# U.S. ATOMIC ENERGY COMMISSION

DIRECTORATE OF REGULATORY STANDARDS

#### **REGULATORY GUIDE 5.40**

## METHODS FOR THE ACCOUNTABILITY OF PLUTONIUM DIOXIDE POWDER

#### A. INTRODUCTION

Paragraph 70.22(b) of 10 CFR Part 70, "Special Nuclear Material," requires, among other things, that an applicant for a license to possess certain quantities of special nuclear material in an unsealed form describe his procedures for control of and accounting for special nuclear material. This regulatory guide identifies methods and criteria acceptable to the Regulatory staff for the sampling, subsampling, sample handling, dissolution, chemical and isotopic analysis, and error analysis for the accountability of plutonium dioxide powder.

#### **B. DISCUSSION**

The Lawrence Livermore Laboratory and the U.S. Atomic Energy Commission have recently published a document (WASH 1335) entitled "Methods for the Accountability of Plutonium Dioxide Powder." This document describes methods for the sampling, subsampling, sample handling, dissolution, chemical and isotopic analysis, and error analysis of plutonium dioxide powder. Topics included in the report under the section on sampling methods are general principles and precautions of sampling, homogeneity problems, and the chemical and physical characteristics of PuO<sub>2</sub> powders which influence sampling. The sections on sample handling and subsampling include discussions on containers, labeling, and sources of error. Both acid and fusion techniques are covered in the section on dissolution. The analytical procedures presented and discussed are (1) controlled-potential coulometry, amperometry, and modified amperometry as assay methods; (2) ion exchange cleanup, spectrographic analysis and iron determination by spectrophotometry for impurity removal or determination; and (3) isotopic analysis by mass spectrometry and alpha-plus-height analysis (Pu-238). The criteria for the selection of the destructive analytical methods described in this document are as follows:

- 1. High precision and accuracy combined with ease of use and low cost.
- 2. Broad applicability to various types of plutonium oxide material and relative freedom from interfering substances.
- 3. The extent that the methods have been studied by various research laboratories, the success of their use under actual plant conditions, and the historical evaluations that have been performed on them under various exchange programs.

#### C. REGULATORY POSITION

The accountability methods and related criteria for the sampling, subsampling, sample handling, dissolution, chemical and isotopic analysis, and error analysis of plutonium dioxide powder contained in WASH 1335. "Methods for the Accountability of Plutonium Dioxide Powder,"\* are generally acceptable and provide an adequate basis for the accountability of plutonium dioxide powder.

#### D. IMPLEMENTATION

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

This guide reflects current regulatory practice. Therefore, except in those cases in which an applicant or licensee proposes an alternative method for complying with specified portions of the Commission's regulations, the methods described herein will be used immediately in the evaluation of applications for licenses to possess at any one time special nuclear material in a quantity exceeding one effective kilogram as described in §70.22(b) of 10 CFR Part 70 and in the evaluation of the performance of holders of such licenses.

\*Copies may be obtained from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C.

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