for mass discrimination (see 6.2).

8. Calculation

8.1 Calculate the ratio of effective fission yields of ^{150}Nd to ^{148}Nd, $E_{50/48}$ as follows:

$$E_{50/48} = \left[R_{50/48}\left(R_{50/42} - C_{50/42}\right)\right] / \left[R_{50/42} - R_{50/48}\left(C_{48/42}\right)\right] \quad (12)$$

where:

$R_{50/48}$, $R_{50/42}$ = atom ratio of ^{150}Nd-to-^{148}Nd and ^{150}Nd-to-$_{142}$Nd in the unspiked sample, corrected for mass discrimination bias, and

$C_{50/42}$, $C_{48/42}$ = atom ratios of ^{150}Nd-to-^{142}Nd and ^{148}Nd-to-^{142}Nd in natural Nd contamination.

8.2 Calculate constants *a, b, c, d, e,* and *f* as follows:

$$a = C_{42/50} - S_{42/50} \quad (13)$$

$$b = C_{48/50} - S_{48/50} \quad (14)$$

$$c = C_{42/50}S_{48/50} - S_{42/50}C_{48/50} \quad (15)$$

$$d = C_{42/50} \quad (16)$$

$$e = E_{50/48}C_{42/50} \quad (17)$$

$$f = \left(1 - E_{50/48}C_{48/50}\right) \quad (18)$$

where:

$C_{2/50}$, $C_{48/50}$ = atom ratio of ^{142}Nd and ^{148}Nd-to-^{150}Nd in natural Nd contamination, which are 4.824 and 1.0195, respectively, and

$S_{42/50}$, $S_{48/50}$ = atom ratio of ^{142}Nd and ^{148}Nd-to-^{150}Nd respectively in the spike solution.

8.3 Calculate $M'_{48/50}$ as follows:

$$M'_{48/50} = K\left(aM_{48/50} - bM_{42/50} - c\right) / \left(d - eM_{48/50} - fM_{42/50}\right) \quad (19)$$

where:

K = factor to correct for a trace of non-fission-caused ^{148}Nd from thermal neutron capture on ^{147}Nd, found in Table 1. K is assumed to be unity for fast reactors.

$M'_{48/50}$ = atom ratio of fission product ^{148}Nd-to-spike ^{150}Nd adjusted for fission product ^{150}Nd, ^{148}Nd spike impurity, and ^{148}Nd and ^{150}Nd from natural Nd contamination, and

$M_{48/50}$, $M_{42/50}$ = measured atom ratio of ^{148}Nd-to-^{150}Nd and ^{142}Nd-to-^{150}Nd of the sample plus spike mixture corrected for mass discrimination bias (see 6.2).

8.4 Calculate the number of fissions per sample, *F'*, as follows:

$$F' = \left(A_{50}/E_{48}\right)M'_{48/50} \quad (20)$$

where:

E_{48} = effective fractional fission yield of ^{148}Nd calculated from the fission yields of ^{148}Nd for each of the fissioning isotopes weighted according to their contribution to fission as measured in Test Method E244.

The fractional yield for ^{148}Nd in thermal fission of ^{235}U, ^{239}Pu, and ^{241}Pu is 0.0167312 ± 0.35 %, 0.016422 ± 0.5 %, and 0.0193209± 0.7 %, respectively; and for fast fission of ^{238}U is 0.0209416 ± 1.0 % **(6)**, and

A_{50} = the number of atoms of ^{150}Nd/mL of spike (see 5.4.3).

8.5 Calculate the atom fraction ^{238}U in the unspiked U sample, A_8, as follows:

$$A_8 = R_{8/8} / \left(R_{4/8} + R_{5/8} + R_{6/8} + R_{8/8}\right) \quad (21)$$

where $R_{8/8}$ (which equals 1) is retained for clarity.

8.6 Calculate $S_{8/3}$ from $S_{3/8}$ (see 5.4.2) as follows:

$$S_{8/3} = 1/S_{3/8} \quad (22)$$

8.7 Calculate the total U atoms per sample, *U'*, from A_{33} (see 5.4.4):

$$U' = \left(A_{33}/A_8\right)\left\{\left(M_{8/3} - S_{8/3}\right) / \left[1 - \left(M_{8/3}/R_{8/3}\right)\right]\right\} \quad (23)$$

8.8 Calculate the atom fraction $_{239}$Pu in the unspiked Pu sample, A_9, as follows:

$$A_9 = R_{9/9} / \left(R_{9/9} + R_{0/9} + R_{1/9} + R_{2/9}\right) \quad (24)$$

where $R_{9/9}$ (which equals 1) is retained for clarity.

8.9 Calculate $S_{9/2}$ from $S_{2/9}$ (see 5.4.2) and $R_{9/2}$ from $R_{2/9}$ as follows:

$$S_{9/2} = 1/S_{2/9} \quad (25)$$

$$R_{9/2} = 1/R_{2/9} \quad (26)$$

8.10 Calculate the total Pu atoms per sample, *Pu',* from A_{42} (see 5.4.5):

$$Pu' = A_{42}/A_9\{(M_{9/2} - S_{9/2})/[1 - (M_{9/2}/R_{9/2})]\} \quad (27)$$

8.11 Calculate the total heavy element atom percent fission, F_T, from

$$F_T = [F'/(U' + Pu' + F')] \times 100 \quad (28)$$

8.12 If desired, calculate the gigawatt days per metric ton from

$$\text{gigawatt days per metric ton} = F_T \times (9.6 \pm 0.3) \quad (29)$$

9. Precision and Bias

9.1 *Precision*—The single laboratory precisions for the average of duplicate determinations of F', U', and F_T are given in Table 2 in percent relative standard deviation. The percent relative standard deviation is the estimated standard deviation of a single laboratory times 100, divided by the average of all laboratories. The corresponding precisions among laboratories are also given in Table 2 for the participating laboratories.

TABLE 2 Precision of Analyses

Value Measured	Single-Instrument Precision (1 σ), relative %	Multilaboratory Precision (1σ), relative %
F	0.8	0.9
U	0.5	0.8
F_T	0.9	1.2

NOTE 1—The precision estimates for F' are based on an interlaboratory comparison with 8 participating laboratories which analyzed 2 samples of irradiated U fuel with 12.0 and 10.6 GWd/ton burnup in duplicate on each of 2 days. Precision estimates for U' are based on a similar comparison with 5 participating laboratories which analyzed 4 samples, including the same 2 irradiated-fuel solutions plus 2 natural U solutions in duplicate on each of 2 days. The precision estimates for atom percent fission, F_T, are computed from the precision estimates of F' and U'. Practice E180 was used in developing these precision estimates. It should be noted that most values in these studies were read from strip chart recorders. It has been reported (**7**) that the most important random error in isotopic analysis is due to the strip chart recorder, although recognition of this fact is not widespread.

9.2 *Bias (Accuracy or Systematic Error)*—In mass spectrometry, the presence of a bias is possible, but mass spectrometers can be calibrated so that mass discrimination bias is eliminated. To accomplish this, measured mass ratios shall be bias corrected according to Section 6. It is expected that the method so calibrated will be free of bias and that the accuracy can be taken to be equal to the precision (see Section 9) except for some additional uncertainty in the fractional fission yield of ^{148}Nd.

10. Keywords

10.1 atom percent fission; neodymium-148; plutonium fuel; uranium fuel

REFERENCES

(**1**) Fudge, A. J., Wood, A. J., and Banham, M. F., "The Determination of Burnup in Nuclear Fuel Test Specimens Using Stable Fission Product Isotopes and Isotopic Dilution." *USAEC Doc.*, *TID-7629*, 1961, pp. 152–165.

(**2**) Rider, B. F., Peterson, Jr., J. P., and Ruiz, C. P., "Determination of Neodymium-148 in Irradiated UO_2 as a Measurement of Burnup," *Transactions of the American Nuclear Society*, Vol. 7, No. 2, 1964, p. 350.

(**3**) Rider, B. F., et al, "Accurate Nuclear Fuel Burnup Analysis XXI," *USAEC Doc.*, *GEAP-5462*, 1967.

(**4**) Rider, B. F., et al, "Accurate Nuclear Fuel Burnup Analysis XII," *USAEC Doc.*, *GEAP-4776*, 1964.

(**5**) Marsh, S. F., et al, "Improved Two-Column Ion Exchange Separation of Plutonium, Uranium, and Neodymium in Mixed Uranium-Plutonium Fuels for Burnup Measurement," *USAEC Doc. LA-5568*, June 1975.

(**6**) England, T. R., and Rider, B. F., "ENDF—349 Evaluation and Compilation of Fission Product Yields: 1993," Los Alamos National Laboratory, Los Alamos, NM, Report LA-UR-94-3106, October 1994.

(**7**) Shields, W. R., " Analytical Mass Spectrometry Section: Instrumentation and Procedures for Isotopic Analysis," *NBS Technical Note 277*, National Institute of Standards and Technology, Washington, DC, 1966, p. 16.

(**8**) Heck, V. D., et al, "Neutron Capture Cross Section of ^{147}Nd," Atomkernenergie, Vol 24, 1974, p. 141.

(**9**) Maeck, W. J., Larson, R. P., and Rein, J. E., "Burnup Determination for Fast Reactor Fuels: A Review and Status of the Nuclear Data and Analytical Chemistry Methodology Requirements," USAEC Document: TID-26209, February 1973.