



Standard Practice for Alternate Actinide Calibration for Inductively Coupled Plasma-Mass Spectrometry¹

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1. Scope

1.1 This standard practice provides guidance for an alternate linear calibration for the determination of selected actinide isotopes in appropriately prepared aqueous solutions by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). This alternate calibration is mass bias adjusted using thorium-232 (^{232}Th) and uranium-238 (^{238}U) standards. One of the benefits of this standard practice is the ability to calibrate for the analysis of highly radioactive actinides using calibration standards at much lower specific activities. Environmental laboratories may find this standard practice useful if facilities are not available to handle the highly radioactive standards of the individual actinides of interest.

1.2 The instrument response for a series of determinations of known concentration of ^{232}Th and ^{238}U defines the mass versus response relationship. For each standard concentration, the slope of the line defined by ^{232}Th and ^{238}U is used to derive linear calibration curves for each mass of interest using interference equations. The mass bias corrected calibration curves, although generated from interference equations, are specific to the instrument operating parameters and tuning in effect at the time of data acquisition. Because interference equations are part of the normal ICP-MS manufacturer's software package, this calibration methodology is widely applicable.

1.3 For this standard practice, the actinide atomic mass range that has been studied is from amu 232–244. Guidance for an extended range of amu 228–248 is given in this practice.

1.4 Using this practice, analyte concentrations are reported at each amu and not by element total (that is, ^{239}Pu versus plutonium).

1.5 *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.*

¹ This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Test Methods—Plasma.

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2. Referenced Documents

2.1 ASTM Standards:²

- C 859 Terminology Relating to Nuclear Materials
- C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis
- C 1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis
- C 1411 Practice for the Ion Exchange Separation of Uranium and Plutonium Prior to Isotopic Analysis
- C 1414 Practice for the Separation of Americium and Plutonium by Ion Exchange
- C 1463 Practices for Dissolving Glass Containing Radioactive and Mixed Waste for Chemical and Radiochemical Analysis
- D 1193 Specification for Reagent Water

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms relating to nuclear materials, refer to Terminology C 859.

3.1.2 *AMU*—atomic mass unit.

4. Summary of Practice

4.1 Calibration for the actinides by ICP-MS can be performed in a variety of ways with varying degrees of data quality. An alternative calibration method has been developed to compensate for instrument mass bias using a generated mass response curve defined by the ^{232}Th and ^{238}U data points. The mass response curve defined by ^{232}Th and ^{238}U approximates the mass response curve from amu 232–244 as verified experimentally and graphically depicted in Fig. 1. The mass response curve shown reflects the operating parameters and tune of the particular instrument in use at the time of data collection. Different tuning parameters or instrumentation could result in varying degrees of negative, neutral, or positive mass bias. Because the mass response curve defined by ^{232}Th

² 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.

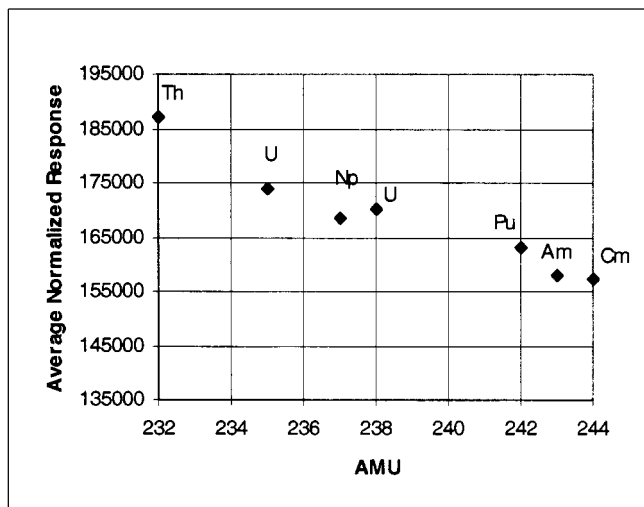


FIG. 1 Atomic Mass versus Average Normalized Response

and ^{238}U used in this standard practice is determined during each calibration, all potential linear variations in mass bias are compensated for.

4.2 The alternative calibration in this standard practice combines the features of an external linear calibration at each mass of interest with the mass bias correction of a mass/response curve. The correction for mass bias is integrated into the acquisition of the standard data through the use of interference equations which, are part of the normal software package for correction of isobaric interference's in ICP-MS analyses. Multipoint calibration curves are generated at each mass of interest, resulting in more accurate quantification than that of the typical "semi-quantitative" single point calibration based on the mass/response curve alone.

4.3 Sample analyses for blanks and samples are performed using a data acquisition method file without the interference equations that were used to derive the calibration curves. After calibration curves are generated, the calibration files are copied or linked to the analytical acquisition method. The sample responses are acquired at each mass, and concentrations calculated from the mass bias corrected calibration curves. Some ICP-MS vendor supplied control software will permit the linking of separate calibration and acquisition files (that is, you can choose which calibration files to use to quantitate any particular data set regardless of the acquisition file that was used to acquire the data).

4.4 Mixed calibration standard solutions are prepared through dilution of single element stock standards of thorium and known abundance uranium (normally depleted in ^{235}U) with dilute nitric acid to develop a calibration series covering the desired concentration range. Standard concentrations are calculated for ^{232}Th and ^{238}U for each calibration solution.

4.5 Bismuth-209 (^{209}Bi) is used as an internal standard and is added in a fixed quantity in all standards and samples to correct for both instrument drift and physical sample transport fluctuations.

5. Significance and Use

5.1 One of the benefits of this standard practice is the ability to calibrate for the analysis of highly radioactive actinides using calibration standards at much lower specific activities (that is, ^{232}Th and ^{238}U). Environmental laboratories may find this standard practice useful if facilities are not available to handle the highly radioactive standards of the individual actinides of interest.

5.2 The degree of actual mass bias is variable and is dependent upon instrument tune parameters. This standard practice uses universal interference equations to derive a mass bias correction that is specific to the instrument parameters and tune used for sample data acquisition and not based on a historical average.

5.3 Mass bias correction uses the instrument software interference equations and does not require additional subsequent off-line calculations.

5.4 The methodology that this standard practice is based on has been used for the determination of ^{232}Th and ^{237}Np in enriched uranium solutions and the determination of ^{241}Am in plutonium and uranium legacy oxides following dissolution and ion extraction chromatography separation.

6. Interferences

6.1 Isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z) may cause isobaric interferences in ICP-MS if present in sufficient quantity (that is, ^{238}U with ^{238}Pu and ^{241}Pu with ^{241}Am). Because the isotopic abundance of actinides vary widely, it is not practical to apply an interference correction unless the isotopic abundance of the interference is well characterized. In addition, the hydride of an abundant isotope can interfere with the adjacent higher mass (that is, $^{238}\text{U}^1\text{H}$ on ^{239}Pu). For these reasons, it is prudent to implement actinide separation methods utilizing extraction chromatography resins prior to ICP-MS analysis to significantly reduce these interferences.³

6.2 Analyte memory can occur when there are large concentration differences between standards and/or samples that are analyzed sequentially. Thorium can exhibit memory within the sample introduction system. A rinse solution containing 0.2M Nitric acid and 0.2M Sulfuric acid has been found to be beneficial in reducing thorium carryover.

7. Apparatus

7.1 *ICP-MS*, computer controlled with associated software and peripherals.

7.2 *Autosampler*, optional, with tube racks, disposable plastic sample tubes.

7.3 *Variable Micro and Macro Pipettes*.

8. Reagents

8.1 *Argon (Ar) Gas*, high purity $\geq 99.99\%$.

8.2 *Deionized Water*, high purity, conforming to Specification D 1193, Type I.

³ Maxwell, S. L., "Rapid Actinide-Separation Methods," *Journal of Applied Radioactivity Measurements*, Vol 8, No. 4, 1997, pp. 36-44.

TABLE 1 Universal Interference Equations Used to Perform Calibration Mass Bias Correction

Mass Bias Corrected Calibration Response	Calibration Interference Equation
Corrected (228) =	Response (228) × 0 – (238) × 0.6667 + (232) × 1.6667
Corrected (229) =	Response (229) × 0 – (238) × 0.5 + (232) × 1.5
Corrected (230) =	Response (230) × 0 – (238) × 0.3333 + (232) × 1.3333
Corrected (231) =	Response (231) × 0 – (238) × 0.1667 + (232) × 1.1667
Corrected (232) =	Response (232) × 1
Corrected (233) =	Response (233) × 0 + (238) × 0.1667 + (232) × 0.8333
Corrected (234) =	Response (234) × 0 + (238) × 0.3333 + (232) × 0.6667
Corrected (235) =	Response (235) × 0 + (238) × 0.5 + (232) × 0.5
Corrected (236) =	Response (236) × 0 + (238) × 0.6667 + (232) × 0.3333
Corrected (237) =	Response (237) × 0 + (238) × 0.8333 + (232) × 0.1667
Corrected (238) =	Response (238) × 1
Corrected (239) =	Response (239) × 0 + (238) × 1.1667 – (232) × 0.1667
Corrected (240) =	Response (240) × 0 + (238) × 1.3333 – (232) × 0.3333
Corrected (241) =	Response (241) × 0 + (238) × 1.5 – (232) × 0.5
Corrected (242) =	Response (242) × 0 + (238) × 1.6667 – (232) × 0.6667
Corrected (243) =	Response (243) × 0 + (238) × 1.8333 – (232) × 0.8333
Corrected (244) =	Response (244) × 0 + (238) × 2 – (232) × 1
Corrected (245) =	Response (245) × 0 + (238) × 2.1667 – (232) × 1.1667
Corrected (246) =	Response (246) × 0 + (238) × 2.3333 – (232) × 1.3333
Corrected (247) =	Response (247) × 0 + (238) × 2.5 – (232) × 1.5
Corrected (248) =	Response (248) × 0 + (238) × 2.6667 – (232) × 1.6667

8.3 *Nitric Acid*, (specific gravity 1.42), concentrated nitric acid (HNO₃), trace metal grade or better.

8.4 *Sulfuric Acid*, (specific gravity 1.84), concentrated sulfuric acid (H₂SO₄), trace metal grade or better.

8.5 *Bismuth Stock Solution*, (1000 µg/mL Bi), matrix nominal 10 % HNO₃.

8.6 *Thorium Stock Solution*, (1000 µg/mL Th), matrix nominal 2 % HNO₃.

8.7 *Uranium Stock Solution*, (1000 µg/mL U), matrix nominal 2 % HNO₃.

8.8 *Radioisotope Standards* (²⁴¹Am, ²⁴²Pu, ²⁴⁴Cm, etc.) can be purchased⁴ to verify calibration curves if laboratory and ICP-MS have proper engineering and procedural controls to safely handle radiological material.

9. Hazards

9.1 Personnel using this procedure shall be knowledgeable of the safety precautions necessary for normal chemical, radiological handling protocol and instrumental operation of ICP-MS.

9.2 Nitric and sulfuric acids are strong oxidizers, avoid contact with flammable, powdered, or combustible materials. Avoid contact with skin, eyes and clothing. Do not breathe or ingest vapors.

9.3 Actinide bearing materials are radioactive and toxic. Adequate laboratory facilities and ventilation hoods along with safe handling techniques must be used. A detailed discussion of all safety precautions needed is beyond the scope of this standard practice. Follow site and facility specific radiation protection and chemical hygiene protocol.

10. Procedure

10.1 *Calibration Standard Preparation*—Because the focus of this practice is on mass bias correction and not on any

particular calibration concentration range or sample matrix, minimal instruction is given for the preparation of calibration standards.

10.1.1 *Mixed Calibration Standard* solutions are prepared through the quantitative dilution of single element bench stock standards of thorium and known abundance uranium (normally depleted in ²³⁵U) with bismuth as an internal standard in nominal 1 % nitric acid or other acid concentration appropriate to match sample matrix.

10.1.2 *Calibration Blank* consists of the same acid matrix as the standard solutions with the same concentration bismuth internal standard.

10.1.3 *Reagent Blank* consists of the same acid and chemical matrix as the samples (if different from the calibration blank) with the same concentration bismuth internal standard. Consideration should be given to processing the reagent blank through any sample prep evolutions such as digestion or ion extraction chromatography separation.

10.2 Sample Preparation:

10.2.1 Prior to analysis, digest/dissolve samples as needed using methods appropriate to the sample matrix such as Practices C 1168, C 1347, C 1463, or other laboratory specific procedures.

10.2.2 Use actinide separation procedures when necessary to reduce matrix and isobaric interferences between overlapping isotopes of interest in the digest solutions; such as Practices C 1411 and C 1414, Maxwell's "Rapid Actinide-Separation Methods,"³ or other laboratory specific procedures.

10.2.3 Dilute sample into appropriate acid matrix (1 to 2 % HNO₃ typical) with the same concentration internal standard as in the calibration standards. Dilution of samples should be consistent with the span of calibration standards.

10.3 ICP-MS Instrumental Analysis:

10.3.1 Set up the ICP-MS for the analysis using the parameters given in the manufacturer's operating manual. Following plasma initiation, allow the instrument to reach thermal equilibrium (generally at least 30 min). Optimize the ICP-MS using routine tuning protocol for elemental analysis or tune specific

⁴ Isotope Products Laboratories, 3017 N. San Fernando Blvd., Burbank, CA 91504; (818) 843-7000.

TABLE 2 SRS ²³⁷Np Spike Recovery from HEU Solutions

Date	1 U g/L	2 Np-237 Spk Known' mg	3 Np-237 Smpl w/Spk. mg/mL	4 Np-237 Smpl Avg. µg/mL	5 Np-237 Spk % Rec.
01/24/00	10.2	3.044	3.047	0.069	97.9
01/24/00	9.0	3.044	2.959	0.065	95.1
06/22/00	7.7	3.044	2.928	0.092	93.1
10/05/00	2.2	3.044	4.483	1.188	108.3
10/05/00	4.5	3.044	3.472	0.173	108.4
10/05/00	4.3	3.044	3.383	0.171	105.5
10/05/00	9.3	3.044	3.722	0.369	110.1
02/15/01	2.3	3.044	4.489	1.485	98.7
02/15/01	6.4	3.044	3.403	0.188	105.6
02/15/01	6.1	3.044	3.294	0.155	103.1
02/15/01	9.4	3.044	3.403	0.220	104.6
06/19/01	9.4	3.044	3.285	0.173	102.2
09/13/01	5.5	3.061	2.909	0.031	94.0
09/13/01	5.5	3.061	2.946	0.012	95.8
09/13/01	8.0	3.061	3.176	0.117	99.9
09/26/01	7.9	3.061	3.139	0.122	98.6
10/15/01	5.3	3.061	3.064	0.006	99.9
10/15/01	5.3	3.061	2.864	0.006	93.4
01/04/02	5.2	3.061	3.079	0.091	97.6
01/04/02	5.3	3.061	4.544	1.367	103.8
02/19/02	9.8	3.061	3.200	0.137	100.0
02/25/02	5.5	3.061	4.498	1.383	101.8
02/25/02	5.3	3.061	3.250	0.107	102.7
04/03/02	5.9	3.061	3.269	0.091	103.8
04/03/02	5.9	3.061	3.217	0.105	101.7
04/09/02	NA	3.061	3.090	0.000	100.9
04/09/02	5.5	3.061	4.605	1.322	107.2
04/09/02	NA	3.061	3.255	0.000	106.3
04/09/02	5.3	3.061	3.445	0.084	109.8
04/16/02	4.5	3.061	4.776	1.483	107.6
05/10/02	8.4	3.061	3.063	0.131	95.8
05/30/02	7.3	3.061	2.755	0.161	84.7
05/30/02	5.6	3.061	3.910	1.161	89.8
06/11/02	4.9	3.061	3.236	0.051	104.1
07/01/02	6.2	3.061	3.443	0.143	107.8
07/18/02	4.8	3.061	3.316	0.222	101.1
09/03/02	6.5	3.061	3.248	0.242	98.2
n					37
Avg.					101.0
Std. Dev.					5.8
Bias					1.0

to the mass range of interest. Limit oxide formation through instrument tuning. Oxides are typically monitored using the % ratio of CeO/Ce, usually $\leq 1\%$.

10.4 Actinide Calibration w/Mass Bias Correction:

10.4.1 Actinide calibration with mass bias correction is performed through the use of external linear calibration standards consisting of ²³²Th and ²³⁸U with ²⁰⁹Pb as an internal standard. Multi-point calibration curves are generated for each mass of interest between 228 and 248 AMU using the interference equations in Table 1 to interpolate or extrapolate each mass response relative to its position on the mass response curve as defined by the ²³²Th and ²³⁸U standards.

10.4.2 After the calibration curves are generated, the calibration files are copied or linked to the analytical method.

10.4.3 Some ICP-MS vendor⁵ supplied control software will permit the linking of separate calibration and acquisition

files (that is, you can choose which calibration files to use to quantitate any particular data set).

10.5 Sample Data Acquisition:

10.5.1 Analysis of blanks, samples, and QC checks are performed using a data acquisition method file without the interference equations that were used to derive the calibration curves. The analyte responses are acquired at each mass and concentrations calculated from the mass bias compensated calibration curves.

11. Calculations

11.1 Derivation of Calibration "Interference Equations:"

11.1.1 As discussed earlier, the mass response curve defined by ²³²Th and ²³⁸U is representative of the mass response curve over the mass range of 232–244 as demonstrated in Fig. 1. The correction for mass bias is integrated into the acquisition of the calibration data set through the use of interference equations. User definable interference equations are part of the normal software package and are routinely intended for correction of isobaric mass overlap in ICP-MS.

⁵ Agilent Technologies, ChemStation operating software, windows NT version has been found to be acceptable.

TABLE 3 SRS ²⁴¹Am Analysis of Legacy Material Pu Oxide and Scrap

Date	Am-241 QC % Recovery
08/01/00	94.3
12/06/00	93.8
12/18/00	98.5
01/10/01	105.3
02/07/01	106.0
04/10/01	100.3
04/10/01	103.4
05/15/01	103.5
07/26/01	110.9
09/11/01	103.0
10/08/01	92.9
10/25/01	96.3
12/12/01	95.4
12/27/01	93.3
01/08/02	101.1
03/21/02	97.7
04/03/02	110.0
07/16/02	103.4
08/07/02	106.1
09/03/02	102.5
09/11/02	90.6
09/20/02	99.6
10/07/02	97.2
10/10/02	95.7
10/14/02	102.2
10/23/02	103.2
n	26
Avg.	100.2
Std. Dev.	5.3

11.1.2 In this practice, the interference equations are used to correct analytical results based on a close approximation of the slope of the mass versus response curve (mass bias). Multi-point calibration curves are generated for each mass (amu 228–248) using the interference equations to either interpolate or extrapolate based on their relative amu position on the mass response curve.

11.1.3 The Calibration “Mass Bias Correction” line equation for each mass (other than amu 232 and 238) is defined as:

$$Y_{(amu)} = [R_{(amu)} * 0] + B + XM \quad (1)$$

where:

Y_{amu} = the calibration mass bias corrected response at a given mass,

R_{amu} = response during calibration at specified amu. The amu response is multiplied by zero to make the equations the most flexible for a variety of calibration conditions. A sample blank must be subtracted from the final data set to compensate for any significant background response,

NOTE 1—Alternatively, if desired, $R_{(amu)}$ can be multiplied by 1 (preserving background), but the calibration interference equations should be modified to account for significant contributions of other non-calibration isotopes (that is, ²³⁵U at 0.72 % for natural abundance uranium). See example in 11.1.5.

$B = R_{(238)}$; the y-axis intercept point for the mass response curve with the y-axis arbitrarily placed at amu 238,
 X = number of amu subject mass is from amu 238,

Example(1): $X_{(243)} = 243 - 238 = +5$

Example(2): $X_{(234)} = 234 - 238 = -4$

M = slope of the mass response curve.

$$M = [\text{Response}_{(238)} - \text{Response}_{(232)}] / 6$$

11.1.4 Example for amu 243:

$$Y_{(243)} = [R_{(243)} * 0] + R_{(238)} + 5([R_{(238)} - R_{(232)}] / 6)$$

$$Y_{(243)} = [R_{(243)} * 0] + R_{(238)} + 5/6[R_{(238)}] - 5/6[R_{(232)}]$$

$$Y_{(243)} = [R_{(243)} * 0] + [R_{(238)} * 1.8333] - [R_{(232)} * 0.8333]$$

As shown in Table 1:

$$\text{Corrected (243)} =$$

$$\text{Response (243)} \times 0 + (238) \times 1.8333 - (232) \times 0.8333$$

11.1.5 Example for amu 235 when natural uranium used to calibrate:

$$\text{Corrected (235)} =$$

$$\text{Response (235)} \times 1 + (238) \times 0.5 + (232) \times 0.5 - (238) \times 0.007$$

12. Precision and Bias

12.1 Data Collection Summary:

12.1.1 The analytical data presented in Tables 2 and 3 in the appendix give an indication of the possible precision and bias when using this standard practice.

12.1.2 The QC solutions (²³⁷Np and ²⁴¹Am) used to generate the data in Tables 2 and 3 were prepared and “standardized” in-house and therefore, do not have accepted reference values. The ²³⁷Np spike was prepared in-house from a ²³⁷Np stock solution whose “known” value was previously determined by controlled potential coulometry. Secondary dilution of this stock was by weight. The stated (%)RSD is 0.685 % for the secondary dilution ²³⁷Np stock.

12.1.3 For the ²³⁷Np spike recovery data in Table 2, neptunium was separated from high enriched uranium (HEU) matrix in duplicate prior to analysis using ion extraction chromatography columns per footnote 3. A third aliquot was spiked (by pipette volume) with ²³⁷Np prior to column separation.

12.1.4 Table 2 of the appendix shows the relative spike recovery of ²³⁷Np “known” spiked into high-enriched uranium samples. Column 1 is the U g/L prior to ion exchange to remove the majority of uranium matrix. Column 2 is the ²³⁷Np spike concentration prior to ion exchange. Column 3 is the sample with ²³⁷Np spike post ion exchange. Column 4 is the average of sample duplicates with no spike post ion exchange. Column 5 is the % recovery for the spiked sample ((Col. 3 – Col. 4)/Col. 2) × 100.

12.1.5 For the ²⁴¹Am precision data in Table 3, sample preparation consisted of closed vessel microwave dissolution of plutonium oxide followed by americium separation from the plutonium matrix using ion exchange chromatography per footnote 3.

12.1.6 The ²⁴¹Am QC was prepared in-house from composite ²⁴¹Am samples prepared as previously described. The ²⁴¹Am QC composite was assigned a “known” value determined from the average of 10 analyses using this standard practice. ²⁴¹Am QC composite was assigned a value of 113.5 µg/L as prepared for instrument analysis.

12.1.7 To validate the ²⁴¹Am QC composite, a ²⁴¹Am SRM was also analyzed at the same time. The ²⁴¹Am SRM was

obtained from Isotope Products®, catalogue no. 7241. The ^{241}Am SRM was made up in triplicate with each analyzed 4 times for a total of 12 determinations. The calculated (%)RSD was 1.15 %. The mean relative percent error was -3.5 %.

12.1.8 The results were obtained with an HP4500 quadrupole ICP-MS using 1 second per mass integration with 6 repetitions for calibration acquisition and 3 repetitions for sample acquisition. Bismuth used as an internal standard.

12.1.9 The results were collected from a single instrument under normal operating conditions from the work of five analysts.

12.2 Precision:

12.2.1 The ^{237}Np spike recovery data (Table 2, col. 5) gives an indication of precision with a %RSD of 5.8 where the nominal spiked concentration is 3 $\mu\text{g/mL}$. This data was collected over a period of 33 months for a total of 37 data points.

12.2.2 The ^{241}Am recovery data (Table 3), gives an indication of precision with a %RSD of 5.3 where the nominal

concentration is 100 $\mu\text{g/L}$. This data was collected over a period of 26 months for a total of 26 data points. The average recovery was 100.2 %.

12.3 Bias:

12.3.1 The ^{237}Np spike recovery average was 101.0 % (Table 2, col. 5). The ^{241}Am recovery average was 100.2 % (Table 3). The slight biases observed in the % recovery data of Tables 2 and 3 are not statistically significant.

12.3.2 The analyses presented in Table 2 can provide some indication of the relative biases possible when using this standard practice. The average percent recovery (%Rec.) data suggest that the bias indicated over time may be minimal relative to the precision indicated. The data is provided to allow interested and knowledgeable readers to draw their own conclusions as to the applicability of the standard practice to their own needs and circumstances.

13. Keywords

13.1 actinides; inductively coupled plasma-mass spectrometry (ICP-MS); mass bias; thorium; uranium

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