ORNL/TM-2000/82

Thermal Stabilization of $^{233}UO_2$, $^{233}UO_3$, and $^{233}U_3O_8$

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S. M. Thein
P. J. Bereolos

ADVANCED INTEGRATED MANAGEMENT SERVICES, INC.
702 S. Illinois Avenue, Suite B-203
Oak Ridge, Tennessee 37830

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ABSTRACT

This report identifies an appropriate thermal stabilization temperature for ^{233}U oxides. The temperature is chosen principally on the basis of eliminating moisture and other residual volatiles. This report supports the U. S. Department of Energy (DOE) Standard for safe storage of ^{233}U (DOE 2000), written as part of the response to Recommendation 97-1 of the Defense Nuclear Facilities Safety Board (DNFSB), addressing safe storage of ^{233}U.

The primary goals in choosing a stabilization temperature are (1) to ensure that the residual volatiles content is less than 0.5 wt % including moisture, which might produce pressurizing gases via radiolysis during long-term sealed storage; (2) to minimize potential for water readsorption above the 0.5 wt % threshold; and (3) to eliminate reactive uranium species. The secondary goals are (1) to reduce potential future chemical reactivity and (2) to increase the particle size thereby reducing the potential airborne release fraction (ARF) under postulated accident scenarios.

The prevalent species of uranium oxide are the chemical forms UO_2, UO_3, and U_3O_8. Conversion to U_3O_8 is sufficient to accomplish all of the desired goals. The preferred storage form is U_3O_8 because it is more stable than UO_2 or UO_3 in oxidizing atmospheres. Heating in an oxidizing atmosphere at 750°C for at least one hour will achieve the thermal stabilization desired.

CONTENTS

ABSTRACT iii
LIST OF FIGURES vii
LIST OF TABLES vii
ACRONYMS vii
1. INTRODUCTION 1
2. URANIUM OXIDE PROPERTIES 3
  2.1 THERMAL CONDUCTIVITY 3
  2.2 PARTICLE SIZE 3
  2.3 STABILITY 4
3. HEATING EFFECTS ON VOLATILES 5
  3.1 MOISTURE 5
  3.2 OTHER VOLATILES 5
4. HEATING EFFECTS ON SELECTED URANIUM OXIDES 7
  4.1 $^{233}UO_2$ 7
  4.2 $^{233}UO_3$ 7
  4.3 $^{233}U_3O_8$ AND OTHER OXIDES 8
5. SELECTION OF A STABILIZATION TEMPERATURE 11
6. REFERENCES 13

LIST OF FIGURES

Fig. 1. Rate of decomposition of UO_3 to U_3O_8 in air. 9
Fig. 2. Thermal profile of selected uranium oxides. 12

LIST OF TABLES

Table 1. Uranium oxide thermal conductivities 3
Table 2. Crystalline uranium trioxides 7

ACRONYMS

ADU	Ammonium Diuranate
ARF	Airborne Release Fraction
DNFSB	Defense Nuclear Facilities Safety Board
DOE	U.S. Department of Energy
IAEA	International Atomic Energy Agency
ORNL	Oak Ridge National Laboratory
PWR	Pressurized Water Reactor

1. INTRODUCTION

The purpose of this report is to select a thermal stabilization temperature to treat ^{233}U oxides. This report supports the U. S. Department of Energy (DOE) Standard for the safe storage of ^{233}U (DOE 2000), written in response to Recommendation 97-1 of the Defense Nuclear Facilities Safety Board (DNFSB) (DNFSB 1997).

The existing prevalent ^{233}U oxide powder species present are UO_2, UO_3, and U_3O_8. The merits and deficiencies of different temperatures for the treatment of each species will be discussed. The U:O systems discussed are stoichiometric ratios that indicate the 3 major crystalline phases (with some variation in stoichiometry depending on oxygen potential).

For reasons that will be detailed further in this report, the desired final stabilized form for the uranium oxides is U_3O_8 powder. Moisture and residual volatiles content will be less than 0.5 wt %. Reactive uranium species will have been eliminated; and for some starting materials, the particle size should be increased and the specific surface area reduced.

The primary goal of thermal stabilization is to eliminate moisture and other residual volatiles that could potentially produce pressurizing gases via radiolysis during long-term sealed storage. This goal can be accomplished while converting the material to the most stable form, U_3O_8. Stabilization also minimizes the potential for water readsorption above the 0.5 wt % threshold, stabilizes reactive uranium species, and increases particle size (reduces specific surface area) for some starting species. These characteristics will enable the safe long-term storage of ^{233}U-bearing materials.

2. URANIUM OXIDE PROPERTIES

2.1 THERMAL CONDUCTIVITY

Because of the relatively low thermal conductivity of UO_2, UO_3, and U_3O_8, the temperature for heating the uranium material in this document refers to the temperature of the material itself instead of the oven temperature. Table 1 is a compilation of the uranium oxide thermal conductivities.

Table 1. Uranium oxide thermal conductivities

Uranium oxide	Thermal conductivity (W m^{-1} °C^{-1})
UO_2	0.705[a]
U_3O_8	0.0838[b]

[a]Prepared from Mallinckrodt pressurized-water reactor (PWR)-grade powder; 85% theoretical density, 1400 K (Touloukian 1967).
[b]Pressed at 100 psi, 775 K (Touloukian 1967)

For comparison, the thermal conductivity of aluminum metal is 240 W m^{-1} ºC $^{-1}$ at 100ºC (McCabe, Smith, and Harriot 1993). Because of the relatively low thermal conductivity of uranium oxides, measurement of the material temperature (or compensation for thermal conductivity), rather than relying solely on the oven temperature, is required.

2.2 PARTICLE SIZE

By increasing particle size, the inhalation hazard from uranium oxide powders is reduced. It is generally expected that particle size will increase upon heating (Belle 1961). Heating UO_3 powder to form U_3O_8 has been shown to reduce the surface area, indicating larger particles. This effect seems to be more significant at temperatures in excess of 500°C (Harrington and Ruehle 1959). Heating UO_2 powder at 800°C also decreased the surface area (Smith and Leitnaker 1971).

Much of the current Oak Ridge National Laboratory (ORNL) inventory of ^{233}U oxides was prepared by heating pregranulated ammonium diuranate (ADU) for 6 h at an oven temperature of 800°C. The particle size distribution for these powders shows that greater than 90% of the particles have less than 10 microns equivalent of spherical diameter (Parrott, Sr. et al 1979). Therefore, the magnitude of particle size increase may be significantly more dependent on the characteristics of the starting material than on temperatures in the 500-800°C range.

2.3 STABILITY

Conversion to U_3O_8 creates a more stable chemical form of uranium than other oxides. Upon heating in an oxidizing atmosphere above 650°C, all other uranium oxides (and many other uranium compounds) decompose or convert to U_3O_8. Furthermore, oxygen lost from heating U_3O_8 above 800°C is rapidly replaced upon cooling (Katz, Seaborg, and Morss 1986). Because of this chemical stability, U_3O_8 is the preferred storage form.

3. HEATING EFFECTS ON VOLATILES

3.1 MOISTURE

There has been a continuing concern that moisture and other volatiles theoretically can produce pressurizing gases during long-term, sealed storage via radiolysis. Reduction of this potential source of pressurization is a primary reason for treating the uranium oxides. Heating uranium oxide will reduce moisture content to less than 0.5 wt % and similarly reduce equivalent quantities of residual species (e.g., hydrates), which might produce pressurizing gases. The 0.5 wt % specification is a generally accepted limit that is reasonable to achieve and for which no negative affects have been identified. Reducing the amount of moisture present also reduces the potential for and rate of container corrosion.

Free water is eliminated during heating at temperatures above 100°C (i.e., simple evaporation in a vented vessel). The three principal uranium oxides (UO_2, UO_3, and U_3O_8) all form hydrates. However, UO_2 and U_3O_8 form hydrates only when prepared via a precipitation reaction. On the other hand, UO_3 can form hydrates directly through reaction with H_2O between temperatures of 5 and 75°C (Vdovenko 1960). Heating above 400°C converts hydrated UO_3 to dry UO_3 by driving off all waters of hydration (Harrington and Ruehle 1959). The UO_2 powders fabricated at Oak Ridge National Laboratory (ORNL) have moisture contents between 0.10 and 0.36 wt %, which is below the specified 0.5 wt % (Parrott, Sr. et al. 1979).

The potential for moisture readsorption (during prolonged interim ambient storage before packaging) is reduced by heating. The potential for moisture readsorption is proportional to the surface area. When oxides are stabilized at higher temperatures, the surface area tends to be reduced, thus reducing the potential for readsorption. For stabilized UO_2 and U_3O_8, moisture does not tend to be readsorbed in significant amounts. However, UO_3 hydrates may be able to reform under direct exposure to H_2O below 60°C (Harrington and Ruehle 1959).

3.2 OTHER VOLATILES

Uranyl nitrate, $UO_2(NO_3)_2$, which generally appears with multiple waters of hydration (up to six), is the principle intermediary for the purification of ^{233}U oxides from thorium fuel (Storch 1999). Above 300°C, the uranyl nitrate is denitrated. Since all ^{233}U oxides produced by this method were treated above 300°C, no uranyl nitrate is expected to be present. The conversion to U_3O_8 will eliminate any postulated residual uranyl nitrate.

With the exception of materials existing as part of the packaging (e.g., elastomeric gaskets in container lids, plastic bagging around closed inner containers, or plastic inner containers), there is no known significant quantity of organics in existing ^{233}U materials. If moisture and organics are eliminated, then radiolytic gases cannot be generated.

4. HEATING EFFECTS ON SELECTED URANIUM OXIDES

4.1 $^{233}UO_2$

$^{233}UO_2$ is one of three prevalent, stable, uranium oxides that is present in the inventory. In an oxidizing atmosphere, starting from 300°C, UO_2 can be oxidized to form U_3O_8 via the following reaction (Belle 1961, Bowie 1970, and Katz, Seaborg, and Morss 1986):

$$3UO_2 + O_2 \xrightarrow{300-700°C} U_3O_8$$

The rate of this reaction is a function of particle size, and the temperature requirement will change accordingly. Generally, the reaction rate is fast, and UO_2 may even be pyrophoric when the particle size is very fine. When the particle size is large, the O:U ratio steadily increases as a function of exposure time to oxygen. UO_2 with particle size diameters of 0.05–0.08 μm can take up appreciable amounts of oxygen, while at particle diameters above 0.2–0.3 μm, controlled reaction can be maintained while oxidizing UO_2 to U_3O_8 (Katz, Seaborg, and Morss 1986).

4.2 $^{233}UO_3$

Five distinct crystalline modifications (structural isomers) of $^{233}UO_3$ have been found. In Table 2, these five crystalline modifications are shown along with several of their properties.

Table 2. Crystalline uranium trioxides

Phase	Color	Structure	UO_3 decomposition temperature in air (°C)
α	Brown	Hexagonal	450
β	Red, orange	Orthorhombic	530
γ	Yellow	?	650
δ	Reddish-brown	Cubic	400
ε	Red	?	400

Source: Belle 1961

The decomposition of ADU at 450–500°C is the mechanism by which β–UO_3 is formed (Hoekstra and Siegel 1961). Since the UO_3 currently in inventory was formed by this method (Burney 1966), the β–UO_3 crystalline modification is of primary interest. The β–UO_3 begins to decompose from the 3.0 U:O ratio at 530°C, but it does not completely convert to the 2.67 U:O ratio of U_3O_8 until it approaches about 650°C (Hoekstra and Siegel 1961). Since the β–UO_3

predominates the current inventory, a temperature of at least 650°C is needed for conversion to U_3O_8.

The rate of the reaction

$$3UO_3 \longrightarrow U_3O_8 + \tfrac{1}{2}O_2$$

is rapid at elevated temperatures. Figure 1 shows the theoretical rate based on reported rate constants for the range 600-650°C (Harrington and Ruehle 1959). At 650°C, the reaction is essentially complete in 1 hour. Based on the trend with temperature, the reaction should be even faster at higher temperatures.

4.3 $^{233}U_3O_8$ AND OTHER OXIDES

Heating $^{233}U_3O_8$ drives off any existing free water. However, there is an upper limit as to what temperature U_3O_8 can be heated. At atmospheric pressure, U_3O_8 is stable up to about 800°C; above this temperature, oxygen is gradually lost, favoring $U_8O_{21 \pm x}$ (Hoekstra, Siegel, and Gallagher 1970) having a stoichiometry range of about $UO_{2.61}$ to $UO_{2.67}$. Other work considers this latter phase to be substoichiometric U_3O_{8-x} (Cordfunke 1969, Ackerman and Chang 1973).

A melting point of U_3O_8 cannot be determined because when U_3O_8 melts, oxygen is evolved. At higher temperatures in an oxidizing atmosphere, starting at 925°C, U_3O_8 may "vaporize" (presumably, this means measurable vapor pressure), forming mainly monomeric gaseous UO_3, an undesirable product (Powers, Welch, and Trice 1949 and 1961).

Though it is unlikely that any significant amount of higher uranium oxides are present, it is useful to note that all higher uranium oxides and many other uranium compounds decompose to U_3O_8 above 650°C (Katz, Seaborg, and Morss 1986).

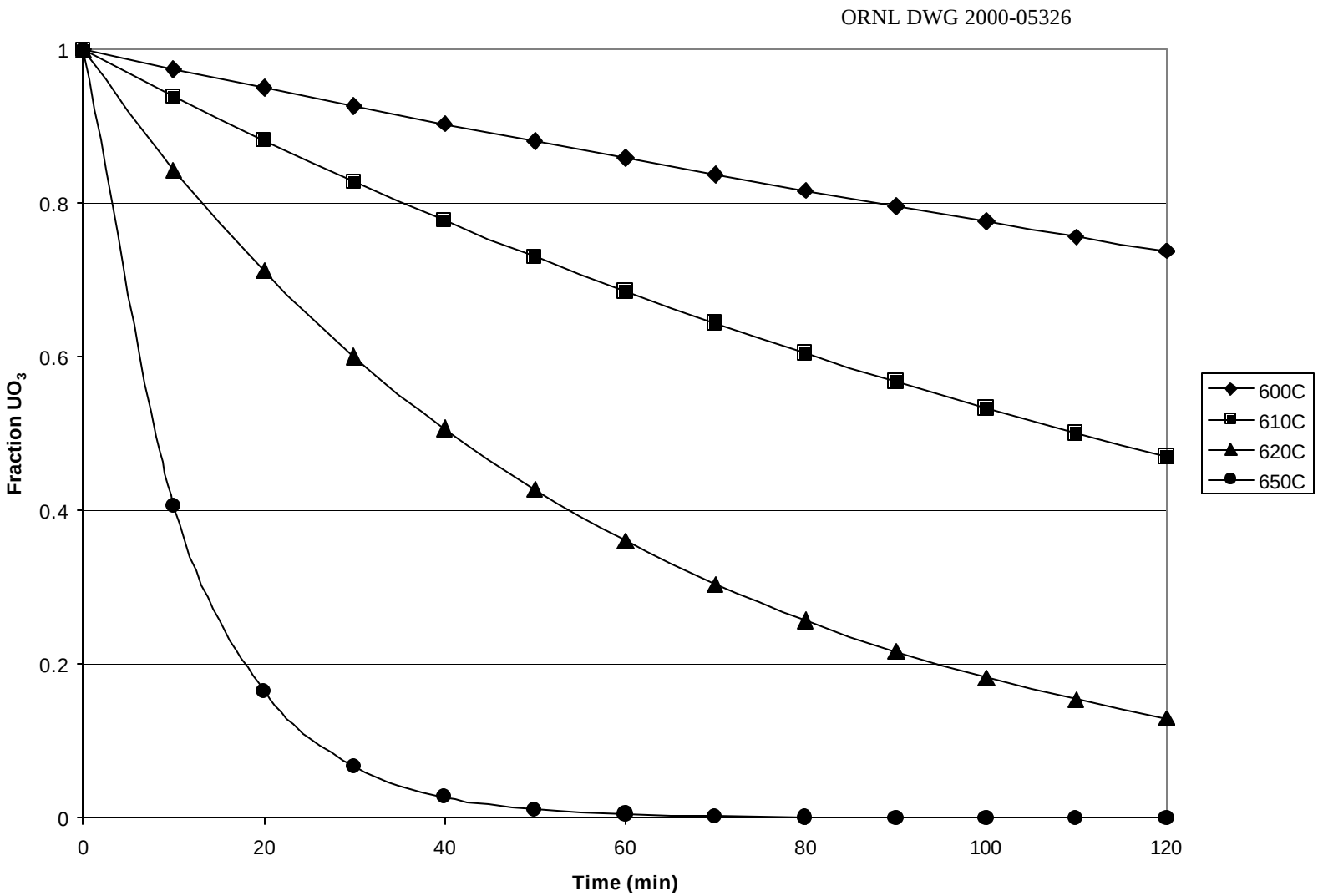

Fig. 1. Rate of decomposition of UO_3 to U_3O_8 in air.

5. SELECTION OF A STABILIZATION TEMPERATURE

Figure 2 illustrates a thermal profile of selected uranium oxides. It is a summary of the reactions of the uranium oxides when heated for conversion to U_3O_8. The standard boiling point of water is used as a baseline.

Heating at a temperature of 700°C is sufficient to convert both the UO_2 and UO_3 to the more stable U_3O_8. However, since some UO_2 may still be present at 700°C, a higher temperature of 750°C is chosen to ensure a more complete reaction within a reasonable heating time. From a practical perspective, this might translate into an operating specification of 750 ± 25°C to ensure that the material is exposed to a greater than 700°C environment.

Since U_3O_8 is stable up to 800°C, existing U_3O_8 will not be affected by this heating— except for the release of moisture, other residual volatiles, and the elimination of residual reactive uranium species. It also will serve to convert any other uranium oxides to U_3O_8. Heating U_3O_8 above 800°C for further stabilization introduces undesirable U_3O_{8-x} oxygen-deficient species into the system. Therefore, the range 750 ± 25°C achieves all the desired stabilization conditions while avoiding undesirable conditions.

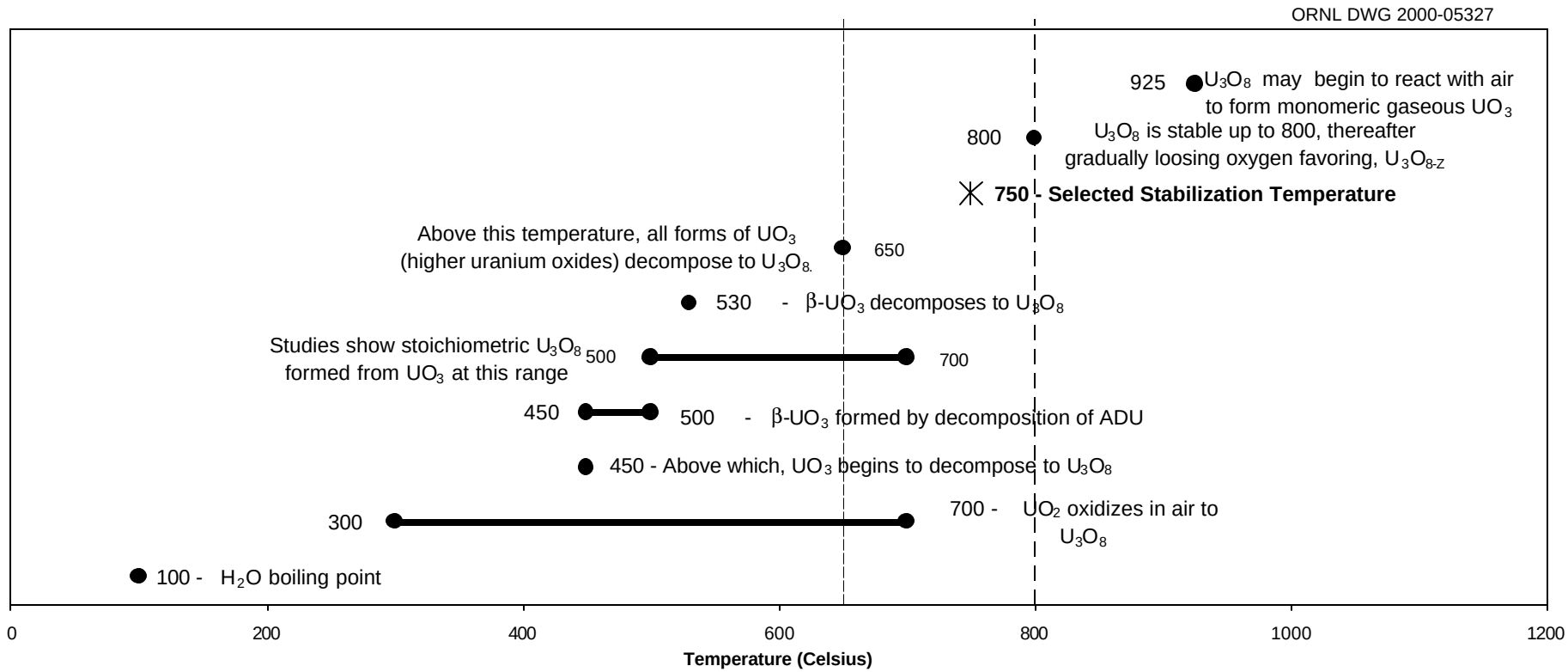

Fig. 2. Thermal profile of selected uranium oxides.

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139. Rodney Lehman, U.S. Department of Energy, DP-24, 19901 Germantown Rd., Germantown, MD 20874

140. Leroy Lewis, Locked Martin Idaho Technologies Company, P.O. Box 1625, Idaho Falls, ID 83415

141. Cary Loflin, U.S. Department of Energy, P.O. Box 5400, Albuquerque, NM 87185-5400

142. Virgil Lowery, III, U.S. Department of Energy, EM-41, 19901 Germantown Rd., Germantown, MD 20874

143. Kathy Martin, U.S. Department of Energy, DOE/GC-52, 1000 Independence Ave., S.W., Washington, D.C. 20585

144. Herbert Massie, U.S. Defense Nuclear Facilities Safety Board, Suite 700, 625 Indiana Ave., N.W, Washington, D.C. 20004

145. T McLaughlin, Los Alamos National Laboratory, ESH-6, P.O. Box 1663, Los Alamos, NM 87545

146. Don McWhorter, Westinghouse Savannah River Company, Savannah River Site, Bldg. 704-F, Aiken, SC 29808

147. Dave Michlewicz, U.S. Department of Energy, DOE/ER-7, 1000 Independence Ave., S.W., Washington, D.C. 20585

148. Lawrence E. Miller, U.S. Department of Energy, DOE/NE-40, 19901 Germantown Rd., Germantown, MD 20874

149. Ed Moore, Westinghouse Savannah River Company, Savannah River Site, Bldg. 773-41A, Rm. 125, P.O. Box 616, Aiken, SC 29808

150. Jim Nail, Lockheed Martin Idaho Technologies Company, P.O. Box 1625, Idaho Falls, ID 83415

151. Dave Neiswander, Advanced Integrated Management Services, 702 S. Illinois Avenue, Suite B-203, Oak Ridge, TN 37830

152. Marty Newdorf, U.S. Department of Energy, DOE/NN-61, Forrestal Bldg., Rm. 6G-050, 1000 Independence Ave., S.W., Washington, DC 20585

153. Jon Nielsen, Los Alamos National Laboratory, P.O. Box 1663, Los Alamos, NM 87545

154. David Nulton, U.S. Department of Energy, DOE/NN-61, Forrestal Bldg., Rm. 6G-050, 1000 Independence Ave., S.W., Washington, D.C. 20585

155. Maureen O'Dell, U.S. Department of Energy, EM-32, 19901 Germantown Rd., Germantown, MD 20874

156. H.B. Peacock, Westinghouse Savannah River Company, Savannah River Site, Bldg. 773-A, Aiken, SC 29808

157. Lee Peddicord, Texas A&M University, Strategic Programs, 120 Zachry, College Station, TX 77843-3133

158. David Peeler, Westinghouse Savannah River Company, Savannah River Site, Bldg. 773-43A, Aiken, SC 29808

159. Per F. Perterson, University of California-Berkeley, Department of Nuclear Engineering, Berkeley, CA 94720-1730

160. Tish Price, U.S. Department of Energy, Rm. 700 N, 1301 Clay St., Oakland, CA 94612

161. D.R. Rhoades, U.S. Department of Energy, DP-24, 1000 Independence Ave., S.W., Washington, D.C. 20585-0002

162. Susan B. Roth, University of California, Nuclear Materials Stockpile Management Division, Nuclear Materials Management Office, MS E524, P.O. Box 1663, Los Alamos, NM 87545

163. Greg Rudy, Westinghouse Savannah River Company, Acting Manager, Suvannah River Operations Office, Road 1, Aiken, SC 29802

164. S.S. Sareen, TRW, Suite 800, 2650 Park Tower Dr., Vienna, VA 22180

165. Thomas F. Severynse, Westinghouse Savannah River Company, Nuclear Materials Stabilization Program, Bldg. 704-7, Aiken, SC 29808

166. Linda Seward, Idaho National Environmental and Engineering Laboratory, P.O. Box 1625, Idaho Falls, ID 83415

167. Alvin Solomon, Purdue University, School of Nuclear Engineering, West Lafayette, IN 47907-1290

168. Jerry Stakebake, U.S. Department of Energy, 10808 Highway 93, Unit A, Golden, CO 80403

169. Robert Stallman, U.S. Department of Energy, 850 Energy Dr., Idaho Falls, ID 38401

170. Kent Sullivan, Westinghouse Savannah River Company, Savannah River Site, Bldg. 773-41A, Aiken, SC 29808

171-180. Soe Thein, Advanced Integrated Management Services, 702 S. Illinois Avenue, Suite B-203, Oak Ridge, TN 37830

181. Dudley Thompson, U.S. Defense Nuclear Facilities Safety Board, Suite 700, 625 Indiana Ave., N.W, Washington, D.C. 20004

182. Jon Thompson, U.S. Department of Energy, MD-4, Forrestal Bldg., Rm. 6G-081, 1000 Independence Ave., S.W., Washington, D.C. 20585

183. Rich Tontodonato, U.S. Defense Nuclear Facilities Safety Board, Suite 700, 625 Indiana Ave., N.W, Washington, D.C. 20004

184. John Tseng, U.S. Department of Energy, DOE/EM-66, Forrestal Bldg., Rm. GA-242, 1000 Independence Ave., S.W., Washington, D.C. 20585

185. Bruce Twining, Manager, Albuquerque Operations Office , Kirkland Air Force Base, Pennsylvania & H Street , Albuquerque, NM 87116

186. Terry S. Vail, Westinghouse Hanford Company, R2-54, P.O. Box 1970, Richland, WA 99352

187. Richard VanKonynenburg, Lawrence Livermore National Laboratory, MS-L269, P.O. Box 808, Livermore, CA 94551

188. K.E. Waltzer, U.S. Department of Energy, Savannah River Site, Bldg. 703-F, Aiken, SC 29808

189. John Wilcynski, Manager, Idaho Operations Office, 765 Lindsay Blvd., Idaho Falls, ID 83401

190. Jeff Williams, Idaho National Environmental and Engineering Laboratory, P.O. Box 1625, Idaho Falls, ID 83415

191. M. Williams, Louisiana State University, Nuclear Science Center, Baton Rouge, LA 70803

192. C.R. Wolfe, Westinghouse Savannah River Company, Savannah River Site, Bldg. 773-A, Aiken, SC 29808

193. Jesse L. Yow, Lawrence Livermore National Laboratory, University of California, 7000 East Ave., Livermore, CA 94550

194. Loong Yong, Advanced Integrated Management Services, 702 S. Illinois Avenue, Suite B-203, Oak Ridge, TN 37830

195. Office of Assistant Manager of Energy Research and Development, P.O. Box 2008, DOE-ORO, Oak Ridge, TN 37831-6269

196. Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831