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Internal Corrosion Analysis of Model 9975 Packaging Containing Pu or Pu02 During Shipping and Storage

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INTERNAL CORROSION ANALYSIS OF MODEL 9975 PACKAGING CONTAINING Pu OR PuO_2 DURING SHIPPING AND STORAGE (U)

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
ASME	American Society of Mechanical Engineers
PCV	Primary Container Vessel
RFETS	Rocky Flats Environmental Technology Site
SARP	Safety Analysis Report for Packaging
SRS	Savannah River Site
SRTC	Savannah River Technology Center
SCV	Secondary Container Vessel
SS	Stainless Steel

1.0 SUMMARY

The Materials Consultation Group of SRTC has completed an internal corrosion analysis of the Model 9975 packaging assembly containing either Pu or PuO_2 for storage in K Reactor under ambient conditions for a period of 12 years. The 12-year storage period includes two years for shipping and up to ten years for storage. Based on review of existing literature and referenced conditions, corrosion of the 304L stainless steel (SS) Primary Containment Vessel (PCV) and the carbon steel food cans containing Pu or PuO_2 is not significant to cause failure during the 12 year time period due to limited water content. The potential for embrittlement of the Primary Containment Vessel and the carbon steel can due to iron diffusion into plutonium (or plutonium diffusion into the steel) and gallium diffusion into the steel have been assessed. This phenomenon should not be significant during the storage period. The integrity of the stainless steel Primary Containment Vessel will not be impacted during shipping or storage. The Materials Consultation Group concludes that there are sufficient data to establish the technical basis for safe storage of Pu and PuO2 at the referenced conditions in the Model 9975 package in the 105-K building for up to 10 years following the 2-year shipping period.

2.0 BACKGROUND AND ASSUMPTIONS

Pu-bearing materials (metal and oxides) currently stored at RFETS (Rocky Flats Environmental Technology Site) are to be shipped to SRS in Model 9975 packaging assemblies for interim storage prior to future disposition. The Model 9975 shipping package design[1-2] includes an inner carbon steel food can (A), 0.025 cm thick, that will contain the RFETS material, Figures 1 and 2. Can A will then be placed into two, 0.005-cm thick nylon bags (double bagging). The bagged can A will be placed inside a larger carbon steel food can (can B), also 0.025 cm thick. The carbon steel food cans are specified per MIL-C-10464 (1990). Two of the can B's will be stacked on one another and placed into the 304L stainless steel Primary Containment Vessel (PCV). The can B's will be supported on the bottom by an aluminum honeycomb structure to stabilize the cans within the primary container. The PCV is then placed inside a Secondary Container Vessel (SCV) that is also 304L SS. Aluminum honeycomb is also used to support the PCV within the SCV. Any interior corrosion occurring in this package will initiate from the inside of the initial food can A. No credit is taken for corrosion protection from any organic or tin coating on the food cans or the nylon bags. A discussion of external corrosion concerns can be found in Reference 3.

Multiple units of the Model 9975 packaging assembly will contain a maximum of either 4.5 kg of Pu or 5.0 kg of PuO_2.[4] This amount will be split in half so that each food can contains a maximum of 2.25 kg Pu or 2.5 kg of PuO_2. No water is specified in the package containing Pu metal but the specifications[4] allow a maximum of 1.5 % water (75 g total for two food cans) in the PuO_2 package. The corrosion analysis assumes that Pu metal compositions will be within the range displayed in Table 1[5] and will contain a minimum of 98% Pu with impurity levels less than or equal to 2%. The impurities are assumed to be homogeneous throughout the metal, except for the lighter metals that may be concentrated near the top of the metal due to their density. Table 2, compiled from Reference 5, shows that the composition of some of the RFETS Pu metal will be outside the acceptable composition limit. The PuO_2 material from RFETS is assumed to be calcined, tested to an acceptable loss on ignition value, and free of any volatile materials. Thus , all impurities are also oxides. Table 3, also compiled from Reference 5, shows maximum and average impurity levels in RFETS PuO_2 material. Volatile materials such as chlorides, fluorides, sulfates and nitrates can produce significant corrosion of carbon steel in the presence of water.[6]

The maximum estimated temperatures in the Pu containing food cans will be uniform at 400°F (204°C) while the PuO_2 containing food cans will be 800°F (427°C) at the can wall and 950°F (510°C) in the middle of the oxide pile.[7]

Upon arrival at SRS, the 9975 packaging assemblies will be stored in Building 105-K process room, crane maintenance area, stack area, and the crane wash area. The building environment will essentially mimic outdoor weather patterns except that the assemblies will be shielded from wind, rain, and storms. Building temperatures will range from 0°F to 130°F (-18°C to 54°C) with relative humidity levels up to 100 %.[8] The high temperature of 130°C is based on the loss of ventilation. The building will be heated but not cooled.

3.0 INTERNAL CORROSION ANALYSIS OF Pu SHIPMENT AND STORAGE

3.1 Corrosion of the Food Can

If the coatings on the interior of the carbon steel food can are assumed to be ineffective in preventing corrosion, corrosion could occur with sufficient moisture. However, the only water available in the can with Pu metal is from the can atmosphere. This atmosphere is supposed to be dry. Pu oxidation is expected to consume any available moisture prior to Fe oxidation of the carbon steel can due to a higher free energy of formation. Thus, the carbon steel can should not show any corrosion degradation.

3.2 Plutonium Reaction with Food Can Atmosphere

Plutonium metal is assumed to be placed in the food can in a relatively dry condition. The atmosphere in the can is determined by the quality of the air in the loading glovebox. Due to the location of RFETS, the atmosphere is expected to be relatively dry as compared with that of SRS. However, the exact partial pressure of H_2O in the air is unknown and will have to be estimated based on literature values. The literature on plutonium corrosion in moist air and other oxygen containing atmospheres is reviewed by Waber[9] and by Colmenares.[10] These two studies are in general agreement although individual studies cover narrow temperature ranges. However, no correlation/interpretation was performed on the various observations. Also, the corrosion rate dependence on moisture levels was not defined. Continuing on this past work, Haschke[11-13] evaluated unalloyed Pu metal in well-defined environments and developed Arrhenius data[12] for reactions with water and oxygen in the high temperature region and with dry air in the low temperature region Figures 3 and 4. The anomaly that occurs with the drop in corrosion rate above 110°C (Figure 4) is due to the change in activation energy that is either positive or negative depending on the amount of water. The authors[12] state that this is due to a mechanisitic change in the kinetics of adsorbed H_2O. The high temperature Arrhenius data for both oxidants was normalized to 160 Torr, the partial pressure of oxygen in air. At temperatures at 200°C and above, the data is independent of moisture effects. This data is represented by the following formula (1):

$$\ln R\ (\mathrm{mg/cm^2min}) = 13.68 - (9010 \pm 280)/T \qquad (1).$$

Where: R = Corrosion rate, $\mathrm{mg/cm^2min}$
T = Temperature, K

Using formula 1 at 204°C, the predicted Pu corrosion rate ranges from 2.58×10^{-3} to 8.42×10^{-3} $\mathrm{mg\ cm^{-2}\ min^{-1}}$ or a total of 1.63×10^4 to 5.31×10^4 $\mathrm{mg\ cm^{-2}}$ for the 12 year shipping and storage period. This converts to 2.73×10^3 to 8.91×10^3 g (surface area = 167.7 $\mathrm{cm^2}$)[14] that exceeds the maximum weight of the Pu in the food can. These corrosion rates are also based on using unalloyed Pu. Additional testing was performed by Stakebake and Saba[15] on Pu-1 wt.%Ga alloy. This composition is similar to the RFETS Pu in Table 2. Their corrosion rate at 200°C and a water vapor pressure of 15 Torr was 4.0×10^{-4} to 2.9×10^{-2} $\mathrm{mg\ cm^{-2}\ min^{-1}}$. These two rates describe two different stages in the oxide growth. Stage I represents the growth of a protective oxide with the faster corrosion rate and Stage II, the period where porous oxide forms on top of the Stage I dense oxide layer. For a 12-year period, this converts to 2.52×10^3 to 1.83×10^5 mg $\mathrm{cm^{-2}}$. These rates are significantly higher but are probably the result of the difference in pressures, 160 Torr for the Haschke data and water vapor equal to 15 Torr for the Stakebake and Saba data. Since the Arrhenius data is based on thin film analysis of Pu samples after relatively short time exposure (less than 300 minutes), it does not adequately represent Pu after a 12-year exposure. Thus, some oxide will be formed but the amount of oxide conversion could not be predicted by these calculations due to the complexity of the oxide formation after long time periods. The significance of this is that if there were any moisture in the can, Pu will oxidize before corrosion of the carbon steel can occur based on the free energy of formation for PuO_2 equal to -524 kJoules/mole, and for Fe_2O3, -31 kJoules/mole.

3.3 Discussion of Potential Degradation Mechanisms

3.3.1 Food Can Embrittlement by Gallium (GA)

GA can cause failure of iron bearing materials by three potential mechanisms: (1) liquid metal embrittlement, (2) corrosion by Ga, and (3) by alloying with Ga. Butt[16] recently reviewed the potential of Ga diffusion into the 316 Stainless Steel (SS) 3013 system[17] containers. The 3013 high level waste containers could see maximum temperatures greater than 200°C near the centerline with wall temperatures of approximately 100°C for up to a 50-year storage period. Gallium is known as a strong embrittling element due to liquid metal embrittlement and was demonstrated in laboratory testing with 4340 steel, iron, and the alloy Fe-3Si.[18] Butt suggests that if the H_2O content is kept low, thereby limiting H_2 formation, the potential for Ga transport by gaseous diffusion would be negligible. Butt also showed that it is thermodynamically possible for Ga oxide to be reduced by Pu oxide to Ga at temperatures less than 527°C. But, the Pu temperature for this shipment will be a maximum of 204°C. There is very little information on past incidents of food can failure due to Ga embrittlement and the above mentioned data may not represent the actual atmosphere in the food can. A recent study by Williamson[19], reported in Reference 17, showed that low melting temperature eutectics of Pu metal and Pu-Ga alloys in stainless steel containers will not result in liquid formation or release plutonium by diffusion from long term storage (greater than 10 years) at 250°C. Diffusion processes require the diffusion couple to have very intimate contact. The formation of an oxide on the Pu surface is likely to prevent any such contact.

3.3.2 Plutonium/Iron Diffusion

Iron diffusion into the plutonium can potentially weaken the carbon steel food can by creating point defects in the BCC crystal structure.[20] Basic diffusion on one metallic element into another and its temperature dependence can be described by an Arrhenius equation (2):

$$D = D_o e^{-Q/RT} \tag{2}$$

The diffusion coefficient (D) is dependent on the experimentally determined diffusion constant (D_o) at temperature (T in K) with an activation energy (Q) and gas constant (R). Using this Arrhenius model to fit laboratory results, Haschke et al.[20] conclude that in delta phase Pu, no significant reaction occurs between Fe and Pu at temperatures of 140-150°C for up to 50 years. Delta phase Pu will not melt until 410-428°C, the eutectic temperature of the Pu-10 atomic percent Fe alloy.[21] Metallic corrosion processes are enhanced by the presence of a liquid phase. An example of this was described by Seidel et al.[22] in laboratory tests of metallic fuel (U-Pu-Zr alloy with up to 19% Pu) at temperatures above 700°C, the liquid eutectic temperature in irradiated fuel. The thickness of the cladding was reduced by only 26% after one hour at 800°C. The cladding was expected to be reduced further but the Zr layer on the alloy's surface retarded interdiffusion between the fuel and the cladding. It is probable that Fe diffusion into Pu will be limited (based on the 50-60°C difference) during the 12 year shipping and storage time for the RFETS material. However, the long term effect of Fe diffusion into Pu at 200°C needs further research. Haschke et al.[20] further conclude that impurity diffusion, such as Pu, into 316 stainless steel would not be a concern until temperatures are above 1000°C. Williamson[19] and an SRS study by Louthan[23] do not specifically eliminate the possibility of thinning of a stainless steel can due to iron diffusion into plutonium when a 250°C temperature exists for 10 years or more. However, as mentioned above, diffusion requires very intimate contact and will likely be inhibited by the oxides on the Pu metal surface.

Radiation enhanced diffusion of impurities within uranium can be a problem at temperatures approaching 1000°C.[20] The diffusion coefficent for Fe in Pu is expected to be similar to that of uranium. Since the maximum food can temperature with Pu is 204°C, radiation enhanced diffusion should not a problem for shipping and storage. On a practical side, no failures have identified in stainless steel containers with Pu metal inside due to Fe diffusion.

4.0 INTERNAL CORROSION ANALYSIS OF PuO_2 SHIPMENT AND STORAGE

Previous documentation[6,24] reviewed various corrosion reactions and corrosion rates that might occur between the RFETS SS&C (sand, slag, and crucible) materials and water in the carbon steel cans and stainless steel vessels of the 9975 packaging assembly. Some of this material included PuO_2. The corrosion interaction between the carbon steel can and water was based on the availability of up to 37.5 g. of free water (i.e. water sitting in the can). Any water used in that report was assumed to be available for reaction as a liquid. In reality, the literature indicates that any water in PuO_2 packaging will be in the form of a molecular layer adsorbed on the surface of the oxide. However, at a temperature greater than 100°C, the water will be vaporized. This brings up the possibility of dew point corrosion with this amount of water in the can could occur. With vaporized water in the food can, condensation may occur on a low temperature surface, the ID surface of the food can. This is not possible if the whole can is at the steady state temperature of 427°C to 510°C, as mentioned earlier. It was also assumed that the coating (organic or tin) on the carbon steel cans did not provide any corrosion protection. On the other hand, if the water is assumed to be free as a vapor and in contact with the carbon steel food can, corrosion may proceed per the following equation (2):

$$2Fe + H_2O + O_2 \longrightarrow Fe_2O_3 + H_2 \tag{2}$$

Considering that up to 37.5 g. of water may be available per can, one mole of water is required for two moles of iron (a 1:2 ratio). Using an atomic weight of 18 for one mole of water and 37.5 g. of available water, the resulting ratio is 2.08. This results in only 2.08 moles of available water per can. This also results in only 4.16 moles of Fe (atomic weight 56) available for corrosion. This amounts to 233 g. of Fe that can corrode or oxidize to Fe_2O_3. Thus, the corrected Formula 2 for the corrosion of Fe is:

$$4.16Fe + 2.08H_2O + 2.08O_2 \longrightarrow 2.08Fe_2O_3 + 2.08H_2 \tag{3}$$

Note that the amount of O_2 is not limited because the food cans are not leak tight. The dimensions of the 9975 shipping assembly are shown in Figure 2. Since the amount of corroded Fe is determined by the above corrosion calculations, the time it takes to consume the steel food can (Can A in Figure 2) also be calculated by using the can dimensions[2] and the standard corrosion calculation based on corrosion coupons.[25] The dimensions for Can A result in an inner surface area of 96.9 in^2. See Reference 25 for typical area calculations. The corrosion calculation (4) is as follows:

$$\text{Corrosion Rate (mils/year)} = \frac{\text{Weight loss (g.) x K (constant} = 3.45 \text{ x } 10^6)}{\text{Area (cm}^2\text{) x Time (hours) x Density (g/cm}^3)} \tag{4}$$

This equation is normally used to measure corrosion on the full surface area of the corrosion coupon, which in this case is the inner surface of the can. The constant K is used for unit conversion. This is a very conservative calculation for corrosion of the carbon steel can since it assumes that the entire ID surface will be corroded. The weight of the inner can A is 186 g. and the outer can B is 230 g..

Assuming that the water in the can will be of high purity and neutral pH with a corrosion rate of 50 μm (2 mils) per year or less,[26] the time to achieve the predicted weight loss can be calculated per Equation 5.

$$\text{Time (hours)} = \frac{\text{Weight loss (g.) x K (constant} = 3.45 \text{ x } 10^6)}{\text{Corrosion Rate (mils/year) x Area (cm}^2\text{) x Density (g./cm}^3)} \tag{5}$$

The time value of 9.1 years was calculated and presented in Table 4 as the number of years to consume the molar value of Fe (233 g.) and also as a % of the 12-year shipping and storage period. Can A (186 g.) will be consumed with sufficient moisture remaining to partly corrode Can B.

$$\text{Corrosion (g.) of Can B} \div 230 \text{ (in \%)} = (233 \text{ g.} - 186 \text{ g.})/230 = 47 \text{ g.} \div 230 = 20\ \% \quad (6)$$

With this amount of corrosion, a safe conclusion is that Can B may be penetrated. The 304L SS PCV will not be impacted since the available water is used in the oxidation of the steel cans.

If the composition of the stored PuO_2 is within the range of Table 1, calcined to an acceptable loss on ignition value, and no volatile materials (fluorides, chlorides, nitrates and sulfates) corrosion of the carbon steel can is not anticipated. Even if the carbon steel cans are penetrated using the assumption of free water, the integrity of the PCV is not at risk.

5.0 CONCLUSION

Corrosion calculations were performed using Pu metal exposed to relatively dry atmospheres. Although the calculations could not predict the long-term performance, some conversion to oxide is expected. Since no water is expected in the Pu filled package, corrosion of the carbon steel cans and stainless steel vessels is not expected. Potential diffusion mechanisms were discussed, but oxide formation on Pu should prevent intimate contact and subsequent diffusion during the 12-year period. Corrosion in the oxide filled packages is conservatively expected, but will not impact the integrity of the PCV. On an overall basis, internal corrosion of the Model 9975 shipping package with Pu or PuO_2 is not expected during the 12-year shipping and storage period. This conclusion is based on Pu contents greater than or equal to 98% and calcined PuO_2. If any metal shipments have less than 98% Pu, they can be reviewed on a case by case basis.

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ACKNOWLEDGEMENTS

The author appreciates the timely assistance from Susan Issacs of 773-A Library Services.

Table 1. Plutonium Metal Feed Specifications for the Weapons Program

Impurity	Upper Limit 1982 Report[ref]	Upper Limit 1985 Report[ref]
Aluminum + Silicon + Galium[1]	10 x (Al ppm) + 10 x (Si ppm) + 4 x (Ga ppm) ≤ 1300 ppm	10 x (Al ppm) + 10 x Si ppm) + 4 x (Ga ppm) ≤ 1300 ppm
Americium	200 ppm	200 ppm
Beryllium	1 ppm	3 ppm
Boron	5 ppm	50 ppm
Cadmium	10 ppm	10 ppm
Calcium	500 ppm	500 ppm
Carbon	200 ppm	200 ppm
Chromium	100 ppm	100 ppm
Copper	100 ppm	100 ppm
Iron +Nickel	Fe ppm + Ni ppm < 400 ppm	Fe ppm + Ni ppm < 400 ppm
Lead	100 ppm	100 ppm
Lithium	10 ppm	-
Magnesium	500 ppm	500 ppm
Manganese	100 ppm	100 ppm
Neptunium	50 ppm	100 ppm
Phosphorus	100 ppm	-
Tantalum	100 ppm	100 ppm
Thorium	50 ppm	100 ppm
Tin	100 ppm	100 ppm
Titanium	100 ppm	100 ppm
Tungsten	-	200 ppm
Uranium	100 ppm	100 ppm
Zinc	-	100 ppm
Zirconium	100 ppm	-

[1] Delta-phase stabilizing elements

TABLE 2. Maximum and Average Impurity Levels Found in Pu Metal During Elemental Analysis of Specific Group A and B metals (compiled from Reference 6) – Values are in ppm.

Element Group	Am	Al	Be	C	Ca	Cr	Cu	Fe	Ga	K	Mg	Mn	Ni	Pb	Si	Sn	Ta
A(CR)	-	61	0.22	122	500	136	12	518	120	120	1000	44	116	29	90	6	-
Average	-	46	0.07	-	107	38	7	388	58	5	305	4	70	9	9	5	-
A(ER)	91	21	<.38	53	18	42	8	196	-	-	-	-	11	17	42	-	30
Average	77	23	0.10	37	-	18	16	36	349	-	-	-	11	13	33	-	31
B(ER)	191	73	0.58	479	-	182	400	180	1.12%	-	-	-	1088	76	1295	-	150
Avg.	177	52	0.40	188	-	105	94	769	9611	-	-	-	289	45	89	-	88

Notes: CR – Calcium Reduction Process
ER – Electrorefining Process

TABLE 3. Maximum and Average Impurity Levels Found in PuO_2 During Elemental Analysis of Selected Batches (compiled from Reference 6) – Values are in ppm.

Element Group	Al	B	Be	Ca	Cd	Cr	Cu	Fe	Ga	K	Mg	Mn	Mo	Ni	P	Pb	Si	Sn	V
PuO_2	26	3	2.00	220	39	46	10	478	71	50	15	12	10	37	500	50	45	20	5
PuO_2 Average	17	-	0.79	106	-	34	6	388	41	20	4	10	-	27	-	27	19	14	-

TABLE 4. Fe Corrosion vs. Available Water

Water moles	Water g.	Fe moles	Fe g.	Corrosion Rate µm/yr	Time yr's	Time, % of 12 yr's
2.08	37.5	4.16	233	50	9.1	75

Note: The corrosion rate for Fe in high purity water is assumed to be 50 µm per year.

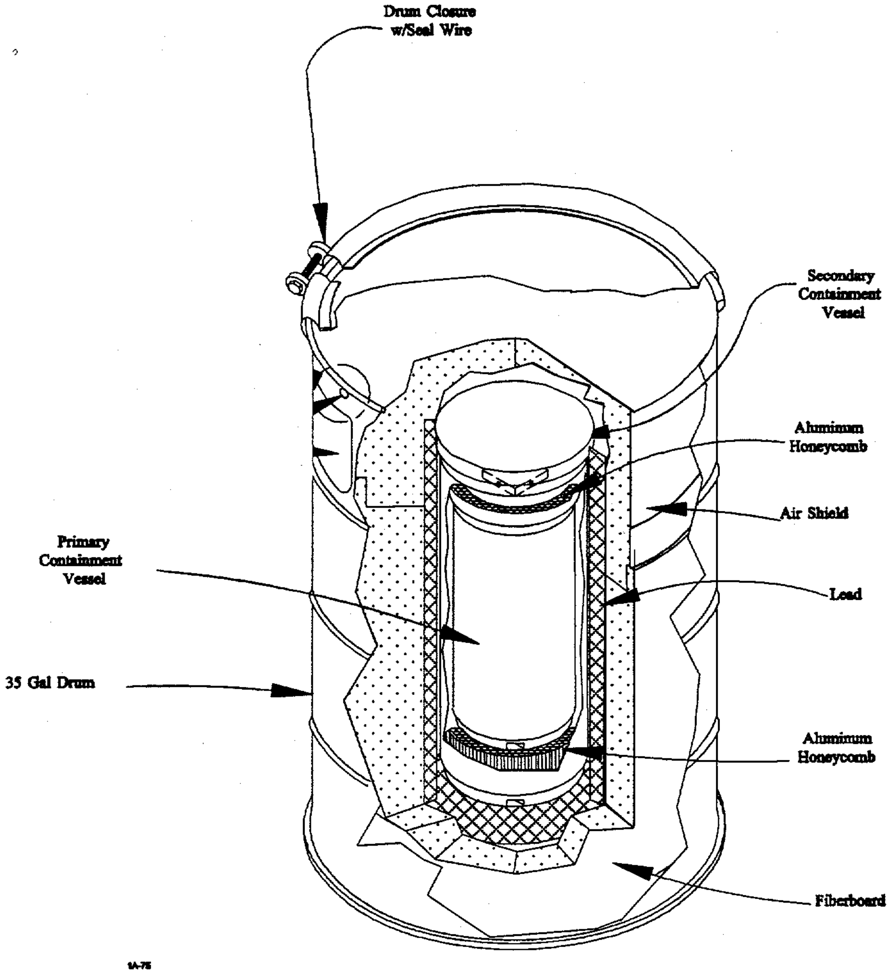

Figure 1. Overall Schematic of the Model 9975 Packaging Assembly.[1]

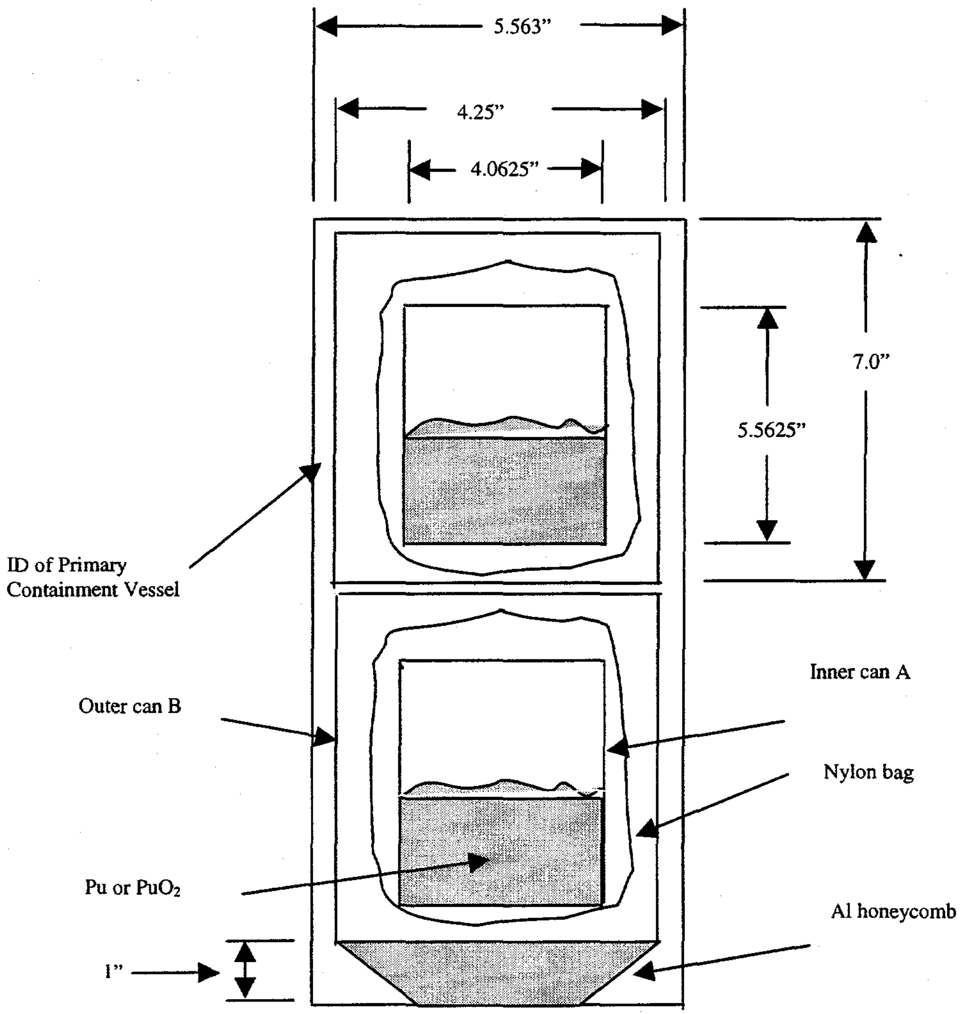

Figure 2. Author's conception of Model 9975 Shipping Container showing location of vessels and food cans with dimensions.[2]

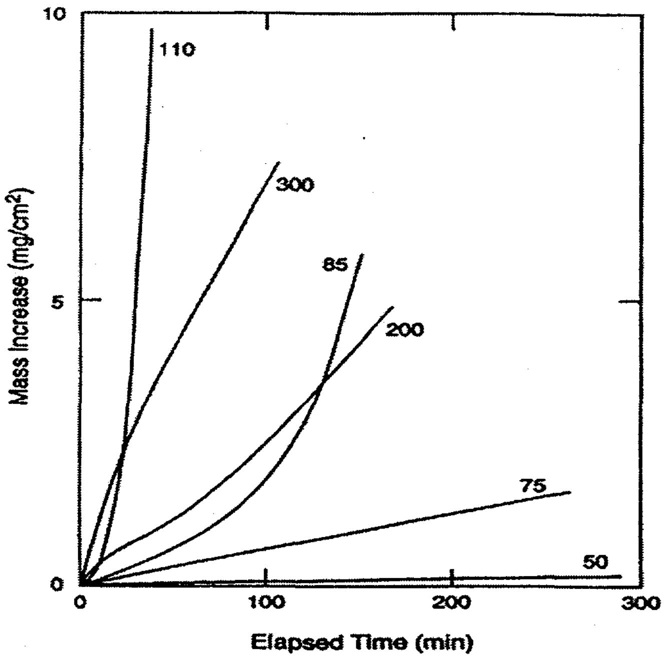

Figure 3. Time dependence of mass gain of unalloyed plutonium with water vapor at 15 Torr pressure and temperatures in the 50-300°C range. (From Ref. 12, p. 25)

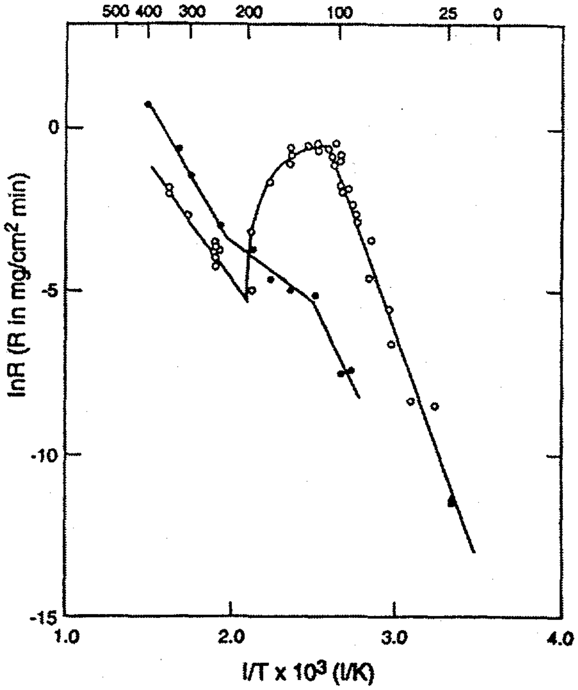

Figure 4. Arrhenius data for the reactions of unalloyed plutonium with water vapor and water-saturated air over the 25-400°C range. (From Ref. 12, p. 25)

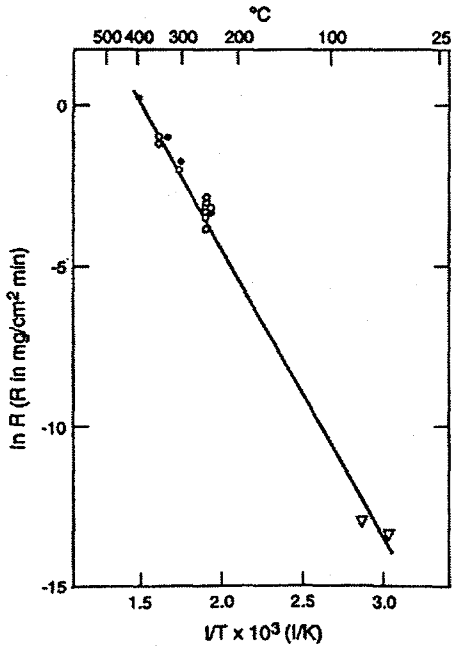

Figure 5. Combined Arrhenius data for the reactions of unalloyed plutonium with water and oxygen in the high temperature regime and with dry air in the low temperature regime. (From Ref. 12, p. 28)

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