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Standard Test Method for
Isotopic Abundance Analysis of Uranium Hexafluoride and Uranyl Nitrate Solutions by Multi-Collector, Inductively Coupled Plasma-Mass Spectrometry[1]

This standard is issued under the fixed designation C 1477; 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 isotopic abundance analysis of ^{234}U, ^{235}U, ^{236}U and ^{238}U in samples of hydrolysed uranium hexafluoride (UF_6) by inductively coupled plasma source, multi-collector, mass spectrometry (ICP-MC-MS). The method applies to material with ^{235}U abundance in the range of 0.2 to 6 % mass. This test method is also described in ASTM STP 1344.

~~1.2~~

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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:* [2]

C 761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride

C 787 Specification for Uranium Hexafluoride for Enrichment

C 996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ^{235}U

D 1193 Specification for Reagent Water

2.2 *Other Document:*

STP 1344 Applications of Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) to Radionuclide Determinations [3]

3. ~~Summary of Test Method~~ Terminology

~~3.1Samples are received either in the form of uranium hexafluoride (UF_6~~

3.1 *Acronyms:*

3.1.1 *amu*—atomic mass unit

3.1.2 *ICP-MC-MS*—Inductively Coupled Plasma Multi-Collector Mass Spectrometer

3.1.3 *ICP-MS*—Inductively Coupled Plasma Mass Spectrometer

3.1.4 *UIRM*—Uranium Isotopic Reference Material

4. Summary of Test Method

4.1 Samples are received either in the form of uranium hexafluoride (UF_6) or aqueous uranic solution. The UF_6 samples are hydrolysed, diluted and acidified with nitric acid. Uranic solution samples are diluted and acidified with nitric acid. ~~Subsequently,~~ If required, an internal reference of thorium isotopes ~~is~~ can be subsequently added to each diluted sample. As detailed in Section 8, isotope pairs of elements other than thorium could be used for an internal reference.

~~3.2The~~4.2 The samples are contained in polypropylene tubes that are inserted into the auto-sampler rack of the mass spectrometer. Sample details are input to the computer and the instrument is prepared for measurement. The automatic measuring sequence is initiated.

~~3~~4.3 Uranium Isotopic Reference Materials (UIRMs) are used to calibrate the instrument. Each UIRM is prepared in aqueous

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[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.

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[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.

[3] Available from ASTM Headquarters.

solution (acidified with nitric acid) and <u>if required</u> spiked with the same internal reference as the samples. This calibration solution is measured and a mass bias parameter is calculated that is stored and subsequently imported into each of the sample measurements[4] to correct the measured uranium isotopic ratios.

~~3.4Measurements of isotopic ratios in the calibration solution and the subsequent samples are initiated by customised software. Using the~~ ^{230}~~Th /~~^{232}~~Th ratios (that are acquired simultaneously to the~~^{234}~~U /~~^{238}~~U,~~ ^{235}~~U /~~^{238}~~U and~~^{236}~~U /~~^{238}~~U ratios) and the mass bias parameter imported from the calibration, the mass bias factor is computed. The mass bias factor is then used to correct the measured~~^{234}~~U /~~^{238}~~U,~~ ^{235}~~U /~~^{238}~~U and~~^{236}~~U /~~^{238}~~U ratios in "real time." The abundances are expressed as %atomic. Details of the mass bias correction are presented in~~

4.4 <u>Measurements of isotopic ratios in the calibration solution and the subsequent samples are initiated by customised software. The mass bias factor is computed from the measured isotopic ratios in the calibration solution. This parameter is then exported to correct the measured isotopic ratios of the samples for mass bias. The corrected isotopic abundances are expressed as % atomic and are converted to % mass prior to reporting. Details of the mass bias correction are presented in</u> Appendix X1.

~~4.~~

5. Significance and Use

~~4.1The~~<u>5.1 The</u> test method is capable of measuring uranium isotopic abundances of ^{234}U, ^{235}U, ^{236}U and ^{238}U as required by Specifications C 787 and C 996.

~~5.~~

6. Interferences

~~5.1~~

6.1 *Mass Bias*—Electrostatic repulsion between uranium ions causes a so-called "mass bias" effect. Mass bias is observed as an enhancement in the number of ions detected at the collectors from the heavier uranium isotopes relative to the lighter uranium isotopes. A calibration procedure is used to correct the mass spectrometer for mass bias.

~~5.2~~

6.2 *Adjacent Isotopic Peaks*—The abundance sensitivity of the ICP-MC-MS at mass 237 is specified to be less than 0.5 parts per million of the ^{238}U ion beam. The method is limited to the measurement of ^{235}U isotopic abundances below 6 %, consequently interference effects with the ^{234}U and ^{236}U ion beams are negligible.

~~5.3~~

6.3 *Isobaric Molecular Interferences*—A molecular interference exists at mass 236 between ^{236}U and a hydride of ^{235}U, which is formed in the plasma. This interference ~~is "real-time"~~<u>can be</u> corrected by measuring the beam height of the ^{238}U hydride at mass 239, and applying the correction defined in Eq 1, to the measured ^{236}~~U beam:~~<u>U ion beam:</u>[5]

$$^{236}U_c = {}^{236}U_m - \left({}^{235}U \times \frac{^{238}UH}{^{238}U} \right) \qquad (1)$$

where:
- $^{236}U_c$ = the corrected ^{236} ~~U signal,~~ <u>U ion beam,</u>
- $^{236}U_m$ = the measured ^{236}~~U signal,~~ <u>U ion beam,</u>
- ^{235}U = the measured ^{235}~~U signal,~~ <u>U ion beam,</u>
- ^{238}UH = the measured ^{238}~~U hydride signal, and~~ <u>U hydride ion beam, and</u>
- ^{238}U = the measured ^{238}~~U signal.~~<u>U ion beam.</u>

~~5.4~~

6.4 *Memory Effects*:

~~5~~6.4.1 Contamination of the sample introduction system from previous samples produces memory interference effects. Such effects are accentuated when samples that are depleted in ^{235}U are measured after enriched samples. Memory effects can be readily assessed by aspirating a ~~2%~~ <u>0.3 M</u> nitric acid solution and measuring the background ^{238}U ion beam. The sample introduction system should be periodically disassembled and cleaned, to minimise the background ^{238}U ion beam.

~~5.4.2A background correction is performed during the measurement run by monitoring the~~ ^{230}~~Th,~~ ^{232}~~Th,~~ ^{234}~~U,~~ ^{235}~~U,~~ ^{236}~~U and~~ ^{238}~~U signals of the 2% nitric acid wash solution. The background correction is measured prior to the mass calibration and is re-measured before each subsequent sample.~~

~~6.~~

6.4.2 <u>A background correction is performed during the measurement run by monitoring the analyte signals of the 0.3 M nitric</u>

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[4] The uranium isotopic ~~measurement precision,~~<u>precision of measurement,</u> limit of detection and uncertainty of measurement are listed in Section 15 ~~, Section 16~~ and Appendix X1~~respectively.~~<u>.</u>

[5] This correction can only be applied to samples which do not contain ^{239}~~Pu.~~<u>Pu (or any other nuclides with mass 239).</u>

acid rinse solution. The background correction is measured prior to the mass calibration and is re-measured before each subsequent sample.

7. Apparatus

~~6.1~~

7.1 *Mass Spectrometer*:

~~6.1.1The~~7.1.1 The mass spectrometer has an inductively coupled plasma (ICP) source and a double ~~focussing~~focusing electrostatic/magnetic sector analyser equipped with twelve Faraday detectors and two ion counters.[6]

~~6.1.2The~~7.1.2 The mass spectrometer is fully computer controlled using customised software and is equipped with an auto-sampler.

~~6.2~~7.2 *Polypropylene Sample Tubes, Screw-Cap*, 50 mL.

~~6.3~~

7.3 *Polypropylene Sample Tubes, Screw-Cap*, 10 mL.

~~6.4Fixed-Volume Pipette, and Tips to Suit~~

7.4 *Positive Displacement Pipette, and Tips to Suit*, 0.01 mL.

~~6.5Fixed-Volume Pipette, and Tips to Suit~~

7.5 *Positive Displacement Pipette, and Tips to Suit*, 1 mL.

~~6.6~~

7.6 *Variable-Volume Dispenser*, 1 to 5 mL, fitted to a 1-L glass storage bottle.

~~7.Reagents~~

~~7.1~~

8. Reagents and Materials

8.1 *Purity of Water*— Demineralised water as defined by Type I of Specification D 1193.

~~7.2Nitric Acid Solution, high purity, various concentrations. Necessary dilutions can be inferred from the stated acid strength (for example, 2% nitric acid solution requires a ×50 dilution of the concentrated acid).~~

~~7.3Reference Solution containing 140 ng/mL of ²³⁰Th and 7 μg/mL of²³²Th.~~

~~7.4Uranium Isotopic Reference Materials (see Section 9 for details).~~

~~8.Internal References~~

~~8.1Requirements— As described in Section 3, thorium is used as an internal reference to be added to the UIRMs and uranium samples. The internal reference must contain at least one pair of isotopes in a fixed ratio. It is not necessary for this isotopic ratio to be accurately known as the same reference is added to both the calibration material and the subsequent samples. Minor fluctuations in instrument calibration (mass bias) are reflected in the measured ratio of the internal reference in the samples. Subsequent correction of the mass bias parameter using the measured ratio of the internal reference provides the necessary adjustment to the mass bias factor prior to result calculation.~~

8.2 ~~Thorium—The~~ High Purity 0.3 M Nitric Acid Solution (~x 50 dilution of the concentrated acid).

8.3 *Uranium Isotopic Reference Material (UIRMs)*—UIRMs are used to calibrate the instrument for multi-collection measurements. The Institute for Reference Materials and Measurements[7] (IRMM) reference material IRMM-024 is used for enriched samples and the New Brunswick Laboratory[8] Certified Reference Material CRM U005-A is used for samples of natural or depleted ^{235}U abundances. The UIRMs are prepared as uranyl nitrate solutions containing 0.4 μg/mL of uranium.

8.4 *Optional—Internal Reference Solution* containing ^{230}Th / ~~Th and ²³² Th ratio is monitored. The thorium solution is prepared by adding²³⁰Th to a calculated quantity of ²³²Th from a 1000 μg/mL stock standard which is then diluted with 2% nitric acid. The quantity of²³⁰Th added is such that the final diluted reference should contain 14 ng/mL of²³⁰ Th and 0.7 μg/mL of²³²Th. This solution is stored in a 1 L bottle fitted with an adjustable dispenser set to 1.0 mL. A 1.0 mL aliquot of thorium solution is dispensed into 2.0 mL of sample solution containing 1.5 μg/mL of uranium. The resultant concentration of²³⁰Th is 4.7 ng/mL and that of²³²Th is 0.23 mg/mL.~~ Th isotopes (or isotopes of another suitable element).

8.4.1 It has been found that the stability of the modern ICP-MC-MS can be such that it is not necessary to use an internal reference to monitor variations in mass bias. The data presented in this paper was obtained without the use of an internal reference. However, if the addition of an internal reference is deemed necessary then isotopes of thorium (230 and 232) can be used as a

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[6] The Nu Instruments "Nu Plasma" is such a mass spectrometer.

[6] The data presented in the paper was obtained using a 'Nu Plasma' mass spectrometer, manufactured by Nu Instruments (Nu Instruments Ltd, Unit 74 Clywedog Road South, Wrexham LL13 9XS, North Wales, UK). The Nu Plasma was supplied with the (optional) BIG80 vacuum pumping system to achieve optimum sensitivity.

230Th was supplied by AEA Technology, Harwell, Didcot, Oxfordshire, UK.

[7] Institute for Reference Materials and Measurement, Retieseweg, B-2440 Geel, Belgium.

[8] Institute for Reference Materials and Measurement, Retieseweg, B-2440 Geel, Belgium.

[8] New Brunswick Laboratory, D-350, 9800 South Cass Avenue, Argonne, Illinois 60439.

suitable internal reference material. The internal reference must contain at least one pair of isotopes in a fixed ratio. It is not necessary for this isotopic ratio to be accurately known as the same reference is added to both the calibration material and the subsequent samples. Minor fluctuations in instrument calibration (mass bias) are reflected in the measured ratio of the internal reference in the samples. Subsequent correction of the mass bias parameter using the measured ratio of the internal reference provides the necessary adjustment to the mass bias factor prior to result calculation.

8.4.2 The internal reference material should be prepared with a dilution appropriate to the sensitivity of the mass spectrometer. If thorium is used as the internal reference then a thorium to uranium ratio of approximately 1:2 should be adequate.

NOTE 1—The quantity of ^{230}Th is minimised to comply with local disposal safety regulations. 1—If an internal reference is added, then the uranic concentration of the samples should be adjusted so that the uranic concentration required for the mass spectrometer is achieved following the addition of the internal reference.

NOTE 2—The decay of ^{234}U to ^{230}Th may present a problem with the analysis of aged-uranic solutions. This should not present a problem with uranium hexafluoride samples that are taken in the gaseous phase, as gaseous UF_6 separates from any non-volatile thorium compounds.

9. Uranium Isotopic Reference Materials (UIRMs)

9.1 UIRMs are used to calibrate the instrument for multi-collection measurements. The Institute for Reference Materials and Measurements (IRMM) reference material IRMM-024 is used for enriched samples and the New Brunswick Laboratory Certified Reference Material CRM U005-A is used for samples of natural or depleted ^{235}U abundances. The UIRMs are prepared as uranyl nitrate solutions containing 1.5 μg/mL of uranium and the same quantity of internal reference as described in Section 8 (which reduces the uranium content down to 1 μg/mL).

9. Hazards

9.1 A number of the materials used in this procedure are radioactive, toxic, corrosive or any combination of the three. Adequate laboratory facilities and safe handling procedures must be used. A detailed discussion of all safety procedures is beyond the scope of this method. Site specific practices for the handling of radioactive materials and hazardous chemicals should be followed.

10. Instrument Setup

10.1 Many ICP-MC-MS designs require the Faraday collectors to be mechanically positioned to align with the ion beams. The instrument used for this work adopts a different approach, where a "zoom lens," which alters the dispersion of the instrument, is used to focus the beams onto a fixed array of detectors. The zoom lens settings were adjusted under software control to achieve the configuration shown in Table 1.

10.2 To minimise measurement uncertainty all minor isotope (^{234}U and ^{236}U) measurements are on the ion counters. The analyser magnet must be calibrated across the mass range 230 to 238, however the instrument manufacturer recommends calibrating across the mass range 80 to 238 (achieved using the beam from the Argon dimer). The magnet must be re-calibrated if the calibration drifts by more than the 0.2 atomic mass units (at uranium).

11. Sample and Blank Preparation

11.1 *Sampling, Test Specimens, and Test Units* [9]

10.1 *Samples Received as UF_6*:

11.0.1.1 Transfer between 0.2 g and 0.25 g of UF_6 gas into a glass sample tube cooled by liquid nitrogen.[10]

11.1.2 Working in a fumecupboard, hydrolyse the UF

10.1.2 Working in a fume cupboard, hydrolyse the UF_6 using demineralised water from a wash bottle. The operator should keep the sample tube pointed away at all times since some toxic HF gas is produced.

11.0.1.3 Pour the hydrolysed UF_6 into a 50 mL screw-cap polypropylene tube and dilute so that the final concentration of UF_6 is 5 mg/mL. For example, if the weight of UF_6 transferred is 0.2 g, dilute to 40 mL with demineralised water.

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[9] New Brunswick Laboratory, D-350, 9800 South Cass Avenue, Argonne, Illinois 60439.
[9] The sample dilutions specified in this section can be varied according to instrument requirements.
[10] Subsampling of UF_6 is detailed in ASTM Standard Test Method C 761.

TABLE 1

Collector	L6	L5	L4	IC1	L3	IC0	L2	L1	Ax	H1	H2	H3	H4	H5
Separation	2U	1U	1U	1U	1U	1U	1U	1U	1U	1U	1U	2U	2U	
Ion Beam	^{230}Th	^{232}Th	–	^{234}U	^{235}U	^{236}U	–	^{238}U	^{238}UH	–	–	–	–	–

where:
Ax = Axial Faraday collector,
L and H = low and high mass Faraday collectors (with respect to the Axial collector),
IC = ion counters, and
U = unit mass dispersion for uranium isotopes.

11̶0̶.1.4 Using a ~~fixed volume~~positive displacement pipette, take a 0.01 mL aliquot of solution and transfer to a clean 50 mL screw-cap polypropylene tube. Dilute to a volume of 2̶3̶42 mL using a ~~2%~~ 0.3 M nitric acid solution. The resulting solution contains 2̶.2̶1.2 µg/mL of UF$_6$ ~~which is equivalent to 1.5 µg/mL of uranium.~~

~~11.1.5 Pour 2 mL of solution into a 10 mL polypropylene tube.~~

~~11.1.6 Add 1 mL of the 0.7 µg/mL thorium internal reference and thoroughly mix the solution.~~

~~11.1.7 Place the tube in the designated rack position in accordance with Section~~ which is equivalent to 0.8 µg/mL of uranium.

10.1.5 Pour 2 mL of solution into a 10 mL polypropylene tube and double the volume to 4 mL using 0.3 M nitric acid solution, to reduce the uranic concentration to 0.4 µg/mL.

10.1.6 If required, add an aliquot of the thorium internal reference and mix the solution thoroughly (see 8.4).

10.1.7 Place the tube in the designated rack position in accordance with Section 13.

11̶.2̶10.2 *Samples Received ~~As~~as Aqueous Uranyl Nitrate Solutions ~~Of~~of Known Uranic Concentration*:

11̶0̶.2.1 Dilute the sample with a ~~2%~~ 0.3 M nitric acid solution so that the uranium concentration is ~~1.5 µg/mL~~0.8 µg/mL.

11̶0̶.2.2 Proceed in accordance with ~~11.1.5-11.1.7~~10.1.5-10.1.7.

11. Preparation of Apparatus

11.1 Many ICP-MC-MS designs require the Faraday collectors to be mechanically positioned to align with the ion beams. The instrument used for this work adopts a different approach, where a "zoom lens" which alters the dispersion of the instrument is used to focus the beams onto a fixed array of detectors. The zoom lens settings were adjusted under software control to achieve the configuration shown in Table 1.

11.2 To minimise measurement uncertainty, minor isotope (^{234}U and ^{236}U) abundances are measured with ion counters. The analyser magnet must be calibrated across the mass range 230 to 238, however, the instrument manufacturer recommends calibrating across the mass range 80 to 238 (achieved using the beam from the Argon dimer). The magnet should be re-calibrated if the calibration drifts by more than 0.2 atomic mass units.

12. Calibration and Standardization

12.1 Calibration of the mass spectrometer using a UIRM produces a mass bias factor. The mass bias factor for the UIRM in question is defined in Eq ~~2.~~ 2:

$$\text{Mass Bias Factor} = \left(\frac{\frac{^{235}U}{^{238}U} \text{ quoted}}{\frac{^{235}U}{^{238}U} \text{ measured}} \right)^{\frac{1}{\Delta m}} \tag{2}$$

where

Δm = ~~ratio mass difference (that is, 3).~~ratio mass difference (that is, 3 in the case of the ^{235}U/^{238}U ratio).

~~12.2 The mass bias factor is applied to the measured isotope ratio of the internal reference to produce a mass bias parameter. This parameter is exported to all subsequent sample measurements to correct for mass bias effects. Details of how the mass bias correction is applied can be found in~~

12.2 The mass bias factor is exported to all subsequent sample measurements to correct for mass bias effects. Details of how the mass bias correction is applied can be found in Appendix X1. As stated in Section ~~9~~8, IRMM-024 is used to calibrate for mass bias for samples enriched in ^{235}U and NBL CRM 005-A is used to calibrate for mass bias for natural samples or samples depleted in ^{235}U. Stock solutions of both these uranium reference materials (containing 1̶.5̶0.4 µg/mL of uranium in a 2̶%̶0.3 M nitric acid solution) are held in the laboratory. Mass bias calibration is an integral part of each sample run (that is, no separate calibration procedure is necessary).

13. ~~Operational~~ Procedure

13.1 The instrument software is used to assign sample names, measurement procedures and auto-sampler rack positions to both UIRMs and samples. Two sample racks are used. The first rack contains the UIRM IRMM-024 and those samples that are enriched in ^{235}U. The second rack contains the UIRM NBL CRM U005-A and those samples at natural level or depleted in ^{235}U. The plasma is then struck and after a period of 60 minutes (to allow the system to thermally stabilise), a tuning solution of UIRM IRMM-024 is aspirated and the ion beams are optimised. The automatic measurement sequence is then initiated under computer control.

14. ~~Calculation~~

~~14.1 The measurement software produces three mass bias corrected ratios as follows:~~ $r_4 = \frac{234}{238}$ $r_5 = \frac{235}{238}$ $r_6 = \frac{236}{238}$ ~~The~~ Calculation or Interpretation of Results

14.1 The measurement software produces three mass bias corrected ratios as follows:

$$r_4 = {}^{234}U/{}^{238}U \qquad r_5 = {}^{235}U/{}^{238}U \qquad r_6 = {}^{236}U/{}^{238}U$$

14.2 The ~~^{235}U abundance is calculated from the following expression:~~ U abundance is calculated from the following expression,

where the figure of 1 is to account for the ^{238}U/^{238}U ratio:

$$^{235}U = \frac{r_5}{1 + r_4 + r_5 + r_6} \times 100 \text{ \% atomic} \tag{3}$$

similarly

$$^{234}U = \frac{r_4}{1 + r_4 + r_5 + r_6} \times 100 \text{ \% atomic} \tag{4}$$

and

$$^{236}U = \frac{r_6}{1 + r_4 + r_5 + r_6} \times 100 \text{ \% atomic} \tag{5}$$

~~Finally the~~

14.3 Finally the ^{238}U % atomic is calculated by difference:

$$^{238}U = 100 - (^{234}U + {}^{235}U + {}^{236}U) \text{ \% atomic} \tag{6}$$

~~Before reporting, the isotopic abundancies in % atomic are converted to % mass.~~

14.4 The isotopic abundances in % atomic can be expressed as % mass using the following equations:

$$^{235}U = \frac{b \times B}{(a \times A) + (b \times B) + (c \times C) + (d \times D)} \times 100 \text{ \% mass} \tag{7}$$

where:
a = ^{234}U% atomic
b = ^{235}~~U atomic~~ U% atomic
c = ^{236}~~U atomic~~ U% atomic
d = ^{238}~~U atomic~~
~~A~~U% atomic
A = atomic weight of ^{234}U (234.0409)
B = atomic weight of ^{235}U (235.0439)
C = atomic weight of ^{236}U (236.0457)
D = atomic weight of ^{238}U (238.0508)

similarly

$$^{234}U = \frac{a \times A}{(a \times A) + (b \times B) + (c \times C) + (d \times D)} \times 100 \text{ \% mass} \tag{8}$$

$$^{234}U = \frac{a \times A}{(a \times A) + (b \times B) + (c \times C) + (d \times D)} \times 100 \text{ \% mass} \tag{8}$$

~~× 100 % mass~~

and

$$^{236}U = \frac{c \times C}{(a \times A) + (b \times B) + (c \times C) + (d \times D)} \times 100 \text{ \% mass} \tag{9}$$

$$^{236}U = \frac{c \times C}{(a \times A) + (b \times B) + (c \times C) + (d \times D)} \times 100 \text{ \% mass} \tag{9}$$

~~× 100 % mass~~

15. Precision and Bias

15.1 *Precision*—The intermediate precision of the method for multi-collection measurements at various ^{235}U abundances is presented in the following tables. Certified uranium ~~isotopic~~ abundances are quoted with 2 sigma confidence intervals. The units of precision are % Atomic (see Table 2).

15.2 *Bias*—In practice the observed agreement (at the 2 sigma level) between the certified and measured isotopic abundances for NBS050, NBS030, NBS020 and NBS005 ~~(tabulated in~~ (see ~~15.1~~ Table 2) indicates that there is no significant bias. ~~16.~~

15.3 *Limit of Detection*

~~16.1 The~~—The limit of detection was derived from measurements of ^{236}U in samples derived from natural uranium (that is, samples with negligible ^{236}U content). The results of 10 determinations of ^{236}U both on natural uranium and an enriched (3.3 % ^{235}U) material derived from natural are summarised as follows. Each determination is based on an individual sample measurement. The 10 determinations were distributed over several measurement runs (by more than one operator).