

REGULATORY GUIDE

OFFICE OF STANDARDS DEVELOPMENT

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REGULATORY GUIDE 5.16

STANDARD METHODS FOR CHEMICAL, MASS SPECTROMETRIC, SPECTROCHEMICAL, NUCLEAR, AND RADIOCHEMICAL ANALYSIS OF NUCLEAR-GRADE PLUTONIUM NITRATE SOLUTIONS AND PLUTONIUM METAL

A. INTRODUCTION

Paragraph (b) of Section 70.22, "Contents of Applications," of 10 CFR Part 70, "Special Nuclear Material," requires an applicant for a license to possess certain quantities of special nuclear material in an unsealed form to describe, among other things, his procedures for control of and accounting for special nuclear material. This guide identifies methods acceptable to the NRC staff for chemical, isotopic, and impurity analyses that an applicant may specify as part of his procedures for accounting for special nuclear material.

B. DISCUSSION

Committee C-26 on Fuel, Control, and Moderator Materials for Nuclear Reactor Applications of the American Society for Testing and Materials (ASTM) has developed standards containing methods for the chemical analysis of (1) nuclear-grade plutonium nitrate solutions and (2) nuclear-grade plutonium metal. These standards are ASTM C759-73, "Standard Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions,"¹ and ASTM C758-73, "Standard Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Metal."¹ On June 20, 1974, these standards were approved by the ANSI Board of Standards review and were designated ANSI N572-1974 (ASTM C758-73), "Chemical, Mass Spectrometric, Spectrochemical, Nuclear and Radiochemical Analyses

of Nuclear-Grade Plutonium Metal,"² and ANSI N573-1974 (ASTM C759-73), "Chemical, Mass Spectrometric, Spectrochemical, Nuclear and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions."² As used in these standards, *nuclear-grade* material means material that is to be used *exclusively* for the fabrication of nuclear fuel.

Plutonium Nitrate Solutions

Included in this standard are two methods for the determination of plutonium:

1. Controlled-Potential Coulometry. The optimum quantities of plutonium to be determined are 2 to 6 mg with a stated precision of 0.14 percent relative standard deviation (RSD).

2. Amperometric Titration with Iron (II). The optimum quantities of plutonium to be determined are 10 to 20 mg with a stated precision of 0.03 to 0.06 percent RSD and a bias of 0.02 percent or less.

This standard also includes a method for the determination of the absolute isotopic composition of plutonium nitrate solutions allowing for the use of reference standards to determine bias. The stated precision of the method for Pu-239 at the 91% concentration level is 0.034% RSD; for Pu-240 at the 7.9% concentration level, 0.182% RSD; for Pu-241 at the 0.63% concentration level, 0.978% RSD; and for Pu-242 at the 0.033% concentration level, 4.6% RSD. Various impurities such as uranium, thorium, iron, chloride, fluoride, and sulfate can be determined using

¹Copies may be obtained from the American Society for Testing Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

*Lines indicate substantive changes from previous issue.

²Copies may be obtained from the American National Standards Institute, Inc., 1430 Broadway, New York, New York 10018.

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Comments and suggestions for improvements in these guides are encouraged at all times, and guides will be revised, as appropriate, to accommodate comments and to reflect new information or experience. However, comments on this guide, if received within about two months after its issuance, will be particularly useful in evaluating the need for an early revision.

Comments should be sent to the Secretary of the Commission, U.S. Nuclear Regulatory Commission, Washington, D.C. 20555, Attention: Docketing and Service Section.

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the methods that are included in this standard. Two methods for the determination of the free acid content of the plutonium nitrate solutions are also included in this standard.

Plutonium Metal

Included in this standard are three methods for the determination of plutonium:

1. **Controlled-Potential Coulometry.** The optimum quantities of plutonium to be determined are 2 to 6 mg with a stated precision of 0.14 percent relative standard deviation (RSD).

2. **Ceric Sulfate Titration.** The stated precision is 0.27 percent RSD.

3. **Amperometric Titration with Iron(II).** The optimum quantities of plutonium to be determined are 10 to 20 mg with a stated precision of 0.03 to 0.06 percent RSD and a bias of 0.02 percent or less.

This standard also includes a method for the determination of the absolute isotopic composition of plutonium metal allowing for the use of reference standards to determine bias. The stated precision of the method for Pu-239 at the 91% concentration level is 0.034% RSD; for Pu-240 at the 7.9% concentration level, 0.182% RSD; for Pu-241 at the 0.63% concentration level, 0.978% RSD; and for Pu-242 at the 0.033% concentration level, 4.6% RSD. Various impurities such as uranium, thorium, iron, chloride, fluoride, nitrogen, carbon, sulfur, and americium-241 can be determined using the methods that are included in this standard.

C. REGULATORY POSITION

The analytical methods for the measurement of nuclear-grade plutonium nitrate solutions contained in ASTM C759-73¹ or ANSI N573-1974², "Standard Methods for Chemical, Mass Spectrometric,

Spectrochemical, Nuclear and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions," and the analytical methods for the measurement of nuclear-grade plutonium metal contained in ASTM C758-73¹ or ANSI N572-1974², "Standard Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear and Radiochemical Analysis of Nuclear-Grade Plutonium Metal," are acceptable to the NRC staff and provide an adequate basis for the assay, isotopic measurement, and impurity analysis of nuclear-grade plutonium nitrate solutions and plutonium metal, subject to the following:

1. **Precision and Accuracy Statements.** The listed statements provide guidance to the levels of performance which may be attained using the described methods. The actual precision and accuracy of a method applied within a selected laboratory can only be determined through a well-planned measurement control program.

2. **Calibration and Standardization.** The standards should be prepared in the same matrix as the samples, and the calibration points should bracket the estimated range of the samples.

3. **Safety.** Procedures involving the use of perchloric acid in radiochemical hoods and glove boxes should be performed with caution because of the potential explosion and fire hazard.

D. IMPLEMENTATION

The purpose of this section is to provide information to applicants and licensees regarding the NRC staff's plans for utilizing this regulatory guide.

This guide reflects current regulatory practice. Therefore, except in those cases in which the applicant proposes an alternative method for complying with specified portions of the Commission's regulations, the method described herein will be used in the evaluation of submittals in connection with special nuclear material license or operating license applications docketed after this guide is issued.



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