PNL-5747
UC-41

6A

Radioactive Particulate Release Associated with the DOT Specification 6M Container under Hypothetical Accident Conditions

J. M. Taylor
P. J. Raney

February 1986

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute

Battelle

PNL-5747

DISCLAIMER

PACIFIC NORTHWEST LABORATORY
operated by
BATTELLE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC06-76RLO 1830

Printed in the United States of America
Available from
National Technical Information Service
United States Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22161

NTIS Price Codes
Microfiche A01

Printed Copy

Pages	Price Codes
001-025	A02
026-050	A03
051-075	A04
076-100	A05
101-125	A06
126-150	A07
151-175	A08
176-200	A09
201-225	A010
226-250	A011
251-275	A012
276-300	A013

PNL-5747
UC-41

RADIOACTIVE PARTICULATE RELEASE ASSOCIATED WITH THE DOT SPECIFICATION 6M CONTAINER UNDER HYPOTHETICAL ACCIDENT CONDITIONS

J. M. Taylor
P. J. Raney

February 1986

Prepared for
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Richland, Washington 99352

SUMMARY AND CONCLUSIONS

A testing program was conducted by the Pacific Northwest Laboratory[a] to determine the leakage of depleted uranium dioxide powder (DUO) from the inner containment components of the U. S. Department of Transportation's (DOT) specification 6M container under hypothetical accident conditions. Depleted uranium dioxide was selected as a surrogate for plutonium oxide because of the similarities in the powder characteristics, density and particle size and because of the special handling and special facilities required for plutonium oxide.

The DUO was packaged inside food pack cans in three different configurations inside the 2R vessel of the 6M container. The gas leak rates of the food pack cans tested ranged from $<6 \times 10^{-4}$ atm cc/min to ~1320 atm cc/min. A test was also conducted with a leaky 2R vessel (~110 atm cc/min) that was placed inside a sealed outer container and loaded with DUO powder. The different packaging configurations were subjected to 30-foot drops, 40-inch drops onto a 6-inch-diameter cylinder, and to heating at 300°F in a furnace that could be rotated and vibrated. The leakage rate of the DUO from the containment barriers after the impact and heating tests was measured using a dissolution technique and a laser fluorometer.

The amount of DUO powder leakage ranged from none detectable ($<2 \times 10^{-7}$ g) to a high of 1×10^{-3} g. The combination of gravity, vibration and pressure produced the highest leakage of DUO. Containers that had hermetic seals (leak rates $<6 \times 10^{-4}$ atm cc/min) did not leak any detectable amount ($<2 \times 10^{-7}$ g) of DUO under the test conditions. Impact forces had no effect on the leakage of particles with the packaging configurations used.

The tests showed that when the gas leak rate is below 96 atm cc/min, the amount of particulate material that could be transmitted through the leak sites would be less than the allowable release limits ($<3.5 \times 10^{-5}$ atm g/h) of plutonium (10 C.F.R. Part 71.51).

(a) Pacific Northwest Laboratory is operated by Battelle Memorial Institute for the U.S. Department of Energy.

CONTENTS

FIGURES

TABLES

RADIOACTIVE PARTICULATE RELEASE ASSOCIATED WITH THE DOT SPECIFICATION 6M CONTAINER UNDER HYPOTHETICAL ACCIDENT CONDITIONS

INTRODUCTION

Since its introduction in the late sixties, the U. S. Department of Transportation's (DOT) specification 6M container (Figure 1) has been used to ship large quantities of radioactive materials. The safety record associated with using the 6M container has been outstanding(a). However, the U.S. Nuclear Regulatory Commission (NRC) expressed concern to the DOT that the containment system of the 6M container may not be adequate to meet current regulations (see Appendix A). In particular, the NRC indicated that the requirements of 10 CFR Part 71.52 concerning no loss or dispersal of radioactive material to a sensitivity of A_2(b) quantity per week under hypothetical accident conditions has not been demonstrated.

Sandia National Laboratories was commissioned by the U.S. Department of Energy to compile a summary analysis report that would provide the technical basis and justification that the DOT 6M container complies with regulatory requirements. Sandia asked the Pacific Northwest Laboratory (PNL) to study whether the inner packaging components could contain particulate material within the stated requirement. PNL subjected the 6M container to hypothetical accident tests and measured the particulate release.

The purpose of this report is to describe the results of these tests. The following sections present the background information, and the test methods and experimental procedures used to determine particle leakage. In addition, a packaging procedure for dispersible plutonium was developed as a result of this study and has been included in Appendix B.

(a) No accidents or incidents involving the DOT specification 6M container could be found from 1971 to 1984 on the Radioactive Incident Report Data Base (Emerson and McClure 1983).

(b) For a definition of A_2 quantity see 49 CFR 173.403b.

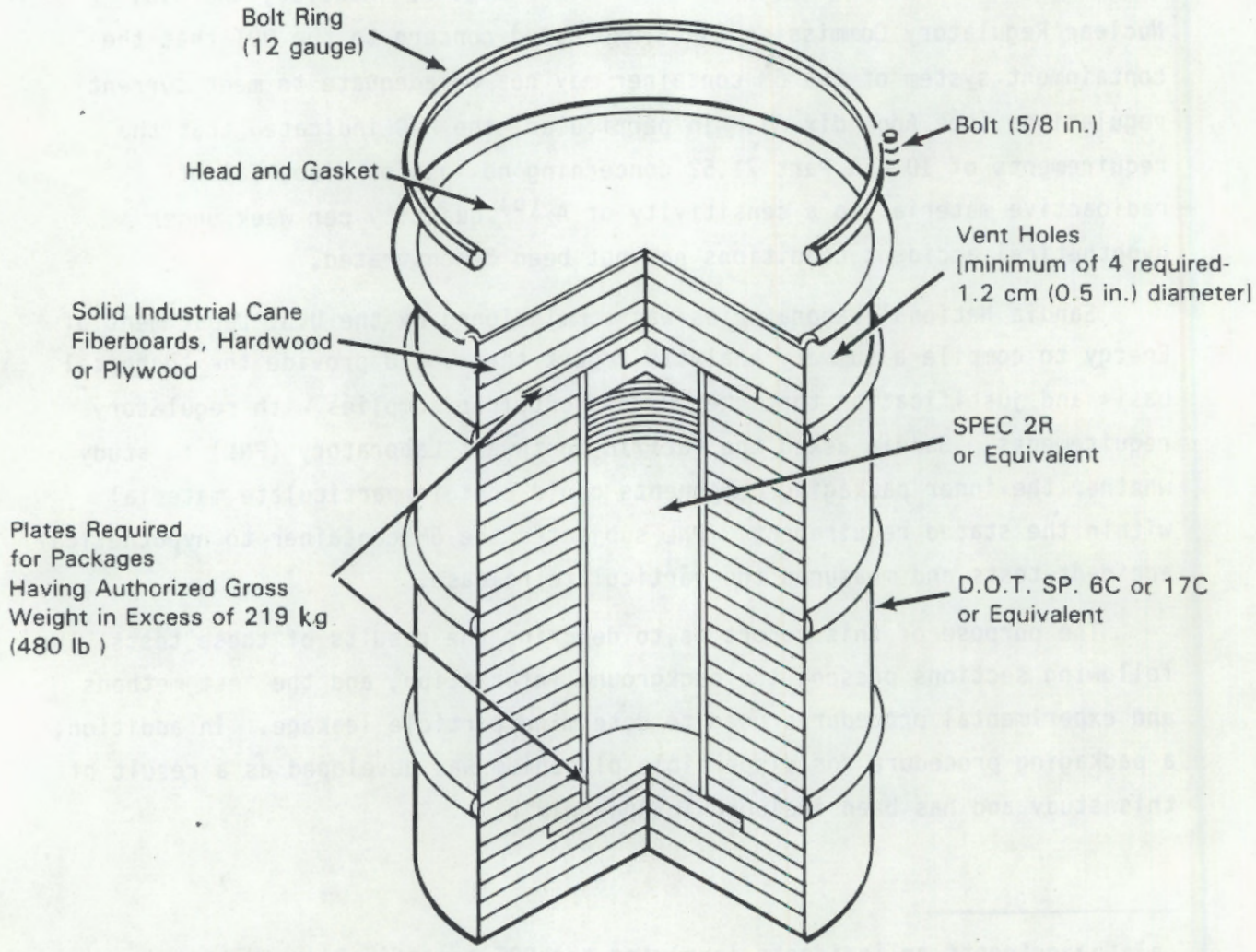

FIGURE 1. DOT Specification 6M Container

BACKGROUND

Containers that are approved for packaging sizable amounts of radioactive materials must meet certain test criteria, such as hypothetical accident conditions. These test conditions involve putting the container through impact, puncture, heating and immersion tests and assessing the amount of leakage from the containers after the test sequence. The test requirements are described in 10 CFR Part 71.73.

If the container does not leak radioactive material exceeding a total amount of A_2 in one week (6 A_2 x 10^{-3}/h), the container system will meet the leakage test requirement. The NRC has indicated (NRC 1975) that the ANSI N14.5 standard (ANSI 1977) describes acceptable test methods to demonstrate radioactive material leakage rates below the values listed above. ANSI N14.5 describes methods for converting maximum permissible material leakage rates to that of a gas leak rate. The standard defines leak tightness as a gas leak rate below 10^{-7} atm cc/sec (6 x 10^{-6} atm cc/min).

In 49 CFR 173.417(b)(2), it states that radioactive material in normal form shall be packaged in one or more tightly sealed metal cans or polyethylene bottles within a DOT specification 2R containment vessel. Metal food pack cans are commonly used to package solid radioactive material in the 6M container. When dispersible or powder type materials are packaged in the 6M container, a minimum of two hermetically sealed metal cans, one nested inside the other, are usually used to contain the material. According to a study by Taylor (1985), when the food pack cans were properly protected and sealed and when the threaded closure of the 2R vessel was luted with a silicon rubber compound, the components leaked less than 6 x 10^{-4} atm cc/min of air under accident conditions.

As a result of Taylor's study (1985), it was felt that a gas leak rate of less than 6 x 10^{-4} atm cc/min would be small enough to contain particulate radioactive material under accident conditions. However, the NRC defines leak

tightness as 10^{-7} atm cc/sec (6×10^{-5} atm cc/min) under a differential pressure of one atmosphere. In comparing the leakage rate of powders with that of gases, it could be assumed that gases would leak through openings or leak paths that may be impassable for powders. The leak site may be too small for the powder particles to pass through or the particles hang up or stack up as they try to pass through the opening and plugging occurs.

Consequently, using gas leak criteria to assess the allowable releases of solid radioactive material could be considered too conservative. The NRC has indicated (Lake 1983) that leakage tests other than those specified in ANSI N14.5 would be acceptable providing the method used has high enough sensitivity and avoids false acceptance of containment.

Correlating gas leakage with radioactive material release for fine powders has been studied (Sutter et al. 1980; Curren and Bond 1980; Yesso et al. 1980), but no consistent correlation between gas flow and particle flow through orifices and capillaries could be established because the openings became plugged (Sutter et al. 1980). When vigorous vibration was used to prevent plugging, a particle flow was established under certain conditions (Curren and Bond 1980). The powder leak studies provided a conservative upper limit for leakage of particulate material through very small openings. However, the conservative limits established may not describe the actual leakage that occurs with radioactive material containers under accident conditions. When actual packaging configurations can be tested for particulate releases, a more realistic leakage assessment can be made.

The 6M container, because of its size and weight, can be handled easily for testing, plus the 2R vessel can be removed from the drum and heated separately without subjecting the whole container to the fire test. Consequently, the 6M container is a convenient container to determine radioactive material releases under accident conditions.

Particulate release studies have been conducted using the 6M container. Blankenship (1980) determined the release of OUO powder from food pack cans inside the 2R vessel after subjecting the 6M container to a 30-foot drop and subsequently heating the 2R vessel to 250°F. In Blankenship's method of analysis, the outside of the food pack cans was smeared before and after the

testing. The smears were analyzed for total activity using an alpha counting technique. UO_2 powder release measurements were also made on another drum-type container called the plutonium air transportable package, model PAT-1 (Office of Nuclear Material Safety and Safeguards 1978). The method of analysis used to determine leakage was similar to that of Blankenship. The surfaces of the inner containers that contained the UO_2 powder were smeared before and after testing. The smears were analyzed using a fluorimeter technique. In both studies, the detection limits were mentioned, but the accuracy of the methods was not given. Also, the efficiency of removing the UO_2 from the smeared surfaces was not discussed.

The Blankenship (1980) report implied that all the UO_2 that escaped from the containers containing the powder was deposited on the surfaces of those containers. Depending on the release mechanisms, the powder could have been deposited on packing material (vermiculite, plastic bags) or on the inner surfaces of the 2R vessel. The surfaces of the packaging material and the 2R vessel were not analyzed, and no reason for this omission was given.

When Lake (1983) evaluated containment systems, he considered the measuring of material loss from a containment system to be an indirect test. He also indicated that in order for an indirect test to determine leakage in a quantitative manner, the test method must have the necessary resolution and accuracy (sensitivity) and must avoid false acceptance. False acceptance could occur either from failure of the contents to find existing leak paths or dispersal and nondetection of the released radioactive material. Thus, to satisfy the conditions mentioned by Lake (1983), PNL used a test method that had high sensitivity and avoided false acceptance of containment. The method is described in the next section.

TEST METHOD AND EXPERIMENTAL PROCEDURE

During the gas leak study (Taylor 1985), a procedure was developed for producing seals that leaked air less than 6×10^{-4} atm cc/min from the 2R vessels under accident conditions. Andersen (1983) reported that leak rates below 6×10^{-4} atm cc/min of air would have leak site pathways of less than 0.008 µm. Finely divided DUO powders usually do not have particle sizes less than 0.4 µm (see Figure 2), whereas PuO_2 powders typically have larger minimum particle sizes. Andersen (1983) reported that, of four samples of PuO_2 powder produced from an oxalate precipitation, no sample analyzed had particles less than 6 µm. Consequently, DUO or PuO_2 powder would not leak out of openings having leak rates less than 6×10^{-4} atm cc/min. Therefore, a 2R vessel that had a leak rate less than 6×10^{-4} atm cc/min would provide absolute confinement for any DUO or PuO_2 powders that could escape from food pack cans inside the 2R vessel even under accident conditions. The amount of powder material released inside the 2R vessel could be determined by dissolving the material and analyzing the solutions. The same procedure could be used on each barrier. A solution could be introduced inside each sealed can (excluding the innermost can containing the powder) and then analyzed.

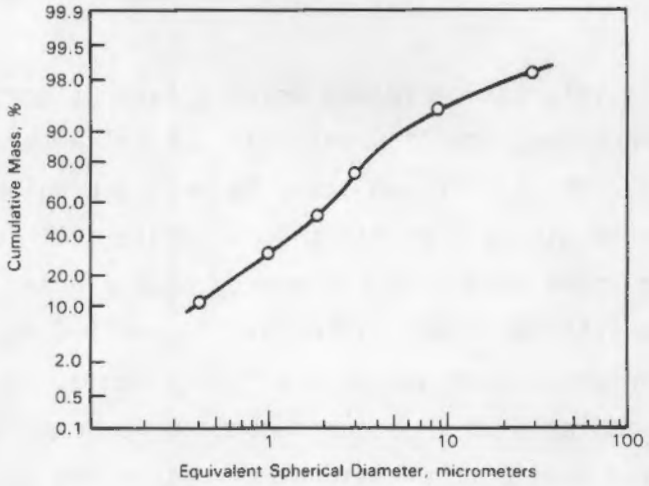

FIGURE 2. DUO Particle Size Distribution

Plutonium oxide powder is the most hazardous of the radioactive materials that is authorized for packaging in the 6M container; however, it was not used in this study because it requires special handling and special test facilities. Acquiring these facilities would have increased the time and costs of the project. Thus, DUO powder was selected because it has a similar density and particle size and should closely relate to the leakage characteristics of PuO_2 powder. In addition, a number of solutions are available that dissolve DUO powders readily and are not corrosive.

The solution selected to dissolve the DUO powder was developed (with some modification) to decontaminate carbon steel components of a primary loop in a production reactor (Mendel 1960). The composition of the solution used was as follows:

0.25M hydrogen peroxide, H_2O_2
0.25M sodium carbonate, Na_2CO_3
0.25M sodium bicarbonate, $NaHCO_3$.

The peroxide bicarbonate (PBC) solution does not corrode steel and has been effective at dissolving uranium and DUO (Ayres 1970). Dissolution studies (Neibaur and Stice 1961) showed that up to 3.2 g/l of DUO is dissolved in the PBC solution at room temperature. To get some idea of the dissolution rate, 25 mg of DUO was added to 50 ml of PBC solution. The solution was gently agitated by hand, and 5-lambda samples at 5-minute intervals were taken and analyzed for uranium. After 10 minutes, all of the DUO powder had been dissolved.

The uranium analysis was performed using a laser fluorometer developed to analyze very low concentrations of uranium in natural waters (Robbins 1978). The instrument uses a small nitrogen laser to excite uranium atoms. The laser supplies monochromatic ultraviolet light to a sample cell containing uranium solutions. Uranium atoms absorbing the energy emit a green luminescence that is read by a photomultiplier tube. Interferring species may exist in the solution that either enhance or quench the fluorescence. Any long-lived green fluorescence from samples high in organic content is compensated for with a balance control while reading the residual or background fluorescence of the

sample before the addition of a proprietary buffered pyrophosphate reagent, "fluran," which causes the uranium to fluoresce. Interfering species that decrease the fluorescence either by absorbing the excitation light or the fluorescence emission are compensated for by a standard addition technique (Saxberg and Kowalski 1979). Standards ranging from 13.67 ppb to 683.5 ppb U were made up to determine the accuracy of the method. The results are presented in Table 1.

The DUO powder used in the tests had a mean particle size of <2 μm. The particle size distribution of the DUO powder (shown in Figure 2) was determined using a sedimentation method. The particle morphology is shown in Figure 3. The bulk density of the DUO powder was 4.9 g/cc. Heating a sample of the DUO powder at 350°F for 2 hours in helium resulted in a 0.47 wt% loss. The weight change was due mainly to the loss of water.

The 6M container is authorized to contain up to 4.5 kg of plutonium material. In the gas leak study (Taylor 1985), a similar packaging configuration was tested, as shown in Figure 4, except that lead shot and sand were used to simulate the radioactive particulate material. The total mass of the lead shot and sand exceeded 4.5 kg. By using spacer places and empty cans as impact absorbers, the product cans were protected and remained undamaged during impact testing. More details on proper packaging assemblies are given in Appendix B.

TABLE 1. Comparison of Analytical Results for Various Standard Uranium Solutions

Concentration of Standard Solution, ng/ml	Average Value of Analytical Samples and Standard Deviation, ng/ml
13.67	13.20 ± 1.03
136.70	136.62 ± 6.48
273.4	260.1 ± 6.5
683.5	600.0 ± 61.6

The intial testing on loaded 6M containers was done using the packaging configuration shown in Figure 4. Both can assemblies had DUO powder in the innermost can. In later tests, only one can was filled with DUO powder, as shown in the packaging configurations in Figures 5, 6, 7, and 8. The No. 3 can, which was stacked on the bottom, was filled with approximately 2.3 kg of dry sand. The cans containing the sand were hermetically sealed and had gas leak rates less than 6×10^{-4} atm cc/min.

The configurations shown in Figures 5 through 8 prevented leakage of the dissolver solution into the can assembly containing the DUO powder. These configurations were used because some of the can assemblies tested had leaky seals. The can lids had been partially crimped onto the can bodies so that they would leak, thus the gas leak rate depended on how well the lids had been crimped.

The gas leak rate was measured by pressurizing the can with nitrogen and measuring the flow of nitrogen with calibrated rotometers. After testing, the 2R vessel was flooded with solution to dissolve any DUO powder that may have been released from the product cans. If the cans had hermetic seals, then the 2R vessel was filled to 90% capacity with solution. If the cans were leaky, then the 2R vessel was partially filled with enough solution to immerse only the hermetically sealed No. 3 can containing the sand. The 2R vessel was then rotated, and the solution immersed the product can for only a short period of

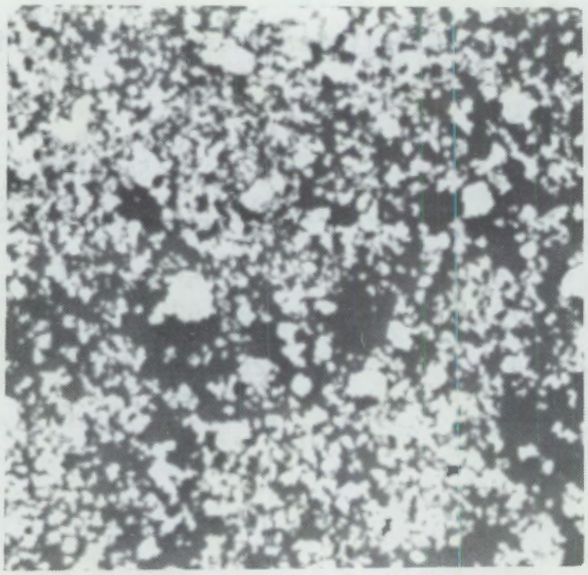

FIGURE 3. Photomicrograph Showing the Particle Size and Morphology of DUO Powder (100 X)

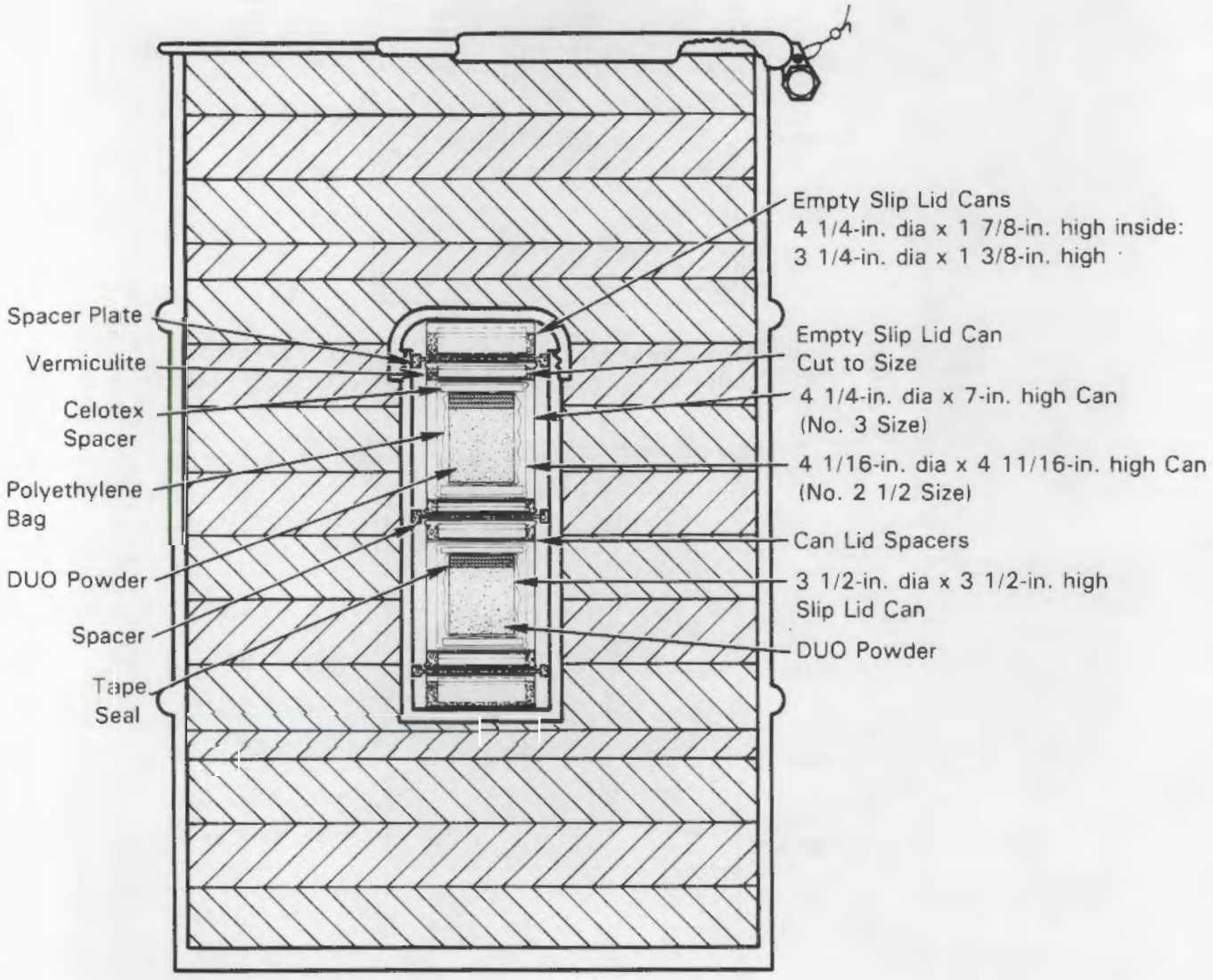

FIGURE 4. Packaging Configuration for DUO Powder Inside a 6M Container with Four Barriers

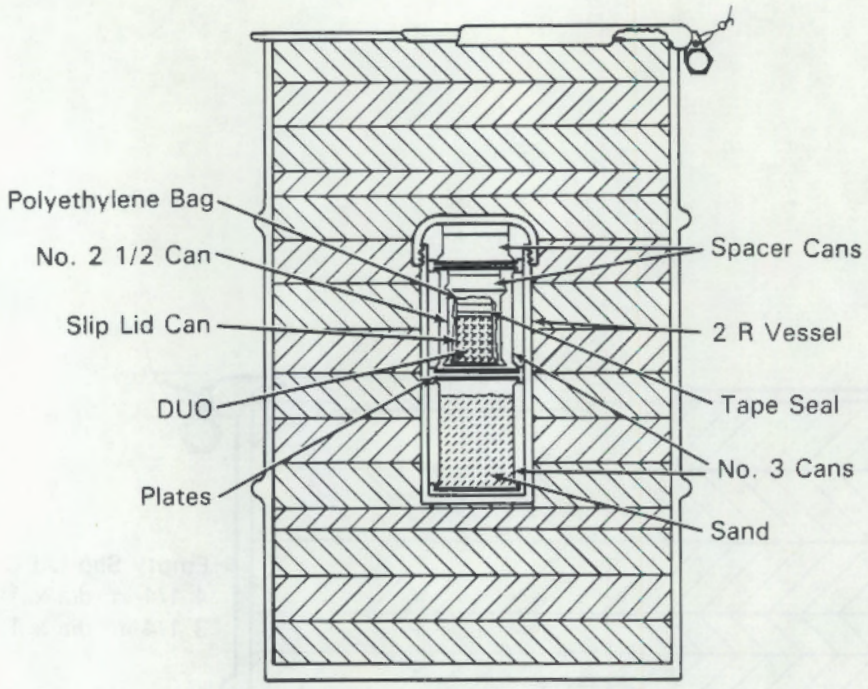

FIGURE 5. Packaging Configuration for Single Container of DUO Powder Inside a 6M Container with Four Barriers

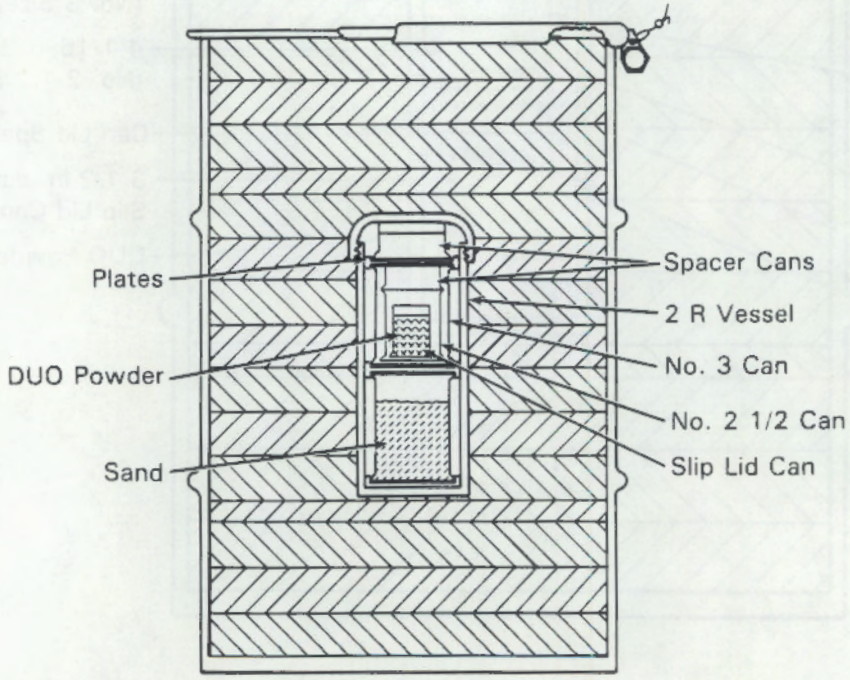

FIGURE 6. Packaging Configuration for Single Container of DUO Powder Inside a 6M Container with Three Barriers

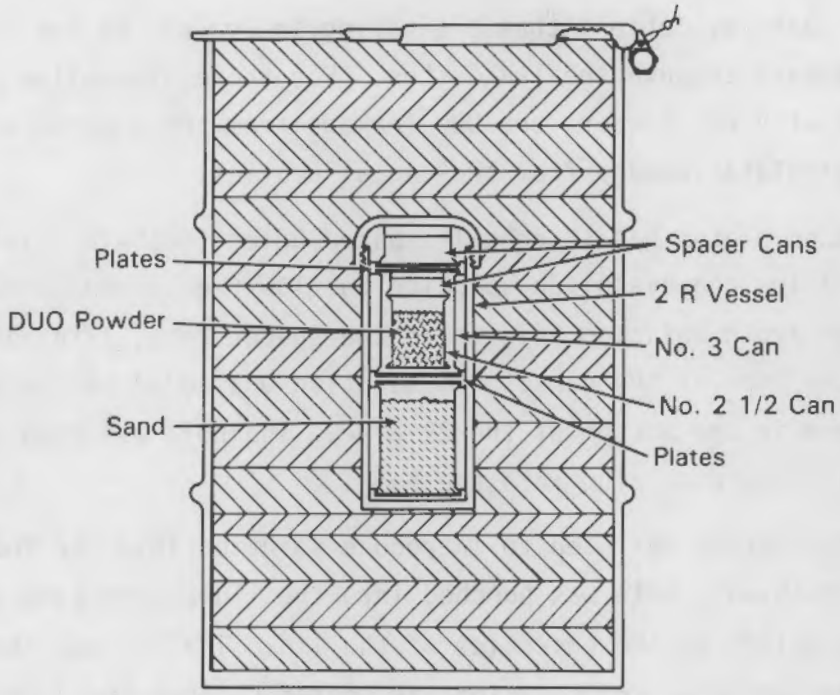

FIGURE 7. Packaging Configuration for Single Container of DUO Powder Inside a 6M Container with Two Barriers

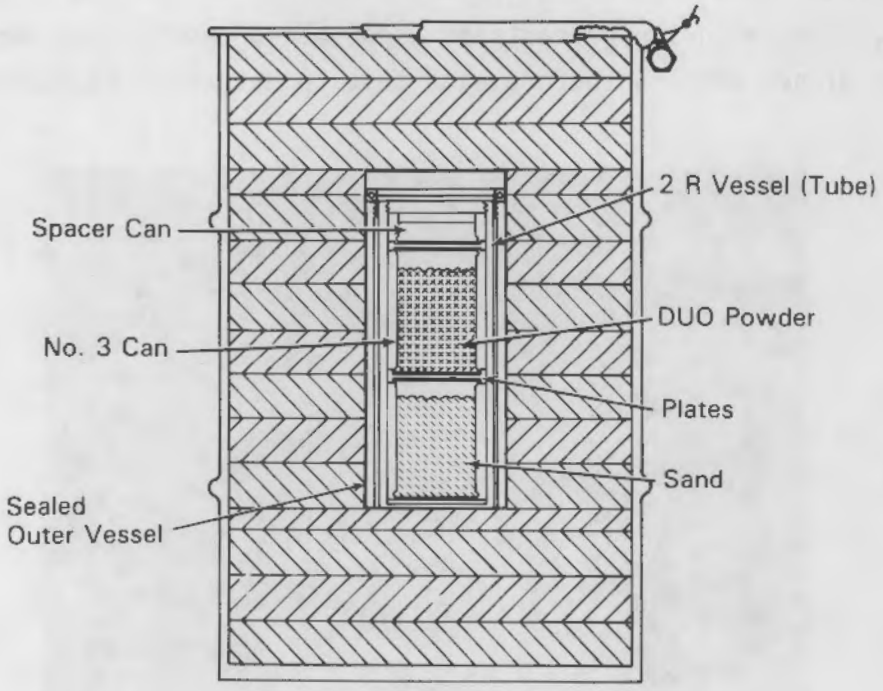

FIGURE 8. Packaging Configuration Used to Contain DUO Powder Inside a Leaky 2R Vessel

time during each revolution, thus preventing in-leakage of the solution by hydrolic pressure through the leak sites. For those assemblies containing only one can with DUO powder, the DUO leakage from the one-can assembly was doubled to simulate leakage from two-can assemblies.

The spacer plates had 12 equally spaced holes (5/16-in. dia) around the perifery to allow the dissolver solution to flow freely and contact all surfaces. To avoid solution hold-up in the spacer cans, 1-in.-dia holes were punched in the lids of the cans. PBC solution was added to the 2R vessel through a hole in the bottom of the 2R pipe. The hole was then sealed by screwing a 1/4-in. pipe plug into the hole.

A similar method was used to introduce solution into the food pack cans. A 7/16-in.-dia hole was punched into the lid of the cans and a 7/16-in. nut was spot welded to the underside of the lid. The lid was then crimp sealed to the can body, and solution was poured through the hole in the can. The hole was sealed by screwing a threaded plug with an "O" ring gasket into the nut. The plug closure is shown in Figure 9.

To prevent contamination, great care was used in loading the cans with DUO powder. Even with the precautions used, background levels ranged from 3 to 10 ppb. Blanks were run before each test to determine background levels.

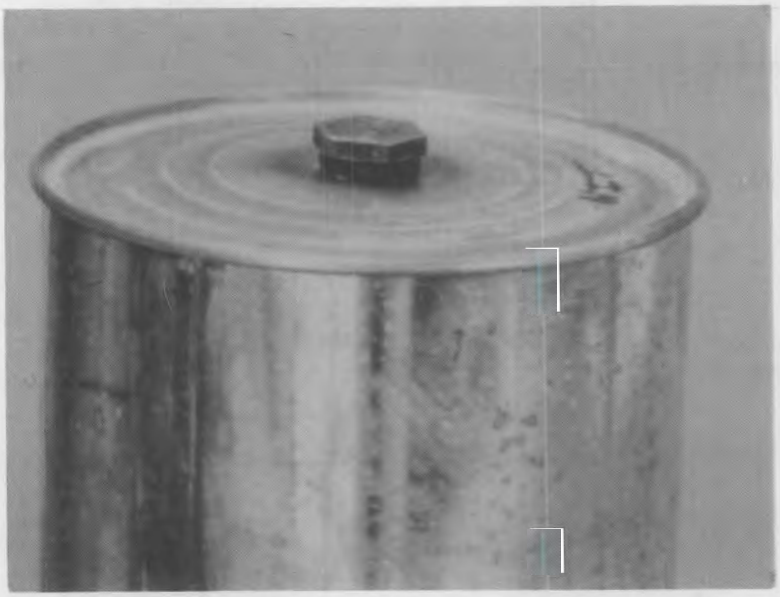

FIGURE 9. Plug Closure Seal Used on Food Pack Cans

After the DUO powder was added to the cans and sealed in the No. 2 1/2 can, the following procedure was used.

1. The No. 2 1/2 can was leak tested and placed in the No. 3 can. A known volume (between 100 to 150 cc) of PBC solution was added to the No. 3 can. All surfaces of the No. 2 1/2 can were contacted with the solution. The No. 2 1/2 can was soaked in the solution for a minimum of 15 minutes. After the can had been soaked, a sample was taken to determine the background level (blank) of the uranium. The can was drained overnight.

2. The No. 3 can was then placed inside the 2R vessel in one of the configurations shown in Figures 4 through 7. The 2R vessel was sealed by applying a silicon rubber compound (see Appendix B for details) to the threads and tightening the cap with 100 ft-lb of torque. The silicon rubber was allowed to cure for at least 20 h.

3. PBC solution was added (between 600 to 1500 cc, depending on can seals) to the 2R vessel after sealing the cap. The solution was sloshed around inside the 2R vessel by rotating it through 360° for 15 minutes. The solution was then sampled and analyzed for uranium, and the 2R vessel was drained overnight.

4. After draining, the 2R vessel was plugged and bubble tested for leaks. The sealed 2R vessel was placed in the 6M drum and taken to the drop site.

5. The loaded 6M drum was dropped 30 ft end-on onto a cement pad with a steel plate embedded in it.

6. The 6M drum was then dropped from a height of 40 in. onto a 6-in.-dia by 8-in-long cylinder, impacting the drum at the center of its sidewall.(a)

(a) A 40-in. drop onto a 6-in.-dia cylinder was only done in two of the tests because the damage to the drum and inner packaging was so insignificant that it did not warrant including the drop in each test.

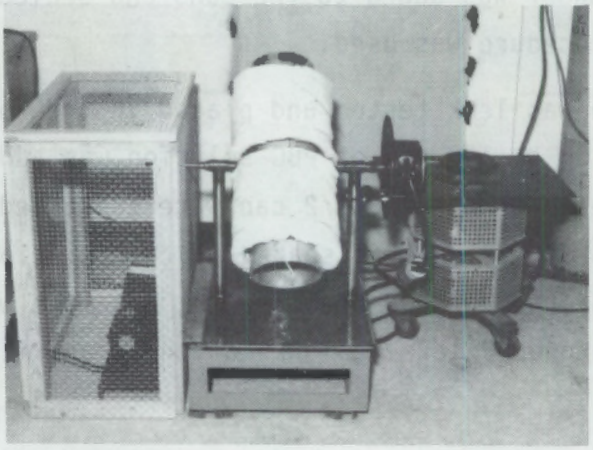

FIGURE 10. Tube Furnace Used to Heat, Tumble and Vibrate 2R Vessels

7. The 2R vessel was removed from the 6M drum and bubble tested. After bubble testing, a filter was attached to the 2R vessel so the vessel would not be pressurized during heating. Then the vessel was placed in a preheated tube furnace (Figure 10) and heated to 300°F for a minimum of 1 hour. The temperature of the 2R vessel was measured by thermocouples attached at the end and center of the vessel. During heating and soaking of the 2R vessel, the tube furnace was rotated 360° at 2 rpm and vibrated. The vibration frequency was 120 hertz with a sinusoidal input that ranged between 0.5 to 0.8 g depending on the position of the tube furnace during rotation. The input g levels approximated the levels observed during the transportation of fuel bundles by trucks (Loewen 1980).

8. After cooling, PBC solution was added (between 600 to 1500 cc depending on can seals) to the 2R vessel, and the vessel was rotated for 15 minutes and sampled.

9. The cans were removed from the 2R vessel and leak tested. After leak testing, PBC solution was added to the No. 3 can (100 to 150 cc) and after thorough mixing, the solution was sampled and analyzed.

To test the response and accuracy of the test procedures used to determine DUO leakage, a very small amount of a uranium standard solution was deposited on the lids of two sealed No. 3 cans. The solution was allowed to air dry on the can lids, and then the cans were assembled in the same

configurations as shown in Figure 4. A known amount of PBC solution was added to the 2R vessel and sloshed around for twenty minutes. A sample was taken and analyzed using the same procedures as for the other tests. The amount of uranium added to the lids was 3.2×10^{-4} g, and the amount determined by analysis was 3.1×10^{-4} g. The method produced results that were accurate to within 3 to 4%.

The same procedure was used for the packaging configuration shown in Figure 8. PBC solution (300 cc) was added to the sealed container before and after the testing.

In some of the tests, the 2R vessel was not rotated and vibrated continuously while it was being heated. In those tests, the 2R was only rotated every 20 or 30 minutes during heating.

RESULTS AND DISCUSSION

The results of DUO powder transmission from metal cans and 2R vessels having gas leak rates ranging from $<6 \times 10^{-4}$ to ~2200 atm cc/min after having been subjected to hypothetical accident conditions is presented in the first part of this section. A comparison of the leakage of DUO powders from metal cans and 2R vessels that have complicated leak paths with orifices and capillaries is discussed in the second half of this section.

RESULTS OF POWDER TRANSMISSION UNDER HYPOTHETICAL ACCIDENT CONDITIONS

In 10 CFR Part 71.51(a)(2), it states that under hypothetical accident conditions, the loss of radioactive material from the container must not exceed a total of an A_2 quantity in one week ($6\ A_2 \times 10^{-3}$ per hour). A_2 values are activity (radioactive) values expressed in curie units. Since uranium and plutonium materials have different specific activities, the release limits were converted to grams of material. Table 2 shows the allowable release limits of various plutonium isotope mixtures given in grams. The plutonium composition with the highest percentage of ^{239}Pu is typical of weapons grade plutonium, while the low percentage of ^{239}Pu is typical of reactor grade plutonium. If the measured leakage of uranium was less than 3.15×10^{-5} g/h after undergoing the testing described, the containment system was considered leak tight.

The results of the tests are given in Table 3. The uranium leakage shown in column 6 of Table 3 was the total uranium released after undergoing all the tests described in column 3. With the exception of test 9, all the tests described in Table 3 exceed the time designation of 1/2 hour at the temperature specified in 10 CFR part 71.73(c)(3). The 2R vessel was heated to temperatures exceeding 300°F for many of the tests. During actual fire tests with the 6M drum (heated to 1475°F), the 2R vessel surrounded by Celotex insulation was heated to about 300°F (Chalfant 1984).

The leakage of uranium was normalized to time periods of one hour; the values are shown in parentheses in column 6 along with the total uranium released. The procedure used to normalize the values was to divide the total release by the time period the 2R vessel was being heated and/or vibrated and

TABLE 2. Allowable Release Limits of Various Plutonium Isotope Composition Under Accident Conditions

Allowable Release, g/h	Isotopes, %				
	238	239	240	241	242
3.15×10^{-5}	1.6	54.7	24.7	12.2	6.7
6.85×10^{-5}	0.1	76.9	19.3	3.2	0.5
1.38×10^{-4}	0.02	93.2	6.1	0.55	0.03

rotated. During this time period, the driving forces of pressure and gravity were available to promote leakage of powder through the barriers. The time to cool the 2R vessel to ambient temperature was not included in the time period even though a pressure differential was present.

The detection level (sensitivity) for the dissolution procedure used had a lower limit of 2×10^{-7}g of uranium. Even though the instrumentation could detect as little as 0.1 ppb of uranium in standard PBC solutions, the smallest difference that could be detected between the blanks and the active sample was 2×10^{-7} g of uranium because of contamination in handling the blanks and active samples and the volume of samples required. This detection level was considered adequate because it was two orders of magnitude less than the allowable amount of uranium that could be released (3.15×10^{-5} g). In tests 1 and 2, the 2R vessel was only rotated periodically during heating. The rotation was done so the powder would contact potential leak sites. All of the cans were deformed to different degrees showing evidence of having been pressurized. The cans in test 2 were deformed more than in test 1 (Figure 11). The higher pressure could have been due to more moisture being present in the test 2 cans.

The polyethylene bags surrounding the slip lid cans inside the No. 2 1/2 can melted during the heat test (Figure 12). The melting of the polyethylene bags at 300-350°F did not produce any significant pressure inside the cans.(a)

(a) A test was performed where 15 grams of a polyethylene bag were placed inside a hermetically sealed can and heated to 350°F for 1 hour. The maximum pressure recorded inside the can was 7.9 psig, which indicated that the pressure increase was due only to the expansion of air and not degradation of the polyethylene.

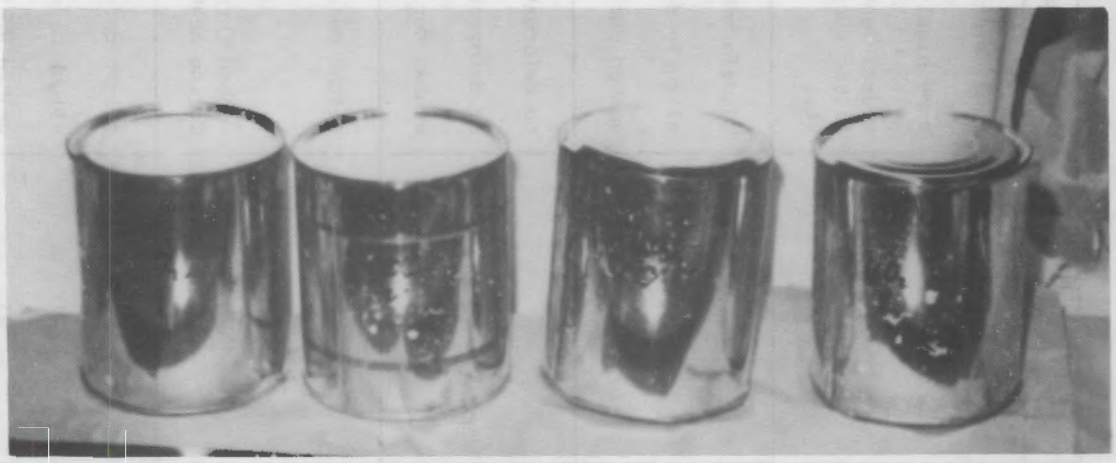

FIGURE 11. Deformation of Cans Due to Pressurization During Heat Test (Note bulged cans on right.)

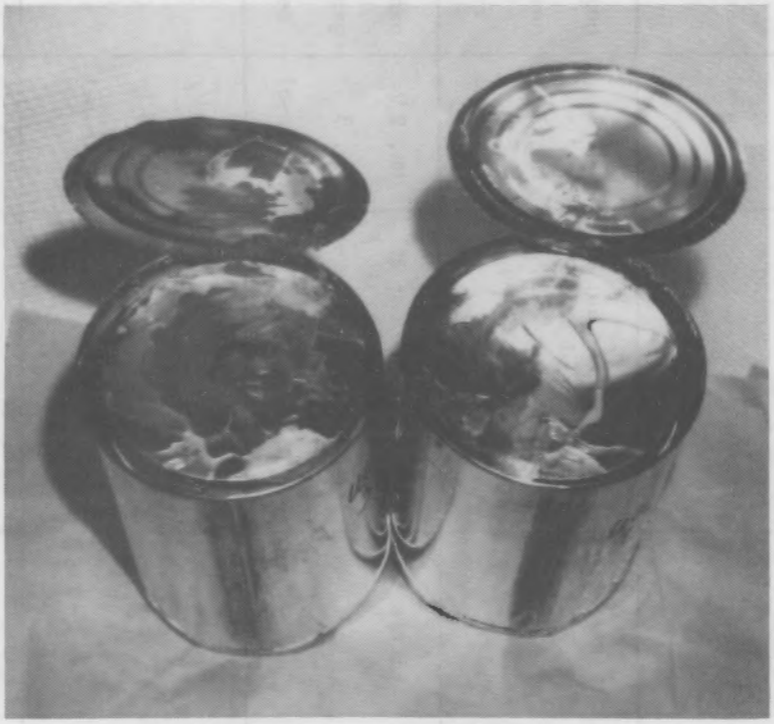

FIGURE 12. Remnants of Polyethylene Bags that Melted During the Heat Test

TABLE 3. Particulate (DUO) Release Data of Inner Packaging Components for the 6M Container Subjected to Hypothetical Accident Tests

Test No.	Packaging Configuration	Test Conditions	Can Type	Gas Leak Rate, cc/min		Uranium Leakage Inside Barriers, g	Comments
				Before	After		
1	Figure 4	30-ft drop end on; 2R vessel heated at 320°F for 3 hours; rotated every 30 min during heating	No. $2\frac{1}{2}$	gas tight	gas tight		cans deformed
			No. 3	gas tight	gas tight	ND[a]	cans highly deformed
			2R vessel	sealed	sealed[b]	ND	vessel vented through filter during heat test
2	Figure 4	30-ft drop end on; 2R vessel heated at 320°F for 1.5 hours; rotated every 30 min during heating	No. $2\frac{1}{2}$	gas tight	gas tight		no deformation
			No. 3	gas tight	gas tight	ND	slightly deformed
			2R vessel	sealed	sealed[b]	ND	vented during heating
3	Figure 5	30-ft drop end on; 2R vessel heated at 315°F for 1.5 hours; rotated and vibrated for 4.5 hours	No. $2\frac{1}{2}$	gas tight	approx. 150		no deformation
			No. 3	gas tight	gas tight	ND	no deformation
			2R vessel	sealed	sealed[b]	ND	vented during heat test
4	Figure 5	30-ft drop end on; 2R vessel heated at 300°F for 2 hours; rotated every 20 min	No. $2\frac{1}{2}$	275	NM[c]		slight deformation
			No. 3	165	NM	8×10^{-6} (4×10^{-6})[d]	
			2R vessel	sealed	sealed	ND	vented through filter during heating
5	Figure 5	30-ft drop end on; 2R vessel heated at 315°F for 2 hours; rotated every 20 min	No. $2\frac{1}{2}$	155	NM		slight deformation
			No. 3	gas tight	NM	ND	slight deformation
			2R vessel	sealed	sealed	ND	vented during heating

(a) ND = none detected (i.e., $<2 \times 10^{-7}$ g).
(b) 2R vessel was vented through a microfilter to generate higher pressures within the metal cans during heating and cooling.
(c) NM = not measured.
(d) Normalized values.

TABLE 3 (contd)

Test No.	Packaging Configuration	Test Conditions	Can Type	Gas Leak Rate, cc/min		Uranium Leakage Inside Barriers, g	Comments
				Before	After		
6	Figure 5	30-ft drop end on; 2R vessel heated at 300°F for 1.5 hours; rotated continuously for 3.5 hours	No. $2\frac{1}{2}$	140	500		slighty bulged
			No. 3	190	190	$2 \times 10^{-6}(6 \times 10^{-7})$(d)	no deformation
			2R vessel	sealed	sealed	ND	vented during heating
7	Figure 6	30-ft drop end on; 40-in. side drop onto 6-in.-dia cylinder; 2R vessel heated to 482°F; held above 375°F for 2 hours; rotated and vibrated for 6 hours during heat test	No. $2\frac{1}{2}$	400	NM		slightly deformed
			No. 3	gas tight	gas tight	$2 \times 10^{-3}(3 \times 10^{-4})$(d)	highly deformed
			2R vessel	gas tight	sealed	ND	vented through filter during heating
8	Figure 7	30-ft drop end on; 2R heated to 347°F; held above 300°F for 3 hours rotated and vibrated for 4.5 hours total	No. $2\frac{1}{2}$	150	2300		very slight deformation
			No. 3	150	NM	$2 \times 10^{-4}(4 \times 10^{-5})$(d)	slightly bulged
			2R vessel	gas tight	sealed	$6 \times 10^{-6}(1 \times 10^{-6})$(d)	vented through filter
9	Figure 7	rotation and vibration for 3 hours	No. $2\frac{1}{2}$	140	140		(no impact or heat test)
			No. 3	100	100	$1.2 \times 10^{-5}(4 \times 10^{-6})$(d)	
			2R vessel	sealed	sealed	$4 \times 10^{-6}(1 \times 10^{-6})$(d)	
10	Figure 8	30-ft drop end on; sealed outer container held at 300°F 2 hours; rotated and vibrated for 5 hours during heat test	No. 3	500	2250		no deformation
			2R vessel	110	2200	$1.2 \times 10^{-3}(2 \times 10^{-4})$(d)	no deformation
			outer container	sealed	sealed	$8 \times 10^{-6}(2 \times 10^{-6})$(d)	vented through filter during heat test

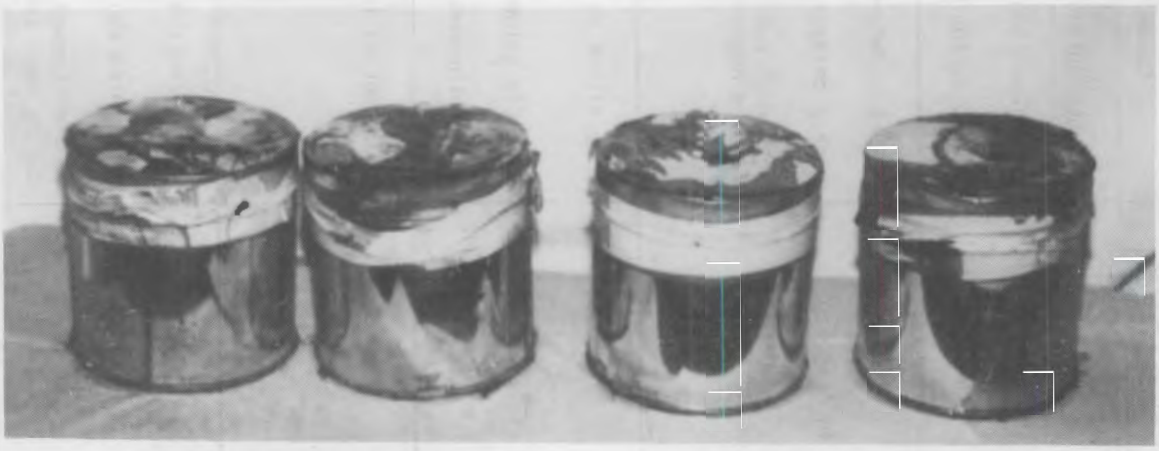

FIGURE 13. Tape Seal After Heat Test on Slip Lid Cans Containing DUO Powder

The tape seal on the slip lid cans was displaced (as shown in Figure 13), but had not discolored or degraded at 350°F. The black material shown on the lid and side wall of the cans is DUO powder.

To promote higher leakage and to avoid the problem of false acceptance, a test (test 3, Table 3) was performed on gas-tight inner containers where the 2R vessel was rotated and vibrated during the heat up and soak cycle. Even though a gas leak (~150 atm cc/min) developed in the No. 2 1/2 can during heating, no transmission of DUO from the No. 2 1/2 to the No. 3 can could be detected. The fact that a leak occurred in the No. 2 1/2 can during the heat cycle indicated the can was under pressure. The No. 3 can in which the No. 2 1/2 can was nested showed no signs of deformation.

Known leak rates were introduced in the No. 2 1/2 cans and the No. 3 can in tests 4 and 5. The 2R vessel was rotated 180° every 20 minutes during the heat tests to mix the powder and to promote leakage of the powder. After the impact and heating tests, the No. 3 cans were examined and showed no damage (Figure 14) except for a slight distortion on the sealed No. 3 can in test 5.

Distortion of the No. 2 1/2 can in test 4 indicated that it had been pressurized to about 20 psig during the heat test (Figure 15). It was estimated that very little powder leaked from the slip lid cans during the

FIGURE 14. Number 3 Cans After Impact and Heat Tests (test 5, left; test 4, right)

FIGURE 15. Distortion of No. 2-1/2 Can Due to Pressure During Heat Test

FIGURE 16. Condition of Tape Seal Showing Leakage of DUO Powder After Leak Test

testing. Only slight breaks in the tape appeared on the slip lid cans in tests 4 and 5, Figure 16. A small amount of uranium (8×10^{-6} g) leaked out of the No. 2 1/2 can into the No. 3 can in test 4, but not in test 5. The amount of leakage observed in test 4 was less than the allowable 3.15×10^{-5} g.

During test 6, the 2R vessel was rotated continuously during the heating and soaking cycle. Continuous rotation did not appear to promote greater DUO transmission than periodic rotation (compare test 4). The gas leak rate for the No. 2 1/2 can increased during heating. The amount of leakage inside the No. 3 can was less than the allowable leakage. No leakage was detected inside the 2R vessel.

In tests 7 and 8, the 2R vessel was heated to higher temperatures (see Table 3), and the number of barriers for powder transport was reduced. In

addition, the 2R vessel was rotated and vibrated during heating. Under these conditions, more leakage of DUO powder was observed. The rotation of the 2R vessel during heating allowed the powder to come in contact with the leak sites. In test 8, the powder transmitted to the 2R vessel was below the allowable. In test 7, even though the No. 3 can was badly deformed (Figure 17), no gas leaks developed and no leakage of DUO into the 2R vessel was observed. The leakage of powder from the No. 2 1/2 cans into the No. 3 can was significant in tests 7 and 8 (see Table 3). This leakage was due to the higher gas leak rates and the abundant powder inside the No. 2 1/2 can. Without the tape seal, the slip lid cans released substantial amounts of powder inside the No. 2 1/2 can (Figure 18).

To determine the effect of rotation and vibration without heating, a test (test 9, Table 3) was made with a packaging configuration the same as that shown in Figure 7. The test results show that rotation and vibration provide enough driving force to cause leakage when the inner containers have leak sites.

In test 10, the leakage from a 2R vessel with a leaky threaded fitting was determined. (Table 3, test 10) The gas leak rate after heating was considerably higher than before. Pressure buildup inside the metal can during heating caused the metal can to deform and thus enlarge the leak paths. Heating also caused the luting compound (LA-CO SLIC-TITE®) on the threads of the 2R vessel to dry out and lose some of its sealing capability, and thus larger openings were created under pressure. Even with the high gas leak rates, the amount of DUO powder leaking through the unsealed threads into the sealed container was below the allowable amount.

COMPARISON OF POWDER LEAKAGE FROM ORIFICES, CAPILLARIES AND METAL CANS

The transmission of powders through small openings is a complex phenomena. The flow of powders is dependent on many variables. The physical properties of the particles influence the flow characteristics such as particle diameter, voidages, moisture content and angular properties. These physical properties and their influence on powder flow are discussed by Sutter, et al.

® LA-CO SLIC-TITE is a registered trademark of Lake Chemical Co.

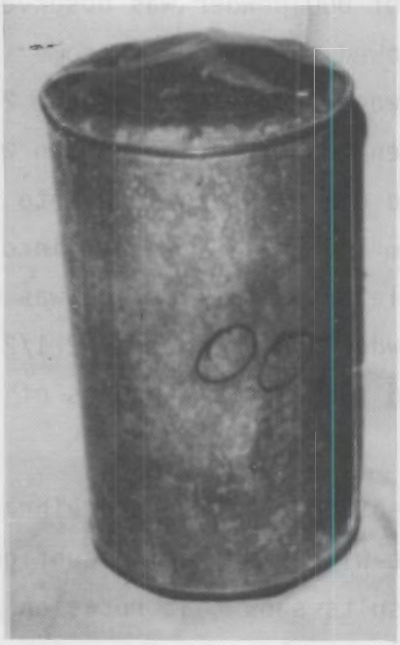

FIGURE 17. Deformation of No. 3 Can Due to Pressure During the Heat Test (Test 7)

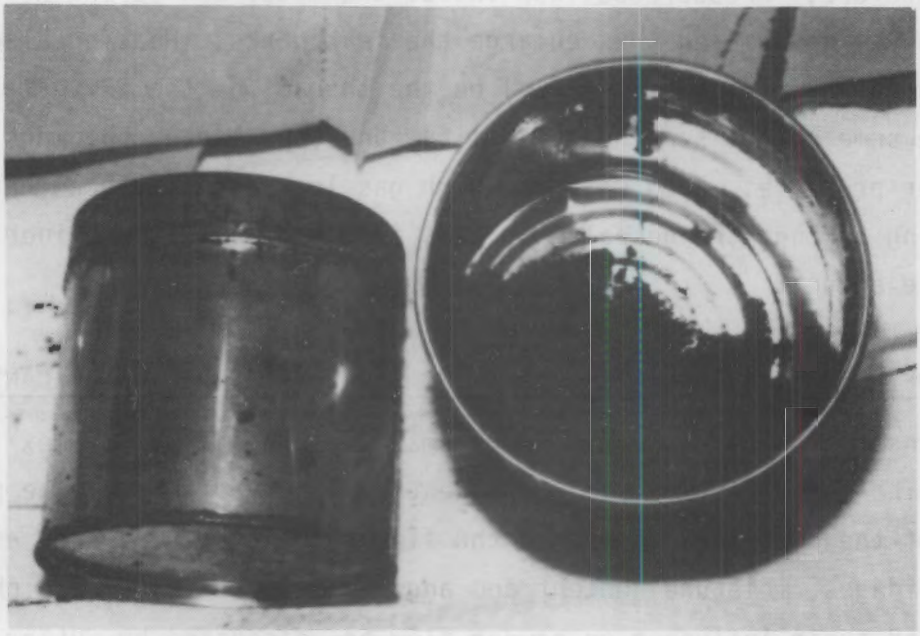

FIGURE 18. DUO Powder Released Inside No. 2-1/2 Can From Slip-Lid Can After Impact and Heat Test

(1980). In addition to the physical properties of the powder, the experimental test variables strongly influence powder leakage such as:

- mechanism of particle transport to leak sites
- size and number of leak sites
- complexity of leak path (path length, path circuit, barriers)
- driving forces applied to move particles through openings.

The mechanisms used in this study to transport particles to the leak sites were gravity (rotation and vibration) and pressure. Pressure could have influenced powder transport either by promoting or hindering the flow depending on the direction of gas flow. During heating a temperature gradient exists, i.e., the inner packaging components have lower temperatures than the outer packaging components. Consequently, the pressure is greater from the outside in, and gas flow would also be from the outside in. During cooling the pressure differential is reversed, and the gas flow would be from the inside out. In this study it appeared that the main influence of pressure on powder transmission was to enlarge the existing leak sites through deformation of the cans.

The gas leak rate is an indication of the size of the leak path. However, a situation might exist where a high gas leak rate might have a lower potential particle leakage than a lower gas leak rate due to the fact that there are more leak sites with smaller openings. In this study, the higher gas leak rates produced higher particle leakage regardless of the number of leak sites. This could be explained by the fact that the particle size of the DUO was much smaller than any of the leak sites.

The use of rotation and vibration also promoted particle leakage. The data shown in Table 3 indicate higher DUO leakage when vibration and rotation were both used simultaneously.

In the test runs (see tests 4, 5, 6, Table 3) where a greater number of barriers was used in the packaging configurations, the leakage of DUO was less. In addition, no vibration was used in these test runs, a factor which could also influence the amount of DUO powder transmitted.

A comparison was attempted between the particulate release observed in this study and the release reported in the studies by Sutter, et al. (1980) and

Curren and Bond (1980), Table 4. The data in Table 4 were obtained from equations presented by Sutter, et al. (1980) and from curves by Curren and Bond (1980).

Direct comparisons among the studies were not possible because of the difference in test parameters; only descriptive comparisons could be made. The comparisons that were made were based on leak site diameter. This was a tentative comparison since the leak paths through the crimp sealed lids were probably not circular and in most cases there was more than one leak site. In the tests by Curren and Bond and Sutter, et al., only one leak site of known circular cross section was present.

Another variable that influenced the leakage of powder in the tests reported here was the increased size or number of leak sites during the heat test. The increase in size or number of leak sites was due to the pressure increase during heating which caused the cans to deform and increased the size or number of the leak sites. The range of diameters seen in Table 4 is due to the increase of the leak site area during the test run. The leak site diameters for the data were estimated by comparing the measured air flow rates through capillaries of known diameter (Owzarski et al. 1979) with the measured gas flow rate through the leak sites in the No. 2 1/2 size cans. A correction factor was applied to account for the number of leak sites. Only the leakage of DUO powder from the No. 2 1/2 cans is presented in Table 4.

There were not enough data from the tests performed to provide the basis for detailed statistical analysis such as was done in the Sutter, et al. (1980) study. However, the tabulated values indicated greater releases were observed by Sutter, et al. (1980) for corresponding leak site diameters. The leakage reported by Curren and Bond (1980) was noticeably greater for corresponding leak site diameter. The significantly higher leakage may be explained in part by the fact that, during Curren's and Bond's tests, vigorous vibration was used. The vibration may have prevented the orifices from plugging. This coupled with maximum gas flow may have produced the higher transmission of powder.

TABLE 4. Comparison of DUO Powder Leakage from Various Experimental Studies

Type of Leak Path	Diameter, μm	Pressure, psig	Gas Flow, atm cc/min	Run Time, min	DUO, μg
Capillary[a]	120	30	96	60	18[b]
Capillary[a]	182	15	29-67	60	89[c]
Capillary[a]	276	30	520-1650	60	649[b]
Orifice[a]	100	15	160-180	60	287[c]
Orifice[a]	200	30	860-1000	60	744[c]
Orifice[d]	100	15	~96	60	7300
Orifice[d]	200	15	~378	60	30400
Tortuous path[e]	200-300	--[f]	--[f]	360[g]	1000
Tortuous path[e]	190-350	--	--	270[g]	100
Tortuous path[e]	140-220	--	--	120[g]	4
Tortuous path[e]	110-135	--	--	180[g]	6
Tortuous path[e]	100-180	--	--	210[g]	1

[a] From Sutter, et al. (1980).

[b] Leak path above the static powder level.

[c] Leak path under the static powder level.

[d] From Curren and Bond (1980).

[e] Distance through crimp seal on lid of No. 2 1/2 food pack can.

[f] Not measured during tests.

[g] Time of test, i.e., period from start to finish of vibration and rotation.

In general, it may be speculated from the results shown in Table 4 that the powder leakage would be greater for the tests made with capillaries and orfices than for metal cans that have more complicated leak path geometries.

The data listed in Table 4 represent upper limits of powder release. The data generated in the studies performed are pessimistic in that the test conditions were much more severe than what would be anticipated in a real accident.

The conclusion can be reached that under accident conditions where gross gas leaks occur (~96 atm cc/min), particle leakage will be less than the allowable amount for the packaging configurations tested in this study. Using proper packaging procedures and quality controls (see Appendix B), particulate material may be packaged in the 6M container and the material will be contained within the regulatory standards in the 6M container even under accident conditions.

REFERENCES

American National Standard Institute (ANSI). 1977. American National Standard for Leakage Tests on Packages for Shipment of Radioactive Materials. ANSI N 14.5-1977. American National Standards Institute, New York, New York.

Andersen, J. A. 1983. "Correlation Between Measured Gas Leaks and Possible Loss of Contents from Radioactive Materials Packagings." In Proceedings of the Seventh International Symposium on Packagings and Transportation of Radioactive Materials, Vol. 1, pp. 646-654, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Ayres, J. A. 1970. Decontamination of Nuclear Reactors and Equipment. Ronald Press, New York.

Blankenship, R. W. 1980. Hypothetical Accident Conditions Free Drop and Thermal Tests Specification 6M. LA-8366-MS, Los Alamos National Laboratory, Los Alamos, New Mexico.

Chalfant, G. G. 1984. Safety Analysis Report - Packages USA/9965/BLF USA/9968/BLF. DPSPU 83-124-1, E. I. Du Pont De Numors and Co., Inc., Aiken, South Carolina.

Curren, W. D., and Bond, R. D. 1980. "Leakage of Radioactive Powders from Containers." In Proceedings of the Sixth International Symposium on Packaging and Transportation of Radioactive Materials, Vol. 1, pp. 463-471. Bundesanstalt für Materialprüfung, West Berlin, Germany.

Emerson, E. L., and J. D. McClure. 1983. Radioactive Materials (RAM) Transportation Accident/Incident Data Analysis Program. SAND82-2156, TIC-0385, Sandia National Laboratories, Albuquerque, New Mexico.

Energy, 10 C.F.R. Part 71.51 (a)(2) (1984).

Energy, 10 C.F.R. Part 71.52 (1984).

Energy, 10 C.F.R. Part 71.73 (1984).

Lake, W. H. 1983. "Containment System Evaluation." In Proceedings of The Seventh International Symposium on Packaging and Transportation of Radioactive Materials, Vol. 1, pp. 633-637. Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Loewen, T., et al. 1980. "Candu Irridated Fuel Transportation System - Dynamic Analysis." In Proceedings of the Sixth International Symposium on Packaging and Transportation of Radioactive Materials, Vol. 11, pp. 1283-1291. Bundesanatalt für Material Prüfung, West Berlin, Germany.

Mendel, J. E. 1960. Laboratory Investigation of Decontaminating Solutions for Primary Loop Decontamination in the New Production Reactor. HW-67364, General Electric Company, Hanford Atomic Products Operations, Richland, Washington.

Neibaur, G. E., and N. D. Stice. 1961. Dissolution of Uranium Oxides Formed During a Fuel Element Failure. HW-69594, General Electric Company, Hanford Atomic Products Operations, Richland, Washington.

Office of Nuclear Material Safety and Safeguards. 1978. Plutonium Air Transportable Package Model PAT-1. Safety Analysis Report, NUREG-0361, National Technical Information Center, Springfield, Virginia.

Owzarski, P. C., et al. 1979. Measured and Predicted Gas Flow Rates through Rough Capillaries. NUREG/CR-0734, PNL-2623, Pacific Northwest Laboratory, Richland, Washington.

Robbins, J. C. 1978. "Field Techniques for the Measurement of Uranium in Natural Waters." CIM Bulletin, pp. 2-8.

Sutter, S. L., et al. 1980. Depleted Uranium Dioxide Powder Flow Through Very Small Openings. NUREG/CR-1099, PNL-3177, Pacific Northwest Laboratory, Richland, Washington.

Taylor, J. M. 1985. Gas Leak Characteristics of Inner Packaging Components Used in the DOT-Spec 6M Container. PNL-5591, Pacific Northwest Laboratory, Richland, Washington.

Transportation, 49 C.F.R. 173.403 (b) (1984).

Transportation, 49 C.F.R. 173.417 (b) (2) (1984).

U.S. Nuclear Regulatory Commission (NRC). 1975. U.S. Nuclear Regulatory Guide 7.4. NRC, Washington D.C.

Yesso, J. D., et al. 1980. Study of Plutonium Oxide Powder Emissions from Simulated Shipping Container Leaks. NUREG/CR-1302, PNL-3278, Pacific Northwest Laboratory, Richland, Washington.

APPENDIX A

CORRESPONDENCE BETWEEN NRC AND DOT

UNITED STATES
NUCLEAR REGULATORY COMMISSION
WASHINGTON, D. C. 20555

Mr. Richard R. Rawl
U. S. Department of Transportation
Office of Hazardous Materials Operations
Washington, D.C. 20590

Dear Mr. Rawl:

This is in regard to your letter of June 6, 1979, concerning the DOT Specification 6M package and its ability to meet the 1973 IAEA criteria.

The 6M packaging consists of an inner Spec. 2R containment vessel and a steel drum overpack containing a media to provide impact and thermal protection for the inner vessel. We believe the current 6M specification is sufficient for the overpack but not for the inner vessel.

We believe package specifications should contain sufficient information to assure that hardware fabricated in accordance with the specification will meet all pertinent requirements. While this may be possible with the current specifications, the information and requirements are not sufficient to assure the 2R vessel would meet the newly quantified leak-rates in IAEA regulations. Secondly, the 6M Specification requires that large quantities of radioactive material in normal form must be packaged in one or more sealed and leak tight metal cans or polyethylene bottles within the 2R vessel. However, 10CFR§71.42 has been amended to require additional containment for plutonium. We believe the 6M Specification should be consistent with the requirements of 10 CFR Part 71. Finally, the maximum heat load permitted in various configurations of the package should be tabulated.

The basic need is for a Specification that will assure the 2R vessel and the secondary inner container will meet the proposed DOT/NRC requirements. This need also extends to other DOT Specification packages which are authorized for greater than Type A quantities in normal form.

We believe that consideration should be given to restructuring DOT specification packages to limit the contents in normal form to no more than a Type A quantity. The administrative reasons that previously existed for authorizing Type B packaging through engineering specifications in Federal Rules have been eliminated by improvements made to the NRC licensing mechanism. Package designs are now authorized by Certificate of Compliance rather than by specific amendment of individual possession or facility licenses; also

other users may now simply register under 10CFR§71.12 or 49CFR173.393a without submitting an application or obtaining a specific approval. Under the present system, there appears to be essentially no need to continue authorizing Type B packages through DOT Specifications.

Sincerely,

Charles E. MacDonald, Chief
Transportation Certification Branch
Division of Fuel Cycle and
Material Safety, NMSS

APPENDIX B

PACKAGING PROCEDURE FOR DISPERSIBLE PLUTONIUM MATERIAL

APPENDIX B

PACKAGING PROCEDURE FOR DISPERSIBLE PLUTONIUM MATERIAL

The purpose of this appendix is to provide procedures for packaging and loading dispersible[a] plutonium materials into DOT specification 6M shipping containers. These procedures are based on the tests performed in this study and a previous study (Taylor 1985). The packaging configuration and procedure outlined herein for the 30-gallon size 6M container will provide a package system that will contain plutonium powders even under hypothetical accident conditions. Any other packaging configuration now being used or planning to be used to package dispersible radioactive material (RAM) will have to be tested to determine if that configuration adequately protects the inner packaging components and restricts the release of plutonium powders to less than A_2/wk quantity under accident conditions.

In order for the procedures outlined in this appendix to be carried out, a quality assurance program is needed. Also, it will be necessary to write procedures where a test method is suggested (e.g., bubble test). From these procedures, instruction or checklists will be required.

The plutonium material being packaged for placement in the 6M container must be characterized as to heat output, thermal stability, and chemical stability. Only stable materials shall be packaged for shipment in the 6M container. This characterization will require documentation to verify the condition of the plutonium material being packaged. The information provided in Packaging of Plutonium for Storage or Shipment (Van Tuyl 1981) should be followed when plutonium materials need to be characterized. Only those materials meeting the conditions listed in 49 CFR 173.417 (b)(2) shall be loaded in the 6M container.

(a)Dispersible plutonium refers to powders with typical mass median diameters of 3.5 μm.

The discussion that follows covers general specifications, general packaging requirements for plutonium materials, packaging of plutonium materials in food pack cans, inspection and loading 2R vessels, and checkout of the 6M drum.

GENERAL SPECIFICATIONS

The following generic specifications are intended to apply to all plutonium packaging for shipment. More detailed requirements are given for dispersible materials in the following section. This set of specifications is intended to call attention to the principal factors that need to be controlled.

- The inner packaging shall not degrade and shall remain intact when subjected to the maximum temperature expected during normal shipment. Determination of any maximum temperature shall include: 1) heat from radioactive decay, chemical reactions within the package, and external heat sources, 2) evaluation of insulation or barriers to heat flow, and 3) evaluation of package configuration inside the insulated drum for the maximum credible time.
- Pressure within the food pack cans and 2R vessel shall not exceed the pressure that these components are subjected to during leak testing (e.g., bubble testing ~15 psig). Considerations relating to the maximum pressure shall include: 1) gas formation due to thermal decomposition of the contents, 2) thermal expansion of gases, 3) radiolytic gas generation, and 4) gas formation by chemical reactions within the package.
- The outer surface of the sealed metal containment system shall be free of radioactive contamination.
- Each containment system (metal cans) shall be clearly and uniquely labeled. The label shall be legible after being subjected to the maximum normally expected temperature and radiation dose for the maximum credible time.
- Each package or shipment shall include a complete listing of the contents. The listing shall describe the material within each containment system. If needed, special handling instructions for unpackaging shall be included with the packing list.

GENERAL PACKAGING REQUIREMENTS FOR PLUTONIUM MATERIALS

Solid plutonium compounds that are dispersible (such as powders) and stable in air at the credible shipping temperature[a] shall be placed in a metal container (such as a taped slip lid can), which is then placed in a sealed polyethylene bag. This bag is placed inside a mechanically sealed food pack can. Finally, the food pack can is placed inside another mechanically sealed food pack can.

The compound must have an LOI[b] of <1% when heated in an inert atmosphere at 450°C for 2 hours. Compounds that react with air (such as carbides or hydrides) must be packaged in an inert atmosphere. Prior to shipment, the powders must be stored in dry atmopsheres (dew point -70°F) to prevent adsorption of H_2O by the powder.

PACKAGING OF PLUTONIUM MATERIALS IN FOOD PACK CANS

This procedure pertains to the operation of placing the plutonium material in food pack cans preparatory to placing the cans into the 2R vessel. At this stage of the operation, the plutonium material has been placed in a metal container such as a taped, slip lid can inside a glovebox and the metal container has been bagged out (polyethlene bag) of the glovebox ready for placement in the food pack can. No individual metal container should contain more than 2.25 kg of dispersible plutonium material.

The food pack cans used to contain the plutonium material must conform to the Federal Specification, PPP-C-96D.[c] The cans are classified as Type I, Class 3, Packers Cans. Only ribbed reinforced lids (concentric rings) shall be used. The flat profile (no rings) lids will permanently deform when pressurized to 15 psig during bubble testing.

(a) The temperature expected to be achieved as a result of exposure to the hypothetical accident conditions (~300°F).

(b) LOI means loss of ignition, which is usually a measure of volatile components. Some chemical reactions could result in weight gain on ignition, and if this is possible, a method other than weight change must be used to determine volatile constituents.

(c) Suggested vendor for food pack cans is Freund Can Company, Chicago, IL.

1. Inspect can body and make certain it is not dented or damaged.

2. Inspect the flange on the can body. If the flange has been severely bent or creased, do not use it. Small creases can usually be straightened by using long-nose pliers. Run your finger around the flange to make certain it is smooth and no discontinuities are present. Generally, there is a small ridge in the flange where the side seam intersects it. If the ridge is quite abrupt, smooth it off carefully with a fine-toothed jeweler's file or discard the can.

3. Inspect the lid and make sure the rim where the rubber compound has been applied is smooth and uniform. There should be no exposure of metal showing through the rubber compound. If there are scratches or shiny spots (metal showing through), discard the lid. Also, make sure the curl or roll over at the rim of the lid is uniform and not dented.

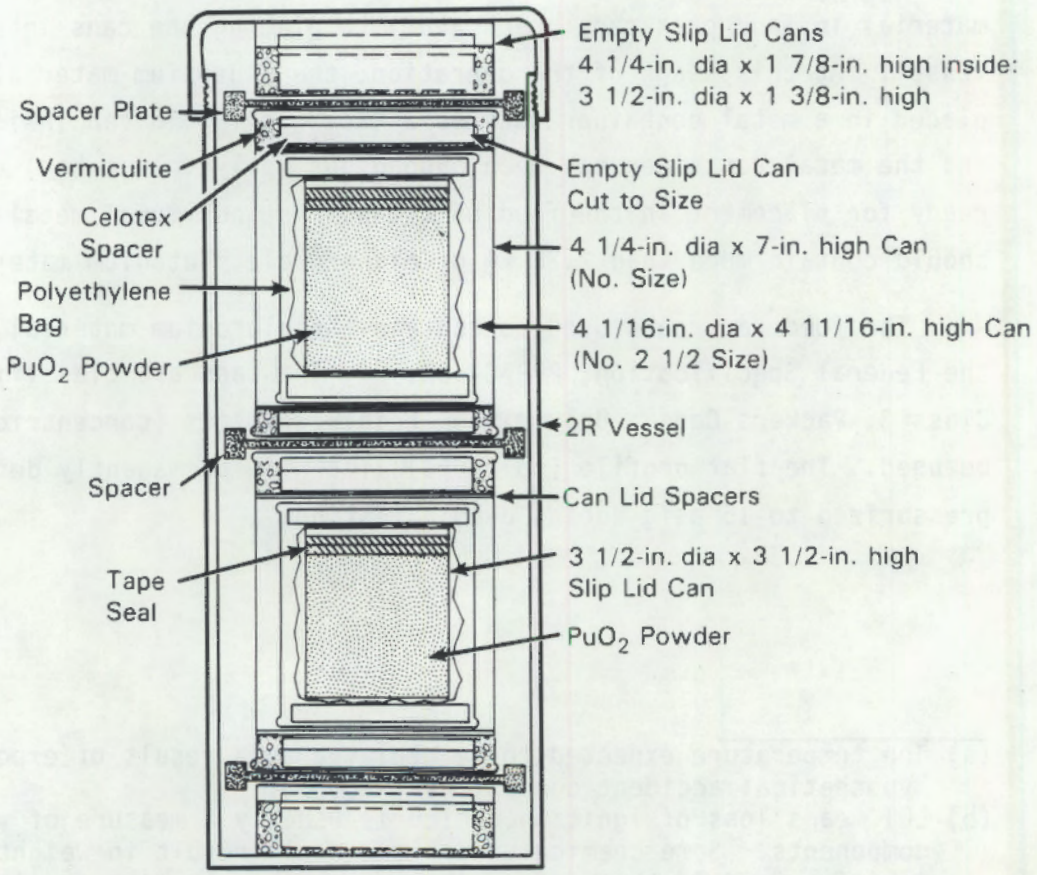

FIGURE B.1. Packaging Arrangement for Containment of Plutonium Oxide Powder Inside 2R Vessel

4. Place the bagged container (polyethylene bag) in the food pack can (No. 2 1/2 size) as shown in Figure B.1. The celotex spacers shown are about 1/2-in. thick. Do not force or stuff the bag into the food pack cans.

5. Place the lid on the can and check to see that the lid is seated properly. The curl on the rim of the lid should be below the flange on the can. Do not rotate the lid on the flange or the rubber compound may be scratched or damaged.

6. Center the cans on a properly adjusted can sealer [see Appendix B of Taylor's (1985) report]. Rotate the base plate to make certain the can is centered.

7. Raise the can or lower the chuck depending on sealer, making sure the lid to be sealed is properly engaged in the chuck. Position the can so that the part of the lid directly over the side seam on the can does not contact the seaming roller first.

8. Before cranking the handle of the sealer (motorized can sealers return to correct starting position automatically), make certain that the seaming rollers are in the proper starting position [see Appendix B of Taylor's (1985) report for discussion on properly setting up sealer].

9. Crank the handle in a clockwise direction. Try to maintain a uniform rotation through the sealing operation. If a motorized can sealer is being used, only use one complete cycle to seal the cans. Repeating the cycle will not provide better seals but may degrade the first sealing operation.

10. Lower the turntable or raise the chuck and remove the can. If the can is stuck on the chuck, the second seaming roller is set too tight.

11. Visually examine the can for obvious defects as illustrated in Appendix B of Taylor's (1985) report. If defects are present, the can sealer is out of adjustment and must be fixed.

12. If the seal looks good, bubble test the can.[a] If no streaming bubbles are observed, the can is hermetically sealed.

13. Select a No. 3 can that has been inspected as per steps 1 through 3, and place the No. 2 1/2 can into the No. 3 can as shown in Figure B.1. The empty cans shown on the top and bottom of the No. 2 1/2 cans are made by cutting up a 3 1/2-in.-dia x 3-1/2-in.-high slip lid can and are 3/4 in. high. To provide the proper impact absorbing properties, notch the side wall of the can as shown in Figure B.2. After notching the sidewall, place the slip lid on the can body. Place three lids, cut from a No. 3 can, in between the empty cans and the No. 2 1/2 can as shown in Figure B.3. Pour vermiculite around the cut off slip lid cans to center them inside the No. 3 can.

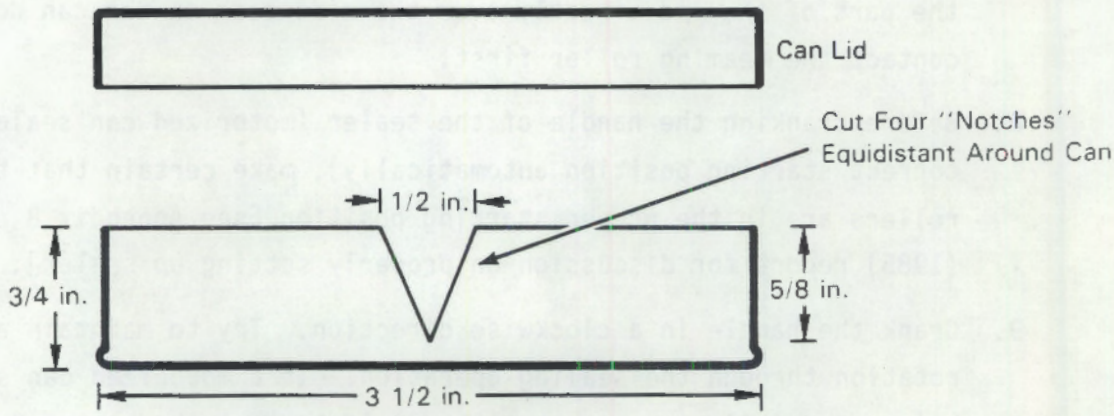

FIGURE B.2. Cut-Off Slip Lid Can for Use as Impact Absorber

(a)Where large numbers of cans are being prepared for shipment, a sampling system could be set up where only one in five or ten cans is actually bubble tested or a dummy can is sealed up and bubble tested.

FIGURE B.3. Stacking Arrangement of No. 2 1/2 Cans Inside No. 3 Cans

PROCEDURE FOR INSPECTING AND LOADING 2R VESSEL

1. Visually inspect the threads on the 2R pipe body and pipe cap or plug. If the threads are damaged such that the damage is continuous from the bottom of the thread to the top, reject the part. Repair minor damage by using a thread dressing tool.

2. Stack the cans inside the 2R vessel as shown in Figure B.1. The impact absorbing cans shown on the top and bottom of the No. 3 cans are made by cutting a 4 1/4-in.-dia x 5 9/16-in.-high slip lid can. The inner can shown can be made up from a 3 1/2-in.-dia x 3 1/2-in.-high slip lid can. The inner can is centered inside the outer can with vermiculite. Spacer plates are required between the No. 3 cans and the impact absorbing cans so that the impact load will be transmitted to the sidewalls of the #3 cans. This will prevent the No. 3 cans from deforming during impact. The details for the spacer plates are shown in Figure B.4. To prevent the can lids from becoming concave during impact, fill in the space between the can lid and the spacer plate. This can be done by forming a plug of a low-melting alloy such as bismuth-cadmium (60% Bi, 40% Cd). Melt the alloy and pour it onto the lid of a sealed

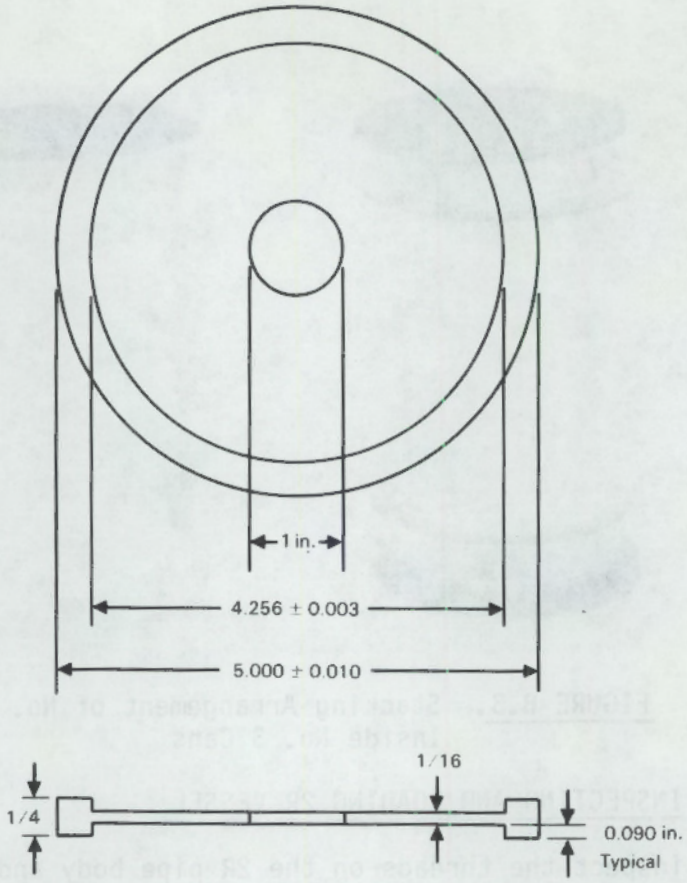

FIGURE B.4. Aluminum Spacer Plate

No. 3 can. Level the plug by drawing a straight edge across the top of the can. After the plug has solidified, remove it and file off enough material around the circumference so it fits easily onto the lid of the can. The plug will conform to the shape of the can lid as shown in Figure B.5. The plugs are easy to fabricate and are reusable.

3. Coat the threads on the pipe body and the cap or plug with a liberal amount of LA-CO SLIC-TITE® paste(a) or G.E. Silicone Hi-Temp Gasket® Material, or equivalent material approved by DOE, and screw the plug or cap into or onto the pipe body by hand until tight.

(a) LA-CO SLIC-TITE paste, a registered trademark, can be obtained from the Lake Chemical Co., Chicago, IL.

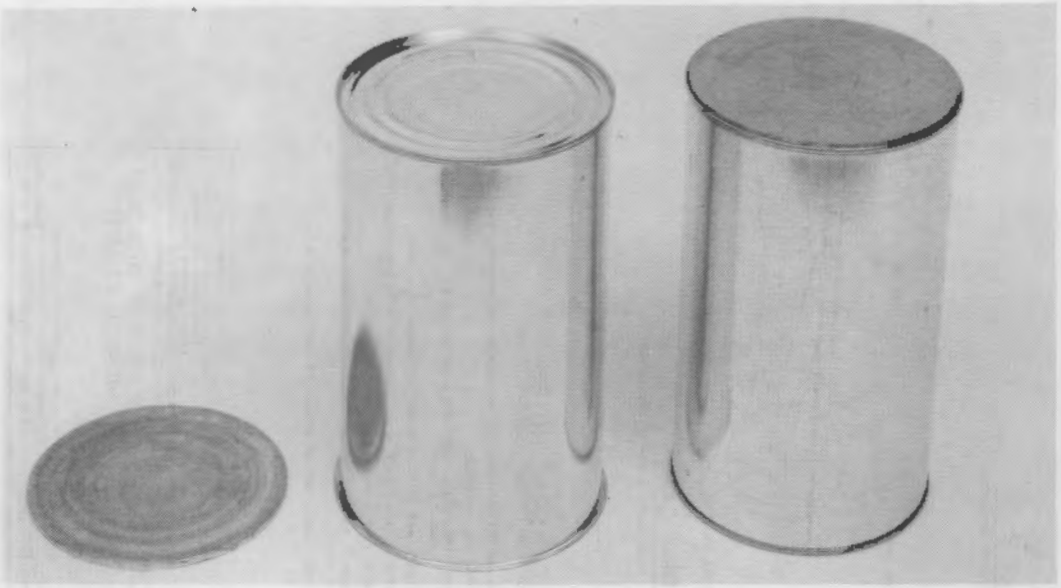

FIGURE B.5. Metal Plugs Used to Protect Can Lids During Impact

4. Place the 2R vessel in a vice or other device and secure it so it will not slip.
5. Using a torque wrench with a pipe cap clamp (see Figure B.6 for details) or plug fixture, torque the cap or plugs to 100 ft-lb.
6. Wipe off the excess pipe compound.
7. Bubble test[a] (see ANSI Standard N14.5, Appendix A, A 3.6 for bubble test procedure) every tenth assembly. If no streaming bubbles are observed, the seal is adequate.

PROCEDURE FOR CHECKOUT, INSPECTION AND LOADING OF 6M DRUM

1. Check identification plate on drum and make certain marking is correct.
2. Check drum to verify it is in good condition. No dents or imperfections which penetrate the drum or prevent attaching lid and locking ring.
3. Check drum cover gasket for cracks, deterioration and proper fit.

(a) If G.E. Silicone Hi-Temp Gasket Material, which is a registered trademark of the G.E. Company, is used on the threads, allow 12 to 14 hours before bubble testing.

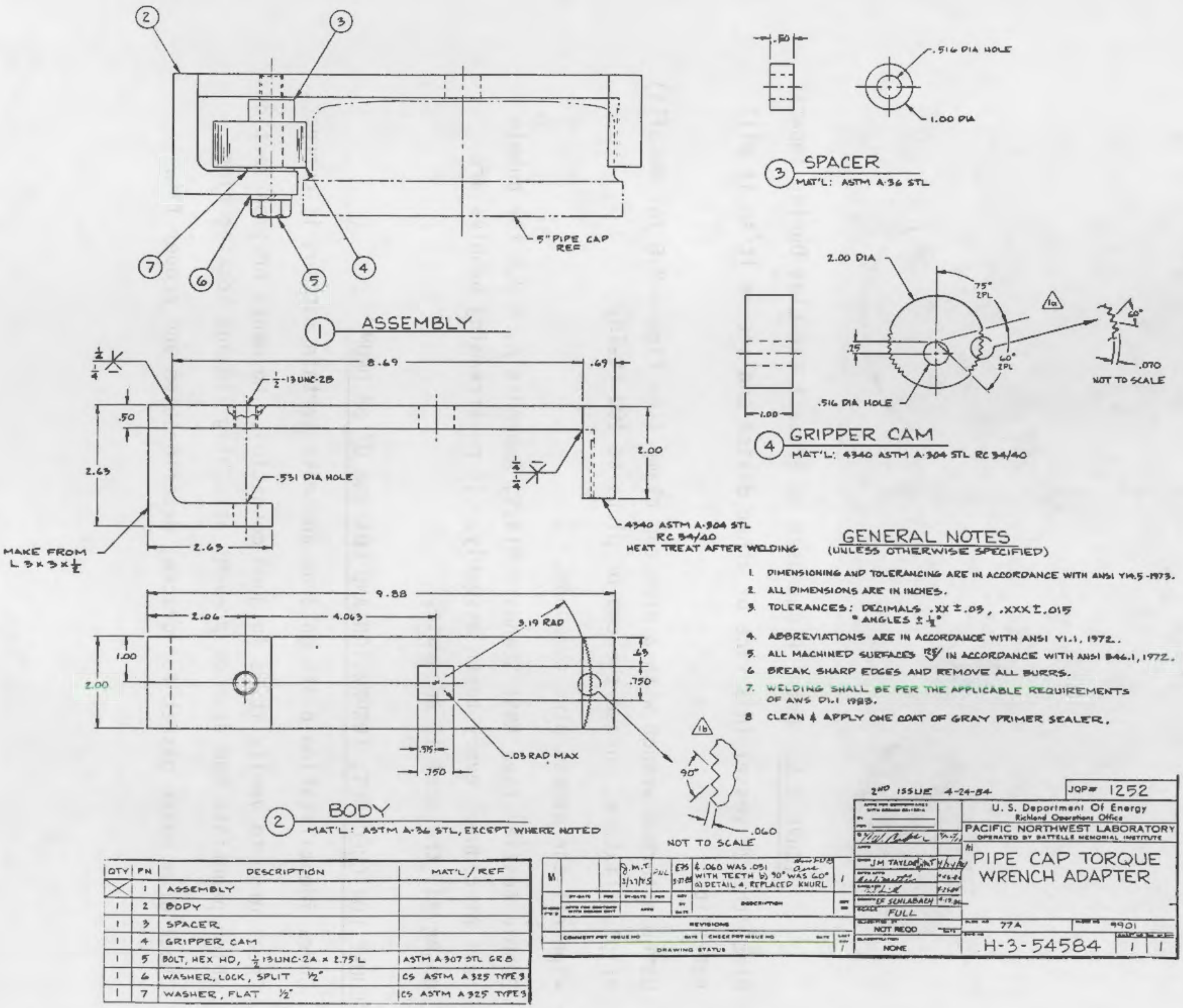

FIGURE B.6. Torque Wrench Adapter for Pipe Cap

4. Check drum cover for damage and proper fit to the drum.

5. Check locking ring for proper fit, check for cracked welds on bolt lugs and check to be sure that bolt properly screws into locking ring. Check bolt to see if a hole is drilled in the end of the bolt to accommodate a tamper-proof seal.

6. Check drum to determine if four vent holes (0.5-in. diameter) are near the top of drum equally spaced around drum and about 1 3/4-in. down from top of drum. Also check that plastic plugs are installed in vent holes.

7. Check cane fiberboard rings for damage. If rings or discs are split or distored, replace them.

8. Verify 2R container is protected by minimum thickness[a] of cane fiberboard disc.

9. Make certain bearing plates are in place on 55 and 110 gallon 6M containers [see 49 CFR 178.104 (3)(e)].

10. Place 2R vessel in 6M drum and replace cane fiberboard discs. Make certain discs fill drum.

11. Install drum cover and locking ring. Make certain the bolt is in the down position. Tighten locking ring. As the bolt is being tightened, tap the ring with a hammer. Torque the bolt to 40 ft-lb.

12. Place lock nut on bolt outside of lock ring lugs. Tighten nut to 10 ft-lb.

13. Install tamper proof seal in bolt hole.

14. Perform radiation survey, apply appropriate markings and labels and prepare shipping papers.

(a) Minimum thickness of 3.75 in. on sides and 3.75 in. on end (except for 15-gallon or less which is 1.88 in. on end).

DISTRIBUTION

No. of
Copies

OFFSITE

30 DOE Technical Information Center

F. E. Adcock
Rockwell International, Rocky Flats Plant
P.O. Box 464
Golden, CO 80402-0464

G. Allen
Division 6323
Sandia National Laboratory
P.O. Box 5800
Albuquerque, NM 87185

F. M. Almeter
U.S. Department of Energy
DP-4.2, GTN
Washington, DC 20545

W. Bateman
International Energy Associates Ltd.
2500 Virginia Avenue, NW
Washington, DC 20057

J. M. Cece
U.S. Department of Energy
EH-122, GTN
Washington, DC 20545

G. G. Chalfant
duPont, Savannah River Plant
Building 723-9A
Aiken, SC 29808

D. A. Edling
Mound - MRC
P.O. Box 31
Miamisburg, OH 45342

K. E. Elliott
U.S. Department of Energy, Albuquerque Operations Office
Environmental, Safety and Health Division
P.O. Box 5400
Albuquerque, NM 87115

F. D. Falci
U.S. Department of Energy
DP-123
Washington, DC 20545

L. Gage
Quality Engineering Division
Albuquerque Operations Office
P.O. Box 5400
Albuquerque, NM 87115

K. Golliher
U.S. Department of Energy, Albuquerque Operations Office
Environmental, Safety and Health Division
P.O. Box 5400
Albuquerque, NM 87115

R. D. Hahn
DP-22.2 Military Application
U.S. Department of Energy
Washington, DC 20545

C. Henn
Quality Engineering Division
Albuquerque Operations Office
P.O. Box 5400
Albuquerque, NM 87115

N. King, Jr.
P-229
Los Alamos National Laboratory
P.O. Box 1663
Los Alamos, NM 87545

D. M. Krieg
Rockwell International, Rocky Flats Plant
P.O. Box 464
Golden, CO 80402-0464

R. E. Luna
Division 6321
Sandia National Laboratory
P.O. Box 5800
Albuquerque, NM 87185

G. W. May
Quality and Materials Assurance
Division
Department of Energy
Savannah River Operations Office
P.O. Box A
Aiken, SC 29802

C. J. Mauch
U.S. Department of Energy
DP-122.2
Washington, DC 20545

J. D. McClure
Division 6321
Sandia National Laboratory
P.O. Box 5800
Albuquerque, NM 87185

E. M. Roybal
U.S. Department of Energy,
Albuquerque Operations Office
Environmental, Safety and
Health Division
P.O. Box 5400
Albuquerque, NM 87115

P. A. Saxman
U.S. Department of Energy,
Albuquerque Operations Office
Environmental, Safety and Health
Division
P.O. Box 5400
Albuquerque, NM 87115

D. R. Smith
F-691
Los Alamos National Laboratory
P.O. Box 1663
Los Alamos, NM 87545

ONSITE

3 DOE Richland Operations Office

E. M. Bowers
R. M. Hiegel
J. J. Sutey

2 Rockwell Hanford

D. L. McCall
R. W. Szempruch

19 Pacific Northwest Laboratory

J. T. Denovan
S. C. Hawley
J. M. Taylor (10)
Publishing Coordination (2)
Technical Information (5)