Designation: C1165 – 12

Standard Test Method for Determining Plutonium by Controlled-Potential Coulometry in H_2SO_4 at a Platinum Working Electrode[1]

1. Scope

1.1 This test method covers the determination of milligram quantities of plutonium in unirradiated uranium-plutonium mixed oxide having a U/Pu ratio range of 0.1 to 10. This test method is also applicable to plutonium metal, plutonium oxide, uranium-plutonium mixed carbide, various plutonium compounds including fluoride and chloride salts, and plutonium solutions.

1.2 The recommended amount of plutonium for each aliquant in the coulometric analysis is 5 to 10 mg. Precision worsens for lower amounts of plutonium, and elapsed time of electrolysis becomes impractical for higher amounts of plutonium.

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

1.4 *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.* Specific precautionary statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable

C758 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Metal

C759 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions

C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets

C859 Terminology Relating to Nuclear Materials

C1009 Guide for Establishing and Maintaining a Quality Assurance Program for Analytical Laboratories Within the Nuclear Industry

C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry

C1108 Test Method for Plutonium by Controlled-Potential Coulometry

C1128 Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials

C1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials

C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis

C1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry

C1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials

3. Summary of Test Method

3.1 In controlled-potential coulometry, the analyte reacts at an electrode having a maintained potential that precludes reactions of as many impurity components as is feasible. In the electrolysis, current decreases exponentially as the reaction proceeds until a selected background current is reached. The quantity of analyte reacted is calculable by Faraday's law. Detailed discussions of the theory and applications of this technique are presented in Refs **(1)**[3] and **(2)**.

3.2 Plutonium and many impurity element ions are initially reduced in a 0.5 *M* H_2SO_4 electrolyte at a platinum working electrode **(3)** maintained at + 0.310 V versus a saturated calomel electrode (SCE). Plutonium is then oxidized to Pu(IV) at a potential of + 0.670 V. The quantity of plutonium is

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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] The boldface numbers in parentheses refer to a list of references at the end of the text.

calculated from the number of coulombs required for oxidation according to Faraday's law.

$$Q = \int_o^t i\,dt = nwF/M \tag{1}$$

Rearrangement to solve for *w* gives:

$$w = MQ/nF \tag{2}$$

where:

w = weight of Pu(III) oxidized to Pu(IV), g,
M = gram-molecular mass of plutonium (adjusted for isotopic composition), grams/equivalent,
Q = number of coulombs to oxidize Pu(III) to Pu(IV), coulombs,
n = number of electron change to oxidize Pu(III) to Pu(IV) = 1, and
F = Faraday constant, coulomb/equivalent.

3.3 An electrolyte of sulfuric acid, that selectively complexes Pu(IV), provides very reproducible electrolysis of Pu(III) to Pu(IV). In a 0.5 *M* H_2SO_4 electrolyte, the reduction potential of + 0.310 V for conversion of Pu(IV and VI) to Pu(III) and the oxidation potential of + 0.670 V for conversion of Pu(III) to Pu(IV) accounts for about 99.9 % (as calculated from the Nernst equation) conversion of the total plutonium in solution. There are few interferences at the selected potentials of the metallic impurities usually listed in specifications for fast breeder reactor (FBR) mixed oxide fuel. A chemical calibration of the coulometric system using the selected potentials technique is necessary to correct for the less than 100 % conversions of Pu(III) and Pu(IV).

3.4 Sulfuric acid is a convenient electrolyte since it is used for preliminary fuming of samples to volatilize interfering components (see 5.3 and 5.4). The preliminary fuming with sulfuric acid also serves to depolymerize any polymeric plutonium species, which tend to be electrolytically inactive (**3**).

4. Significance and Use

4.1 This test method is to be used to ascertain whether or not materials meet specifications for plutonium content or plutonium assay, or both.

4.2 A chemical calibration of the coulometer is necessary for accurate results.

5. Interferences

5.1 Categories of interferences are diverse metal ions that oxidize or reduce at the potential of + 0.670 V used for the oxidation of Pu(III) to Pu(IV), organic matter, anions that complex plutonium, and oxygen.

5.2 The major interfering metallic impurity element, of those usually included in specifications for FBR mixed oxide fuel, is iron (**4**). In the 0.5 *M* H_2SO_4 electrolyte, the Fe(II) − Fe(III) and Pu(III) − Pu(IV) couples have essentially the same E^o value of + 0.490 V. The iron interference, therefore, is quantitative and is corrected based on its measured value that can be determined by a spectrophotometric method (**5**). Alternatively, other techniques such as ICP, DCP, or emission spectrometry can also be used if the iron content is sufficiently low. When the iron result is <20μ g/g, the lower limit of the spectrophotometric method, no correction is necessary. The best available method for iron determination is recommended since the uncertainty in the iron correction contributes to the uncertainty in the plutonium value.

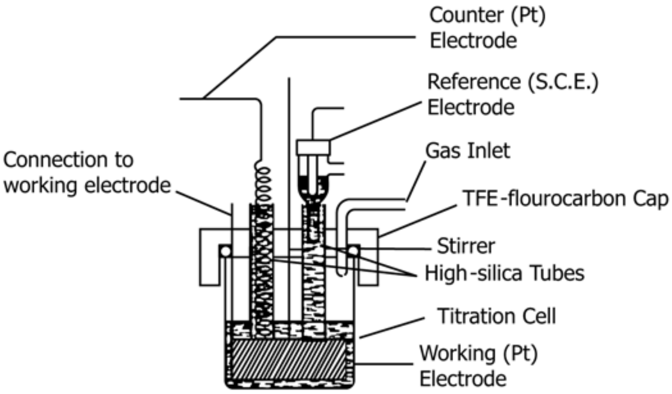

FIG. 1 Example of a Cell Design Used at Los Alamos National Laboratory (LANL)

5.3 Organic matter usually is not present in calcined mixed oxide fuel pellets nor in mixed oxide powder blends prepared using calcined uranium oxide and calcined plutonium oxide. However, it may be introduced as an impurity in reagents. The sulfuric acid fuming of reference material and of samples that precedes the coulometric analysis volatilizes most organic components.

5.4 The sulfuric acid fuming volatilizes nitrate, nitrite, fluoride, and chloride, that are introduced by the use of a nitric-hydrofluoric acid mixture or acid mixtures containing chloride for the dissolution of samples and interfere in the coulometric determination of plutonium.

5.5 Oxygen interferes and must be purged continuously from both the solution and atmosphere in the electrolysis cell with an oxygen-free inert gas before and during the electrolysis.

NOTE 1—The purge gas tube extends through the cell cover and is positioned approximately 1 cm above the sample solution in the cell. The inert gas flow is maintained at a flow rate that causes a dimple to be seen on the surface of the solution with the stirrer off. The inert gas flow rate should be such that no splashing occurs.

5.6 Nitric acid and hydrofluoric acid must be added during the preparation of the plutonium metal to ensure oxidation of the plutonium to Pu(IV) and to match the acid matrix from plutonium oxide dissolution. Plutonium that is dissolved in only hydrochloric acid and then evaporated to dryness in sulfuric acid while in the Pu(III) oxidation state will contain tiny blue crystals within the pink plutonium (IV) sulfate material, and lower recoveries are experienced during the coulometric measurement. Blue crystals are not observed when plutonium oxide materials are dissolved in HNO_3 and HF acids and subsequently fumed to dryness in H_2SO_4.

5.7 Due to a slight overlap between the potential at which Np(VI) reduces to Np(V), +0.660 V, and the potential used in the current method to oxidize Pu(III) to Pu(IV), +0.670 V, a large amount of neptunium will cause the plutonium assay to be biased high and not accurately reflect the plutonium content of the material being analyzed. Thus, neptunium can only be tolerated up to 1 % in the sample, above that level the neptunium must be removed prior to the sample undergoing the coulometry process.

6. Apparatus

6.1 *Controlled-Potential Coulometer*—A potentiostat having stable potential control at approximately 200 mA and 20 V and an integrator capable of 0.05 % reproducibility are required. The linearity of the integrator should be better than 0.1 % for the selected range.[4]

6.2 *Cell Assembly*—A cell assembly similar to the one described in Ref (**5**) has been used satisfactorily. Cell design is very critical in controlled-potential coulometry. There are many factors that must be considered in choosing or designing a cell assembly. It is beyond the scope of this test method to describe all of the factors that should be considered. A thorough detailed discussion of electrolysis cell design is presented in Ref (**2**).

NOTE 2—Drawing (see Fig. 1) of a cell design that has been successfully used at the Los Alamos National Laboratory. The titration cell consists of a 50 mL cut off beaker.

6.3 Timer or stopwatch for measuring electrolysis times (capable of measuring in seconds).

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled or deionized water.

7.3 *Argon, Oxygen-Free (99.99 %)*—Helium, nitrogen, or other pure inert gas may be used.

7.4 *Hydrochloric Acid (HCl, 10.9 M)*—Concentrated HCl, ACS ultratrace grade.

7.5 *Hydrochloric Acid* (HCl, 6 *M*)—Add 500 mL of concentrated 10.9 M HCl to less than 500 mL of water and dilute to 1 L with water.

7.6 *Hydrochloric Acid (HCl, 1.0 M)*—Add 82.6 mL of concentrated 10.9 M HCl to water and dilute to 1L.

7.7 *Hydrofluoric Acid (HF, 29M)*—Concentrated HF, ACS ultratrace grade.

7.8 *Hydrofluoric Acid (HF, 1.3 M)*—Add 4.8 mL of concentrated 29 M HF to water and dilute to 100 mL.

7.9 *Nitric Acid* (HNO_3, *15.9 M*)—Concentrated HNO_3, ACS ultratrace grade.

7.10 *Sulfuric Acid* (H_2SO_4, *18.1 M*)—Concentrated H_2SO_4 , ACS ultratrace grade.

7.11 *Sulfuric Acid* (3 *M*)—Add 168 mL of concentrated H_2SO_4 to water, while stirring, and dilute to 1 L with water.

7.12 *Sulfuric Acid* (0.5 *M*)—Add 28 mL of concentrated H_2SO_4 to water, while stirring, and dilute to 1 L with water.

7.13 *Plutonium Reference Solution*—Dissolve a weighed quantity (balance capable of weighing to 0.01 mg) of 0.5 to 1 g of NBL (Note 4) CRM 126 metal (or its replacement) cleaned per certificate directions in 6 *M* HCl. Use a sufficient amount of 6 *M* HCl to maintain an acid concentration of 1 to 2 *M*. Completely transfer the solution with 1.0 *M* HCl rinses to a tared container, dilute to 100 to 200 g with 1.0 *M* HCl(to give a plutonium concentration of 5 mg/g), and weigh.

NOTE 3—A tared polyethylene bottle has been used successfully to dispense weighed aliquants.

NOTE 4—To minimize measurement uncertainty, it is recommended that the reference and sample aliquants contain approximately the same amount of plutonium. Users of this standard are responsible for validating method performance if aliquants of standards and/or samples containing less than 5 mg of plutonium or greater than 10 mg of plutonium will be measured.

New Brunswick Laboratory (NBL) Certified Reference Materials Catalog (U.S. Department of Energy), http://www.nbl.doe.gov.

7.13.1 Dispense weighed 1 to 2 g aliquants, each containing accurately known 5 to 10 mg quantities of plutonium, to individual electrolysis cells or vials for subsequent use in chemical calibration.

7.13.2 Prior to using, add 0.5 mL of 3 *M* H_2SO_4, 1 drop of 1.3 *M* HF and 1 drop of concentrated 15.9 *M* HNO_3 and fume to dryness.

7.13.3 After cooling, redissolve using a minimal amount of 0.5 *M* H_2SO_4 and again fume to dryness.

7.13.4 Repeat 7.13.3.

8. Safety Precautions

8.1 *Committee C-26 Safeguards Statement*[6]:

8.1.1 The materials (nuclear grade plutonium metal, plutonium oxide powder, plutonium nitrate solutions, and mixed oxide and carbide powders and pellets) to which this test method applies, are subject to nuclear safeguards regulations governing their possession and use. This test method has been

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[4] Coulometer suppliers or designers who have reported instrument performances that are consistent with the specification provided in this standard include: the SRNL Coulometer, Savannah River National Laboratory, Aiken, South Carolina, USA; the Mayak Coulometer PIK-200, Ozersk, Russia; and the coulometer at the LAMM Laboratory, CEA Centre de Marcoule, Bagnols-sur-CèreCedex, France. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[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.

[6] Based upon Committee C26 Safeguards Matrix (C1009, C1068, C1128, C1156, C1210, and C1297).

designated as technically acceptable for generating safeguards accountability measurement data.

8.1.2 When used in conjunction with appropriate certified reference materials (CRMs), this test method can demonstrate traceability to the national measurements base. However, adherence to this test method does not automatically guarantee regulatory acceptance of the resulting safeguards measurements. It remains the sole responsibility of the user of this test method to ensure that its application to safeguards has the approval of the proper regulatory authorities.

8.2 **Warning—**Hydrofluoric acid is a highly corrosive acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid is similar to other acids in that the initial extent of a burn depends on the concentration, the temperature, and the duration of contact with the acid. Hydrofluoric acid differs from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers. Unlike other acids that are rapidly neutralized, hydrofluoric acid reactions with tissue may continue for days if left unattended. Due to the serious consequences of hydrofluoric acid burns, prevention of exposure or injury of personnel is the primary goal. Utilization of appropriate laboratory controls (hoods) and wearing adequate personal protective equipment to protect from skin and eye contact is essential. Acute exposure to HF can cause painful and severe burns upon skin contact that require special medical attention. Chronic or prolonged exposure to low levels on the skin may cause fluorosis.

9. Preparation of Apparatus

9.1 Verify proper equipment operation by performing an electrical calibration according to manufacturers' specifications on each day that the instrument is used.

10. Calibration

10.1 If not done previously as recommended in 7.13.1, completely transfer one of the dispensed aliquants, containing 5 to 10 mg of plutonium of the plutonium reference solution, to a cell using 0.5 *M* H_2SO_4 rinses and place platinum working electrode in the cell. Using 0.5 *M* H_2SO_4, completely immerse the working electrode. (See Note 8.)

10.2 Rinse the exterior surfaces of the counter and reference electrode salt bridges (for example, high-silica tubes) with 0.5 *M* H_2SO_4.

10.3 Raise the cell into position firmly against the cell cover to ensure a tight fit. Purge the cell atmosphere with flowing argon or other inert gas. (See Note 1.)

10.4 Immediately connect the cell electrodes to the coulometer; begin stirring.

10.5 Reduce Pu(IV) to Pu(III) at +0.310 V until the current decreases to 30 µA.

10.6 Reset the integrator and start timer.

10.7 Oxidize Pu(III) to Pu(IV) at +0.670 V until the current decreases to 30 µA. Record the coulomb accumulation and elapsed time.

NOTE 5—All standards (reference material) and samples should be freshly fumed (within 4 h) prior to analysis.

10.8 Remove the solution and thoroughly rinse the cell and electrodes with 0.5 *M* H_2SO_4.

10.9 Repeat 10.1 – 10.8 to attain a desired precision level for the calibration.

NOTE 6—A recommended practice would be to intersperse standards (reference material) and samples during the time the analyses are being done.

10.10 Calculate the calibration factor *F* by

$$F = M/\left(C_C - C_B\right) \tag{3}$$

where:

F = calibration factor, milligrams plutonium per coulomb,
M = mass of plutonium in calibration reference aliquant, milligrams,
C_C = coulombs measured at 0.670 V electrolysis for calibration reference aliquant, and
C_B = coulombs for blank measurement. Use the coulomb value obtained on the blank for the elapsed time (to the nearest minute) as that required for the reference aliquant oxidation time.

11. Procedure

11.1 *Blanks:*

11.1.1 Obtain reproducible blank measurements on each individual platinum electrode by following 11.1.2 – 11.1.8.

NOTE 7—Two platinum working electrodes are recommended to increase sample throughput by alternating the electrodes. While one electrode is being used in an electrolysis, the other electrode is being cleaned by sitting in a beaker of hot concentrated nitric acid. The electrode that is being cleaned is rinsed thoroughly with water and 0.5 *M* sulfuric acid prior to its use.

11.1.2 Add 0.5 *M* H_2SO_4 to the cell to completely immerse the working electrode.

NOTE 8—Avoid overfilling the cell. Fill only to the top of the platinum gauze working electrode. Overfilling the cell will result in longer electrolysis times and larger background currents.

11.1.3 Rinse the counter and reference electrode salt bridges (high-silica tubes) with 0.5 *M* H_2SO_4.

11.1.4 Raise the cell into position firmly against the cell cover to ensure a tight fit. Purge the cell atmosphere with flowing argon or other inert gas. (See Note 1.)

11.1.5 Immediately connect the cell electrodes to the coulometer; begin stirring.

11.1.6 Electrolyze the blank at 0.310 V until a 30-µA current is obtained.

11.1.7 Start the timer, and electrolyze the blank at 0.670 V for a period of time that is consistent with sample electrolysis times.

11.1.8 Record the number of coulombs at elapsed electrolysis times consistent with sample electrolysis times.

11.1.9 Following blanks, run a plutonium cell conditioner sample to equilibrate the cell prior to running standards (reference material) and samples.

NOTE 9—A plutonium cell conditioner sample is a plutonium solution that is run through the complete reduction/oxidation cycle but is not used for calculation purposes. Experience has shown that if a plutonium cell conditioner is not run, the initial plutonium result will be low. A possible cause for this effect is migration of plutonium into the high silica tubes until equilibration is attained.

11.2 *Sample Analysis:*

11.2.1 The plutonium-containing material may be dissolved using the appropriate dissolution procedure described in Practice C1168.

11.2.2 After transferring and diluting, weigh aliquants containing 5 to 10 mg of plutonium.

11.2.3 Add 0.5 mL of 3 M H_2SO_4 to each aliquant and fume to dryness. For aliquants from metal samples only, add 0.5 mL of 3 M H_2SO_4, 1 drop of 1.3 M HF and 1 drop of concentrated 15.9 M HNO_3 and fume to dryness.

11.2.4 After cooling, dissolve the sample using a minimal amount of 0.5 M H_2SO_4 and again fume to dryness.

11.2.5 Repeat 11.2.4.

11.2.6 Dissolve the sample using a minimal amount of 0.5 M H_2SO_4.

11.2.7 Place a platinum working electrode in the cell and completely immerse the working electrode using 0.5 M H_2SO_4.

11.2.8 Proceed with the coulometric analysis of one or more aliquants by following 10.2 – 10.8.

11.2.9 Correct for the iron content of the sample, which has been determined using the recommended spectrophotometric procedure or a suitable alternate procedure.

12. Calculation of Sample Result

12.1 Calculate the plutonium content of the sample by

$$Pu = (D)\ (A_S/A_R)\ F_{avg}(C_S - C_B)/M_S \tag{4}$$

where:

Pu = result, gram plutonium per gram sample,
D = dilution factor, grams of diluted sample/grams of aliquant analyzed,
A_S = atomic weight of plutonium in sample,
A_R = atomic weight of plutonium in plutonium metal reference material,
F_{avg} = average calibration factor, milligrams plutonium per coulomb (see 10.10),
C_S = coulombs measured for 0.670 V electrolysis for sample aliquant,
C_B = coulombs for blank measurement (same elapsed time to the nearest minute as for sample), and
M_S = mass of solid sample initially dissolved, milligrams.

12.2 Calculate the correction for iron by

$$Fe_c = (10^{-6})(Fe)(A_S/55.85) \tag{5}$$

where:

Fe_c = correction for iron, gram plutonium/gram of sample,
Fe = micrograms iron/gram of sample, and
A_S = atomic weight of plutonium in sample.

12.3 Calculate the corrected plutonium content, Pu_c, of the sample by

$$Pu_c = Pu - Fe_c \tag{6}$$

13. Precision and Bias

13.1 For a single measurement on an aliquant, the estimated repeatability relative standard deviation is 0.10 % and the estimated reproducibility relative standard deviation is 0.15 %. These estimates are based on the analysis of 5 samples, 4 aliquants each, by each of 6 laboratories (**6**) and the analysis of 153 aliquants involving 9 distinct dissolutions of a control sample at 1 laboratory. If more than one aliquant is measured (see 11.2.8) and the average reported, the repeatability and reproducibility relative standard deviations are $0.10/\sqrt{n}$ % and $0.15/\sqrt{n}$ %, respectively, where n is the number of measurements in the average.

13.2 Comparison with a potentiometric method, a photometric method, and with 100 % impurities data indicate that the coulometric method is essentially unbiased.

14. Keywords

14.1 controlled-potential coulometry; plutonium analysis; plutonium at platinum electrode; plutonium in sulfuric acid; plutonium-uranium mixtures

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