INTERNAL/EXTERNAL CORROSION ANALYSIS
OF THE SAFKEG 3940A PACKAGE IN KAMS (U)

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DISCLAIMER

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
ASME	American Society of Mechanical Engineers
FTIR	Fourier Transform Infrared Spectroscopy
ICV	Inner Containment Vessel (SAFKEG 3940A)
OCV	Outer Containment Vessel (SAFKEG 3940A)
RFETS	Rocky Flats Environmental Technology Site
SARP	Safety Analysis Report for Packaging
SRS	Savannah River Site
SRTC	Savannah River Technology Center
SS	Stainless Steel

1.0   SUMMARY

An evaluation of the potential for corrosion of the stainless steel components of the SAFKEG 3940A package in the Savannah River Site K-Area Materials Storage (KAMS) was performed for a 10+ year storage mission.

Potential corrosive conditions include those due to the following sources:

- Plutonium materials within the DOE Standard 3013 container. The corrosion sources are the impurities and water that are ascribed to the plutonium material contents. The potential corrosion attack would be to the convenience can and inner container of the 3013 container.
- Constituents/impurities from the resin-bonded cork with moisture initially present in the cork and/or from atmospheric humidity. The potential corrosion attack would be to the Outer Containment Vessel (OCV) of the SAFKEG 3940A package and the inner liner of the keg of the SAKFEG package.
- Leachants from the Thermal-Insulating, Shock-Absorbing Foam (TISAF) with moisture initially present in the foam and/or from the atmospheric humidity through the plug in the bottom of the keg. The potential corrosion attack would be to the outer keg walls and inner keg liner.
- Atmospheric contaminants with atmospheric moisture.

The impurities and limited water (maximum 0.5%) of the plutonium materials in the 3013 container should stay bound in these materials and are not expected to cause significant corrosion to allow penetration of the inner container of the 3013.

Interdiffusion of the plutonium materials in the 3013 in contact with the steel was evaluated. No significant effects of interdiffusion are expected.

The resin-bonded cork contains a high level (approximately 5000 appm) of chloride). This chloride level, in combination with water, would be expected to cause pitting of stainless steel. Chloride stress corrosion cracking may also occur. The limited availability of water under the service conditions should reduce the potential for corrosion attack.

The TISAF was formed using acids (paratoluene sulphonic acid and ortho-phosphoric acid) as foaming agents. Limited immersion tests showed that pH reductions from approximately 7 to 2 occurred. This condition is not expected to cause significant attack to the stainless steel keg and liner.

The conclusions support the use of the SAFKEG 3940A package for interim use in KAMS, maintaining full containment and handleability, for a period of 10+ years. The conclusions are predicated on the surveillance of the package components to verify that corrosion attack significant to the performance of the 3940A package is not occurring

2.0 BACKGROUND AND ASSUMPTIONS

2.1 SAFKEG 3940 Shipping Package

Plutonium materials (metal and oxides) currently stored at the RFETS (Rocky Flats Environmental Technology Site) are to be placed in 3013 containers and shipped to SRS in a 9975 transportation package for interim storage prior to future disposition. Reports on external and internal corrosion analysis of plutonium materials for shipment and storage within the 9975 shipping package were issued previously.[1-2] The SAFKEG 3940A package[3] would be used to transport and store plutonium materials in 3013 containers from the Hanford Site (RL) that may be sent to SRS. An evaluation of the potential for corrosion of the stainless steel components of the 3940A package that provide containment and handleability

The SAFKEG 3940A shipping package design consists of a double-skinned stainless steel keg body (outer keg is 304 stainless steel (SS) with a 304L liner) with a bolted closure lid and O-ring seal (Figures 1-2).[3] The cavity between the keg and the liner is filled with a layer of phenolic foam, also known as TISAF (Thermal-Insulating, Shock-Absorbing Foam). This cavity is normally sealed but will vent through a vent plug (in bottom) during a fire accident condition. Within the liner is a resin bonded cork layer; two removable 304L stainless steel containment vessels, each sealed with a bolted lid and double O-rings as shown in Figures 1-2. Each container body in the SAFKEG 3940A is manufactured from stainless steel components that are welded. The 3013 storage container (Figure 3) is placed inside the inner containment vessel (ICV) and then inserted into the outer containment vessel (OCV). The various layers and their respective thicknesses are displayed in Figure 4.

The 3013 containment vessels will hold an inner stainless steel convenience can (A), containing the Rocky Flats Environmental Technology Site (RFETS) or Hanford material per Figure 3. The convenience can (stainless steel) was known previously as the food can (carbon steel). The use of bagless transfer systems at RFETS and Hanford have all but eliminated plastic bags used in handling of the convenience can. However, there is always a chance that a few 3013 vessels could be shipped with plastic bags inside. In the past the food can was placed into two, 0.005-cm thick nylon bags (double bagging) or other plastic film. The convenience can is placed within the inner 3013 container. The stainless steel convenience can is specified to be compatible with the inner and outer containers per the 3013 standard.[4] Type 304 or 304L are allowable materials per the standard. Two inner 3013 containers may be stacked on one another and placed into the outer 3013 container. In some situations, there may only be one inner container inside the outer container. The inner 3013 container will be supported on the bottom by an aluminum honeycomb structure to stabilize the cans within the outer 3013 container (Figure 3). Any interior corrosion occurring in this package will initiate from the inside of the initial convenience can A.

Five of the SAFKEG 3940A shipping containers are planned to be loaded on a galvanized pallet, similar to Figure 5. The loaded pallets will be stacked three pallets high for storage in KAMS.

2.2 Pu or PuO_2 Material

Multiple units of the SAFKEG 3940A packaging assembly will contain a maximum of either 4.40 kg of Pu or 5.0 kg of PuO_2.[4] This amount will be split in half so that each convenience can

contains a maximum of 2.20 kg Pu or 2.5 kg of PuO_2 or combined so that one convenience can contains the total amount. No water is specified in the package containing Pu metal but the most recent DOE STD-3013-2000 Standard allows up to 0.5 wt% water (25 g. maximum for two convenience cans) in the PuO_2 package.[4] The corrosion analysis assumes that Pu metal compositions will be within the range displayed in Table 1 and will contain a minimum of 98% Pu with impurity levels less than or equal to 2%.[5] 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. All oxide shipped to SRS from Hanford and RFETS will meet the acceptable composition limits per the 3013 standard. The PuO_2 material from RFETS and Hanford has to be calcined, tested to an acceptable loss on ignition value, and free of any volatile materials in order to meet the standard. 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 stainless steel in the presence of water.[6] Hanford oxide is assumed to be similar in composition and comparable impurity levels. Some of the Hanford product solution chemistry from single-pass and double-pass filtrates is displayed in Table 4.[7]

The loading of the SAFKEG 3940A shipping package produces a 19 watt output. The maximum estimated metal temperatures in the Pu containing convenience cans will be 320°F (160°C) while the PuO_2 containing convenience cans will be 442°F (228°C).[8] Temperatures of the SAFKEG 3940A components (Table 5) are a maximum of 266°F (130°C) for the ICV, 232°F (111°C) for the OCV, 225°F (107°C) for the cork and 164°F (73°C) for TISAF.[8] under normal storage conditions.

3.0 INTERNAL CORROSION

3.1 Summary

An internal corrosion analysis was performed for the SAFKEG 3940A 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 3013 304L stainless steel (SS) inner container and outer container and the stainless steel convenience cans containing Pu or PuO_2 is not significant to cause failure due to limited water content. The potential for embrittlement of the Secondary Containment Vessel (inner 3013 vessel) and the stainless steel convenience can due to iron diffusion into plutonium (or plutonium diffusion into the steel) and gallium diffusion into the steel has been assessed. This phenomenon should not be significant during the 10+ year storage period and the integrity of the stainless steel 3013 vessels will not be impacted during shipping or storage. There are sufficient data to establish the technical basis for safe storage of Pu and PuO_2 at the referenced conditions in the SAFKEG 3940A package. This is based upon performing surveillance inspections with verification that degradation is not occurring.

3.2 Specific Background and Assumptions

Upon arrival at SRS, the 3940A packaging assemblies will be stored in KAMS (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 120°F (-18°C to 49°C) with relative humidity levels up to 100 %.[8-9] Calculated internal temperatures for normal operation are presented in Table 5. The temperature of the inner 3013 container is expected to be maximum of between 320-442°F (160-228°C) based on the plutonium centerline temperature and configuration of the convenience cans.[8]

3.3   Corrosion of the Convenience Can Containing Pu

Corrosion of the stainless steel can is unlikely to occur if water is present within the can. However, the only water available in the can with Pu metal is from the can atmosphere. This metal is inserted into the can while in a relatively dry or inert atmosphere. If moisture is present, Pu oxidation is expected to consume any available moisture prior to Fe oxidation of the stainless steel can due to a higher free energy of formation. Thus, the stainless steel convenience can should not show any corrosion degradation.

3.4   Plutonium Reaction with Convenience Can Atmosphere

Plutonium metal is assumed to be placed in the convenience 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 and Hanford, the atmosphere is expected to be relatively dry as compared with that of SRS. However, information from Hanford oxide loading reveals the glovebox humidity in the 234-5Z building may vary from approximately 20% (winter) to the low 40% range (summer). The convenience can is then inserted into the inner container and backfilled with helium three times to reduce oxygen levels to less than 5%.[10]

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[11] and by Colmenares.[12] 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 previous work, Haschke[13-15] evaluated unalloyed Pu metal in well-defined environments and developed Arrhenius data[14] for reactions with water and oxygen in the high temperature region and with dry air in the low temperature region Figures 6 and 7. The anomaly that occurs with the drop in corrosion rate above 110°C (Figure 7) is due to the change in activation energy that is either positive or negative depending on the amount of water. The authors[14] state that this is due to a mechanistic 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):

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

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

Using formula 1 at 204°C, the predicted Pu corrosion rate ranges from 2.58 x 10^{-3} to 8.42 x 10^{-3} mg cm^{-2} min^{-1} or a total of 1.63 x 10^4 to 5.31 x 10^4 mg cm^{-2} for the 12 year shipping and storage period. This converts to 2.73 x 10^3 to 8.91 x 10^3 g (surface area = 167.7 cm^2)[16] that exceeds

the maximum weight of the Pu in the convenience can. These corrosion rates are also based on using unalloyed Pu. Additional testing was performed by Stakebake and Saba[17] 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} 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 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 stainless 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.

3.5 Discussion of Potential Degradation Mechanisms

3.5.1 Convenience 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[18] recently reviewed the potential of Ga diffusion into the 316 Stainless Steel (SS) 3013 system 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.[19] 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 convenience can failure due to Ga embrittlement and the above mentioned data may not represent the actual atmosphere in the convenience can. A recent study by Williamson[20], reported in Reference 4, 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. At temperatures above 800°C with exposure to Ga_2O/Ga_2O_3, 316L SS revealed significant oxidation, Ga uptake, and elemental distribution and embrittlement at 1150°C.[21] With Ga in a gaseous state at high temperatures, diffusion can occur. Since Ga is not likely to be in a gaseous state at 204°C, Diffusion should not occur. Plus, 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.

3.5.2 Plutonium/Iron Diffusion

Iron diffusion into the plutonium can potentially weaken the stainless steel convenience can by creating point defects in the BCC crystal structure.[22] 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.[22] 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.[23] Metallic corrosion processes are enhanced by the presence of a liquid phase. An example of this was described by Seidel et al.[24] 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.[22] further conclude that impurity diffusion, such as Pu, into 316 stainless steel would not be a concern until temperatures are above 1000°C. Williamson[20] and an SRS study by Louthan[25] 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.[22] The diffusion coefficent for Fe in Pu is expected to be similar to that of uranium. Since the maximum convenience 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.

3.6   Corrosion Analysis Of PuO_2 Shipment And Storage

Previous documentation[6,26] reviewed various corrosion reactions and corrosion rates that might occur between the RFETS SS&C (sand, slag, and crucible) materials and water in the stainless steel cans and stainless steel vessels of the 9975 packaging assembly. Some of this material included PuO_2. Previous corrosion calculations were based on the 9975 packaging assembly that could contain as much as 37.5 g. of free water (1.5 wt%).[27] However, for the SAFKEG 3940A containing the 3013 vessel, there is a maximum limit of 0.5% water (hydrolyzed in the oxide). Corrosion interaction between the stainless steel convenience can and water is based on a maximum of 25 g. of water.[4] Any available water is 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 convenience can, condensation may occur on a low temperature surface, the ID surface of the convenience can.

This is not possible if the whole can is at the steady state temperature above 200°C. On the other hand, if the water is assumed to be free as a vapor and in contact with the stainless steel convenience 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 25 g. of water may be available per can, one mole of water is required for every two moles of iron (a 1:2 ratio). Using an atomic weight of 18 for one mole of water and 25 g. of available water, the resulting ratio is 1.39. This results in only 1.39 moles of available water per can. This also results in only 2.78 moles of Fe (atomic weight 56) available for corrosion. This amounts to approximately 156 g. of Fe that can corrode or oxidize to Fe_2O_3. Thus, the corrected Formula 2 for the corrosion of Fe becomes:

$$2.78Fe + 1.39H_2O + 1.39O_2 \longrightarrow 1.39Fe_2O_3 + 1.39H_2 \tag{3}$$

Note that the amount of O_2 is not limited because the convenience cans are not leak tight. The dimensions of the convenience can (SAFKEG 3940A) is assumed to be similar to the food can in 9975 shipping assembly 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 convenience can (Can A in Figure 2) also be calculated by using the can dimensions[28] and the standard corrosion calculation based on corrosion coupons.[29] The dimensions for Can A result in an inner surface area of 96.9 in[2]. See Reference 29 for typical area calculations. The corrosion calculation (4) is as follows:

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

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 stainless steel can since it assumes that corrosion will occur on the entire ID surface. The approximate weight of the inner food can A is 186 g. and the inner 3013 container (B) is 230 g.

Assuming that the water in the can will be of high purity and neutral pH with a conservative corrosion rate of 50 μm (2 mils) per year or less,[30] the time to achieve the predicted weight loss can be calculated per Equation 5.

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

The time value of 6.2 years was calculated and presented in Table 6 as the number of years to consume the molar value of Fe (156 g.) and also as a % of the 12-year shipping and storage period. The convenience Can A (186 g.) will be partially consumed with no remaining moisture for further corrosion of the inner 3013 vessel. The convenience can is considered to be expendable.

Using a cathodic charge calculation method, using 25g. of water and a surface area of 1100 cm[2], corrosion penetration of the stainless steel can was calculated by Kolman[31] as only 39 μm (less than 0.001 mil). The surface area is approximately 75% higher than used in the previous

corrosion calculation, which may be the reason for the lower penetration than noted in the previous calculation.

Recent work at Los Alamos by Viers[32] suggests that loading pure PuO_2 powder in a glovebox humidity of less than 60%, the 0.5% water limit of DOE-STD-3013-2000 would not be exceeded. The other side to this is that with impure oxides, more water is adsorbed than with pure oxides and lower glovebox humidity is required.[33] However, even with the limited amount of corrosion with free water, the safe conclusion is that the inner 3013 SCV should not be penetrated. Thus, the 304L SS PCV will not be impacted since the available water is used in the oxidation of the convenience can per the above calculation.

If the composition of the stored PuO_2 is within the range of Table 1 and calcined to an acceptable loss on ignition value, corrosion of the stainless steel 3013 vessels is not anticipated. Even if the stainless steel convenience can is penetrated using the assumption of free water, the integrity of the PCV is not at risk.

3.7 Stress Corrosion Cracking

All oxide materials except for the SS&C oxides (from RFETS) are expected to have significant amounts of chlorides, less than 17%, due to various processing methods using sodium chloride, potassium chloride, and magnesium chloride. As a result stress corrosion cracking (SCC) is a possibility. Since general corrosion of the convenience can or moisture absorption by Pu or PuO3 would leave very little moisture to initiate a crack initiation site (pit) and with limited stresses to cause crack propagation, the likelihood of SCC is unlikely.[31]

3.8 Corrosion Analysis of TISAF

The TISAF foam is used as an insulator and held in a sealed annular space between two layers of stainless steel in the SAFKEG 3940A. The composition of TISAF below is drawn from the SARP unless otherwise specified. The TISAF material is manufactured by a proprietary process and formulation and is based on a phenolic resin (chemical formula $C_{7.4}H_{7.8}O$) filled with an unknown amount of calcium sulfate dihydrate (gypsum) (based on FTIR analysis and confirmed by XRD at SRTC). The foam composition includes the following; 39.4% carbon, 31.9% oxygen, 13.8% calcium, 11% sulfur, and 3.8% hydrogen. There are also small amounts of phosphorus in the foam. Chlorides at approximately 50 appm were measured using neutron activation analysis (at SRTC). Foam is produced by reacting the phenolic resin and filler with a paratoluene sulphonic acid (3%) and ortho-phosphoric acid (0.6%). All percentages are based on weight.[34] The acid is completely reacted in the foaming process. During the foaming process, the foam adheres to the stainless steel.[35]

In the 1970's and 1980's the roofing and chemical industries suffered bad publicity and replacement costs due to extensive corrosion of roof decking and piping from moisture absorption by foam insulation with subsequent acid and chloride release causing corrosion on steel.[36-37] Specific corrosion problems with steel were noted with phenolic foam insulation.[38]

Due to the historical problem with thermal insulation, tests were conducted on TISAF to determine moisture effects and chloride content. A reduced size TISAF sample was immersed in water for 96 hours per ASTM D-2842.[39] The single test result revealed a 46% water absorption. The TISAF exhibited a constant release of bubbles. Because of the gypsum

content and the closed cell nature of this foam, the high water absorption is thought to be the result of interconnecting paths between individual cells which allows air to be released as exhibited by the bubbles. Water absorption should no be revealed by air being released.

A similar water immersion test was conducted to determine if acid was released from the TISAF as exhibited by pH changes. Within minutes after immersion, pH levels went from 7 (distilled water) to 3.5 and ended at 2.2 after immersion for 96 hours.   Thus, the TISAF foam may be sensitive to moisture and an acid may be released. Of course, this depends on water getting to the TISAF. In the SAFKEG 3940A design, TISAF is sealed in the annular space between the outer keg and the inner liner. The only access to the TISAF is through the vent plug in the bottom of the SAFKEG 3940A through which the TISAF resin compound is originally poured. This vent plug will only open in the event of a fire accident condition per design. The test for chlorides revealed a level of 48 appm which is low enough to avoid chloride SCC in stainless steel. As long as the TISAF stays dry, no corrosion problems are expected with the surrounding stainless steels.

3.9 Corrosion Analysis of Resin Bonded Cork

The cork is a natural wood product produced in granular form and mixed with a synthetic resin binder. The mixture of the two is then compressed with heat to form the required shape. The synthetic resin is a polyurethane resin. A glue is also used and it is a two part compound based on urea formaldehyde with an alkyd plasticizer made by Rustins Ltd.[3] The outside surface of the cork is then sealed with an organic polymer coating. Since the resin bonded and sealed cork is preserved in the SAFKEG with an o-ring to provide for weather protection, it is unlikely that moisture (other than any on the interior during loading) will ever be a corrosion problem between the cork and the stainless steel liner and outer containment vessel. However, the statement in the SAFKEG 2863 SARP states that the O-ring seal is only designed for weather protection and is not designed to be airtight. Since high humidity air infiltration in KAMS is possible and water immersion tests were not conducted, water immersion tests on cork were performed. Using the same tests methods mentioned above for TISAF, the results indicate that 51% water was absorbed with a 19% increase in volume. The pH level at the end of a 96 hour immersion was 6.3. Chloride levels in the cork were measured to be 4890 +/- 65 ppm. This level of chloride would cause pitting of the stainless steel if the chloride was leached from the cork and sufficient water was present.

4.0   External Corrosion Analysis

4.1   Corrosion Analysis of SAFKEG 3940A Outer Container

Austenitic SS is generally a very corrosion resistant material. The composition of 304 SS is approximately 18-20 % chromium, 8-12 % nickel, 2 % manganese and 0.08 % carbon with limited amounts of phosphorus, sulfur, silicon, and nitrogen. The 316 SS grade is similar to 304 but with 4% molybdenum added to the composition. Chromium creates an oxide film that protects the material in oxidizing conditions while nickel aids in protecting the material in reducing conditions. Thus, 304 SS and 316 SS show resistance to both nitric and sulfuric acids. The L-grade reduces the carbon levels to 0.030 % or less to prevent sensitization (precipitation of chromium carbides) during exposure to heating above 800°F. However, the elemental composition of 304L and 316L SS does not prevent stress corrosion cracking from occurring

due to chloride exposure. The corrosion resistance of 304 SS in the outer keg surface is a very important consideration for environmental storage conditions.

While stainless steel's corrosion resistance to building storage conditions have not been found in the literature, exposure to industrial and marine atmospheres is available. Humidity, air temperatures, type of industrial pollution, vicinity to marine exposure, mating of dissimilar materials and fabrication type influence the behavior of austenitic stainless steel in these atmospheres. The SRS atmosphere tends to be more rural since the site is distant from the more populated and industrial areas. Although the SAFKEG 3940A shipping container will be sheltered, data from industrial and/or marine exposure should create a worst case scenario for the 304 SS container.   Table 7 contains marine exposure data (250 m from the ocean) for a 15-year exposure to austenitic SS.[40] The 304 SS sample was found to be spotted with a slight rust stain on 15 % of the samples surface with an average pit depth of 1.1 mils (0.028 mm). The maximum average pit depth for all the austenitic SS tested was 1.6 mils (0.04 mm). While the rust stain is probably due to films of soot and dirt containing iron, pitting is due to the presence of chlorides. In Table 8, the difference in two city atmospheres shows definite effects from chlorides. While samples exposed in the atmosphere in New York City for 26 years showed no rust staining, samples exposed in Niagara Falls near industrial plants producing chlorine and hydrochloric acid were attacked in less than a year.[41] Assuming that the atmospheric level of chlorides around SRS is more similar to New York, then it is to Niagara Falls, a conservative value of 0.0032 in. (0.08 mm) will be used as the maximum expected pit depth for the 105-K storage atmosphere over the 12-year period.   This value is double that of the maximum average pit depth in Table 7. This maximum pit depth value is approximately 4% of the thickness of 0.079 in. (2 mm) of the 3940A outer keg. The maximum pit depth dimension is also within the permissible variation in sheet thickness of $\pm$ 0.005 inches from manufacturing per ASTM standards.[42] Thus, very little corrosion is expected on the 3940 outer keg during storage conditions in Building 105-K.

4.2 Other Corrosion Concerns

The identification (ID) plate on the side of a SAFKEG shipping package is riveted to the outer keg wall per Figure 9. The open sides of the ID plate allow a crevice for corrosion to initiate. Thus, it is recommended that the ID plate be caulked on all sides with Dow Corning 732 RTV or equivalent with chloride levels tested to confirm levels are at or below 250 ppm. The SRS chloride limit for any material in contact with SS is a maximum of 250 ppm.[43]

 Galvanized carbon steel pallets are normally expected to last a long time, even in exterior applications. However, corrosion of zinc will increase with increasing moisture levels (primarily acid rain). Reference 44 discusses interior applications of hot dip galvanized steel and states that it can be used in wet (high humidity) interior applications for up to 20 years before any maintenance is required. A typical application in this environment is structural steel. One caveat is that this assumes that no penetrations have been through the zinc coating. Exposed carbon steel due to small pin hole in the zinc coating will not corrode due to cathodic protection. When large through coating penetrations are made such as a scratch from a drum being scrapped against the coating, the exposed area of the carbon steel may be too large to be cathodically protected. This produces surface corrosion that will be a visual defect. This will not cause significant degradation to the carbon steel pallet over the 12-year period.

Galvanized and painted carbon steel strapping is subject to corrosion during service to bind drums on a pallet. Rust stains have been observed (by the author) on D_2O drums (stainless steel) due to corrosion of the galvanized strapping. The drums were stored in the crane maintenance area of 105-K. Condensate will cause corrosion on existing breaks in the galvanized coating and will drip on the stainless steel drum. It is difficult for a coating to survive the tension, twisting, rubbing, and bending of strapping installation without a coating break. The eventual rust becomes a visual appearance problem. SS strapping should behave the same as mentioned above on the 304 SS outer container in the SAFKEG 3940A shipping assembly. Stainless steel strapping is the recommended material for strapping in this application.

5.0   Risk of Failure

Failures of containers storing plutonium and plutonium oxide materials DOE nuclear complex were investigated from published literature.[45] Two primary failure modes, out those investigated, were identified: 1) metal oxidation due to a non-airtight package and 2) gas pressurization from radiolytic and thermal degradation of inadequately stabilized materials. However, tens of thousands of packages did not fail, despite the lack of stabilization and packaging protocols. Both of these failure characteristics should be a non issue since the establishments of the requirements of the DOE-STD-3013-2000 standard. Only one failure out this study was determined to be the result of corrosion. The specific incident was the result of unstabilized Pu scrap leading to gas generation and corrosion of the tin plated steel can. The tin plated can was only designed to hold dry, stabilized materials. Material shipped to KAMS for storage will be stabilized which should eliminate this problem.

6.0   Conclusions and Recommendations

The impurities and limited water (maximum 0.5%) of the plutonium materials in the 3013 container should stay bound in these materials and are not expected to cause significant corrosion to allow penetration of the inner container of the 3013.

Interdiffusion of the plutonium materials in the 3013 in contact with the steel was evaluated. No significant effects of interdiffusion are expected.

The resin-bonded cork contains a high level (approximately 5000 appm) of chloride). This chloride level, in combination with water, would be expected to cause pitting of stainless steel. Chloride stress corrosion cracking may also occur. The limited availability of water under the service conditions should reduce the potential for corrosion attack.

The TISAF was formed using acids (paratoluene sulphonic acid and ortho-phosphoric acid) as foaming agents. Limited immersion tests showed that pH reductions from approximately 7 to 2 occurred. This condition is not expected to cause significant attack to the stainless steel keg and liner.

The conclusions support the use of the SAFKEG 3940A package for interim use in KAMS, maintaining full containment and handleability, for a period of 10+ years. The conclusions are predicated on the surveillance of the package components to verify that corrosion attack significant to the performance of the 3940A package is not occurring. Corrosion of the galvanized steel pallet is expected to cause a change in visual appearance only during the storage period.

7.0   REFERENCES

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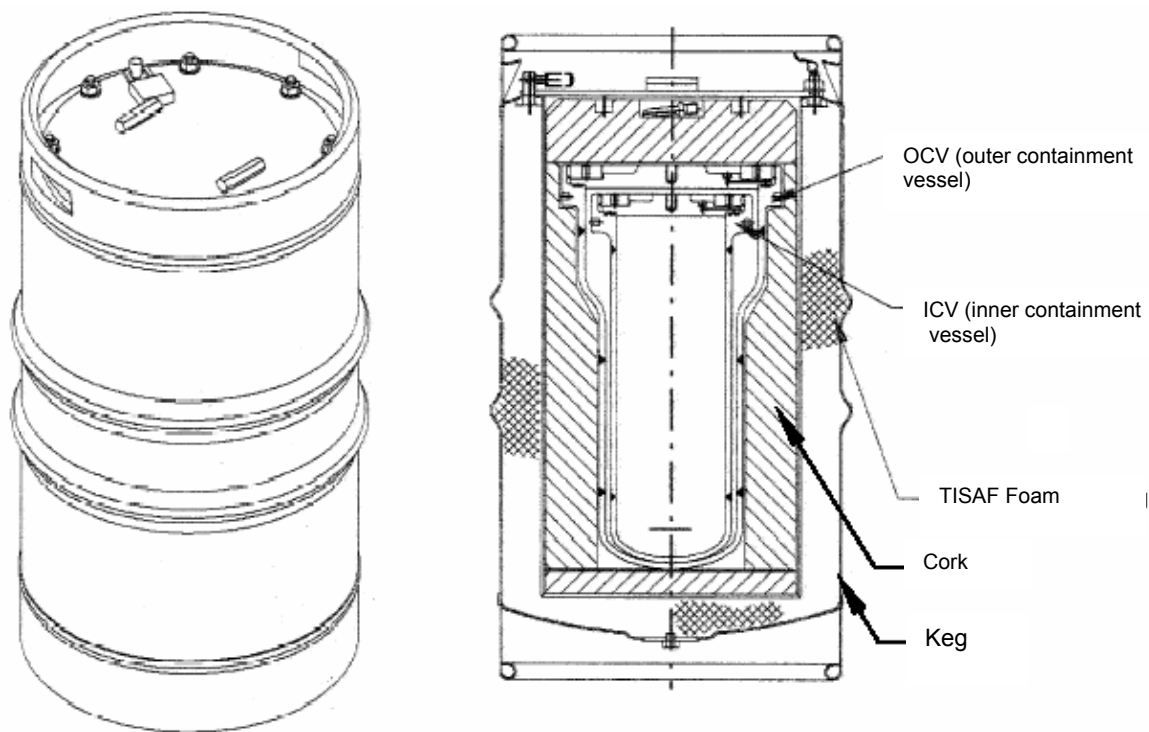
Figure 1. SAFKEG 3940A shipping package showing location of the keg, TISAF foam, cork, outer containment vessel (OCV), and the inner containment vessel (ICV). The 3013 vessels will be inserted into the ICV of the SAFKEG. The SAKEG is approximately 40 inches high.

Double O-rings in OCV

OCV Closure Lid

ICV Closure Lid

Double O-rings in ICV

OCV

ICV

Weld

Figure 2. Inner and outer containment vessels in SAFKEG 3940A. The vessels are welded 304L stainless steels.
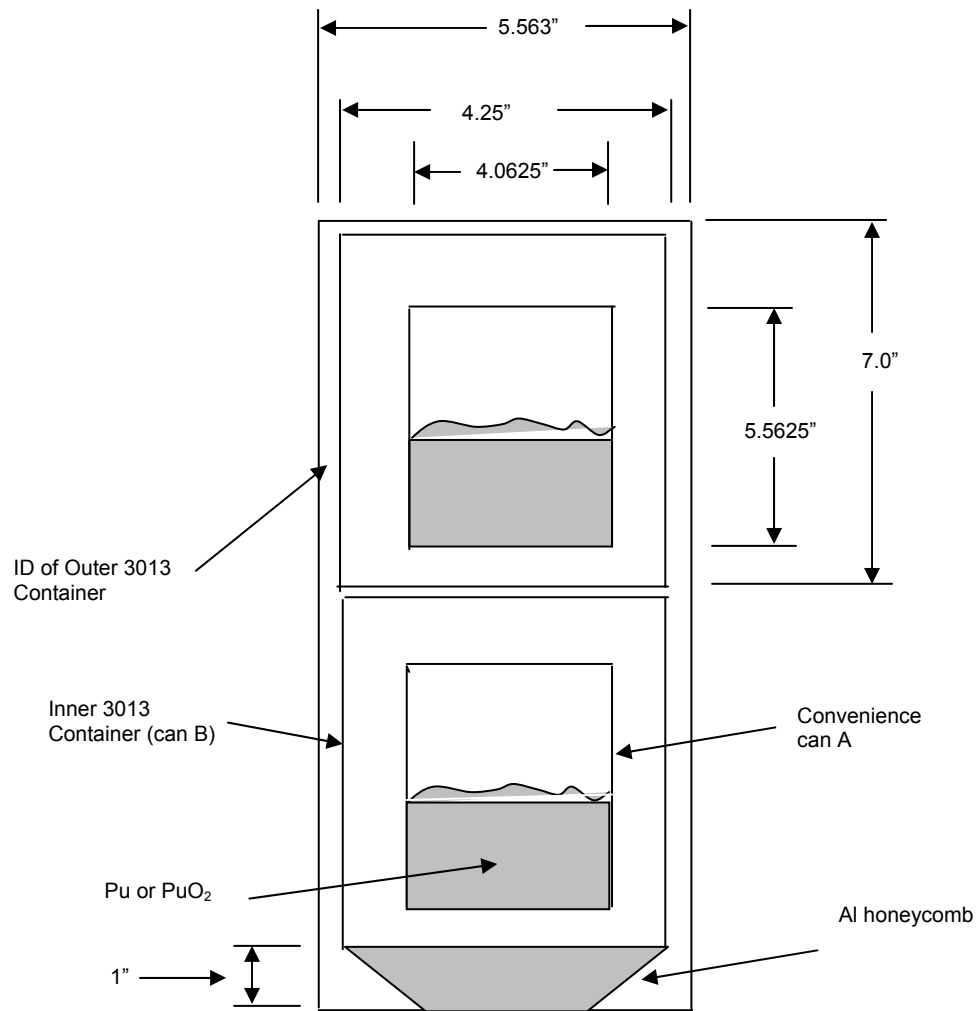
Figure 3. Author's conception of filled 3013 vessels showing location of 3013 inner and outer containers, and convenience cans with approximate dimensions.[4]

Thicknesses

☐ - Keg 304 SS 2 mm (0.079 in.)

▨ - TISAF Foam 45.5 mm (1.79 in.)
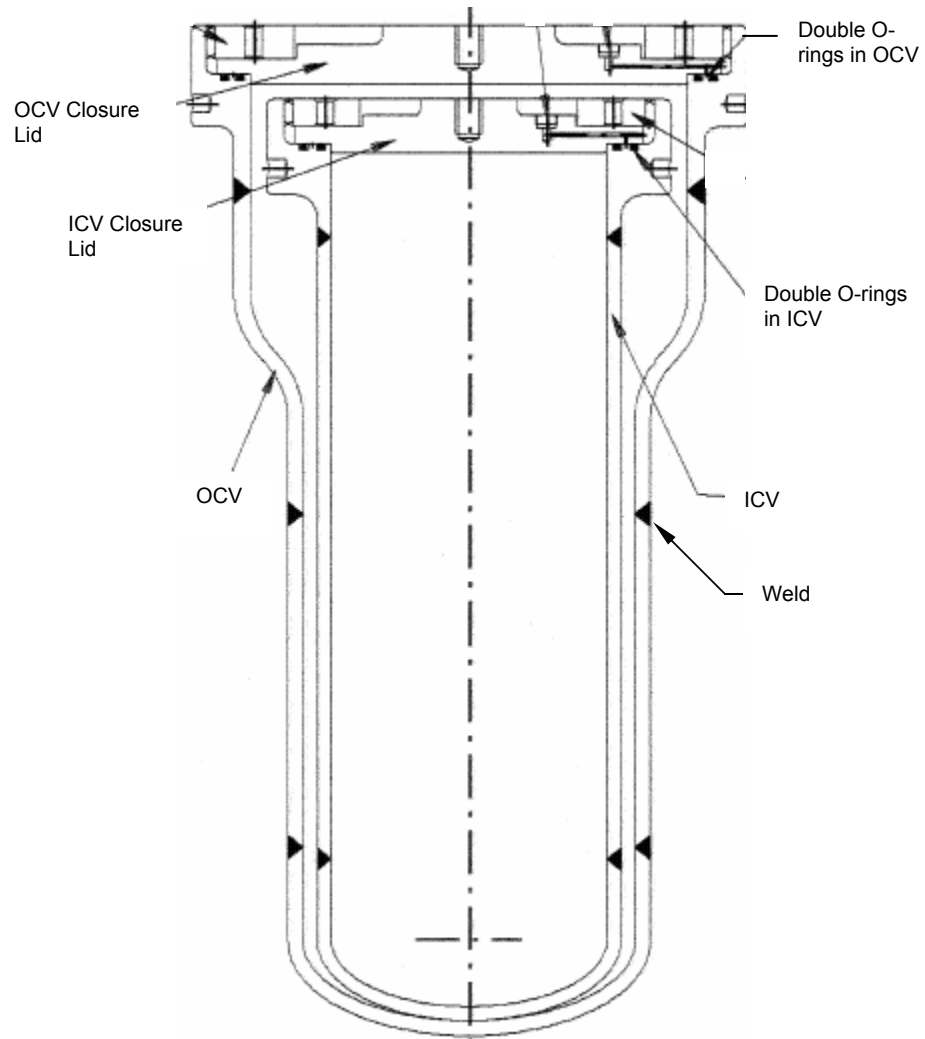
▦ - Liner 304L SS 3 mm (0.118 in.)

▨ - Cork 59.9 mm (2.36 in.)

▤ - OCV 7.11 mm (0.280 in.)

▦ - ICV 6.55 mm (0.258 in.)

◆ - 3013 Outer Can 3.23 mm (0.127 in.)

▩ - 3013 Inner Can 1.52 mm (0.060 in.)
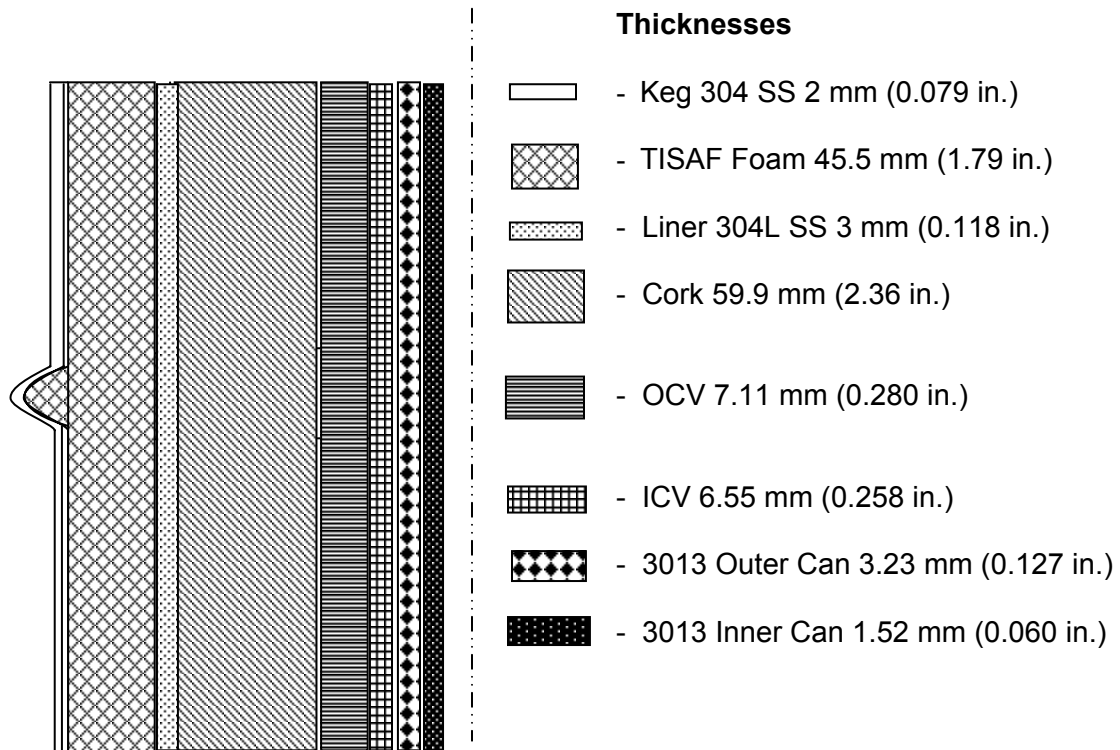
Figure 4. Side cross-section of SAFKEG 3940A and enclosed 3013 vessels with approximate component thicknesses. Air gaps are not shown.

Figure 5. Digital photo of five SAFKEG drums on a galvanized steel pallet. Note strapping (A) securing drums to pallet. Pallet size is 4 feet by 4 feet. Drums will be stored 3 pallets high.
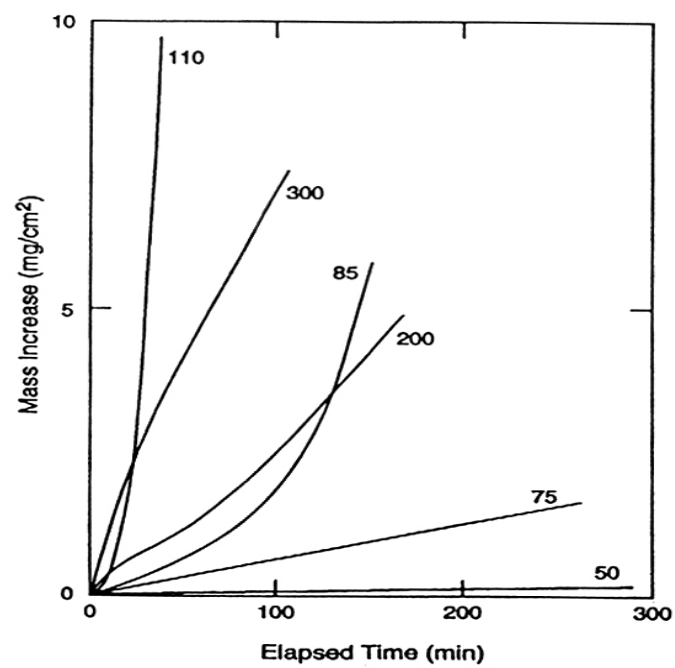
Figure 6. Time dependence of mass gain of unalloyed plutonium with water vapor at 15 Torr pressure and temperatures in the 50-300°C range. (From Ref. 15, p. 25)
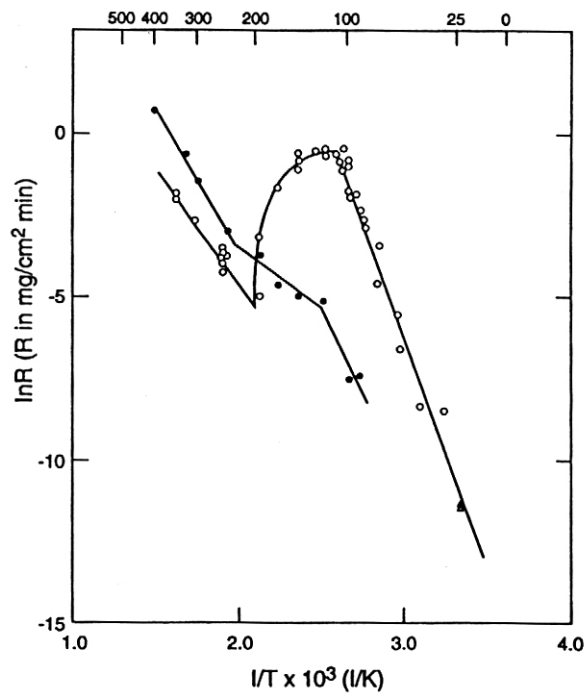
Figure 7. Arrhenius data for the reactions of unalloyed plutonium with water vapor and water-saturated air over the 25-400°C range. (From Ref. 15, p. 25)
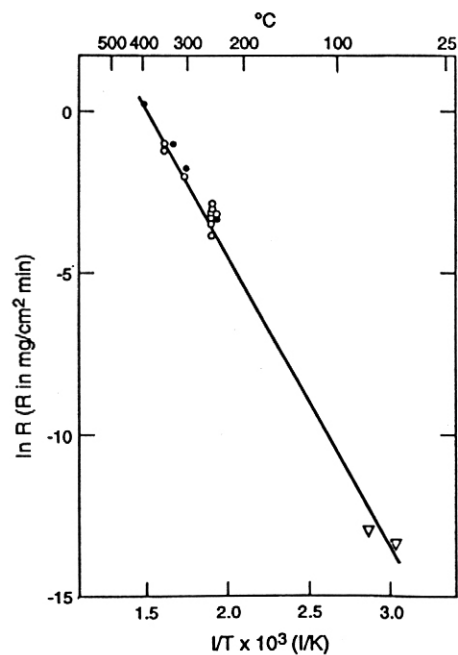
Figure 8. Combined Arrhenius data for the reactions of unalloyed plutonium with water and oxygen in the high temperature regime and with dry air in the low temperature regime. (From Ref. 14, p. 28)
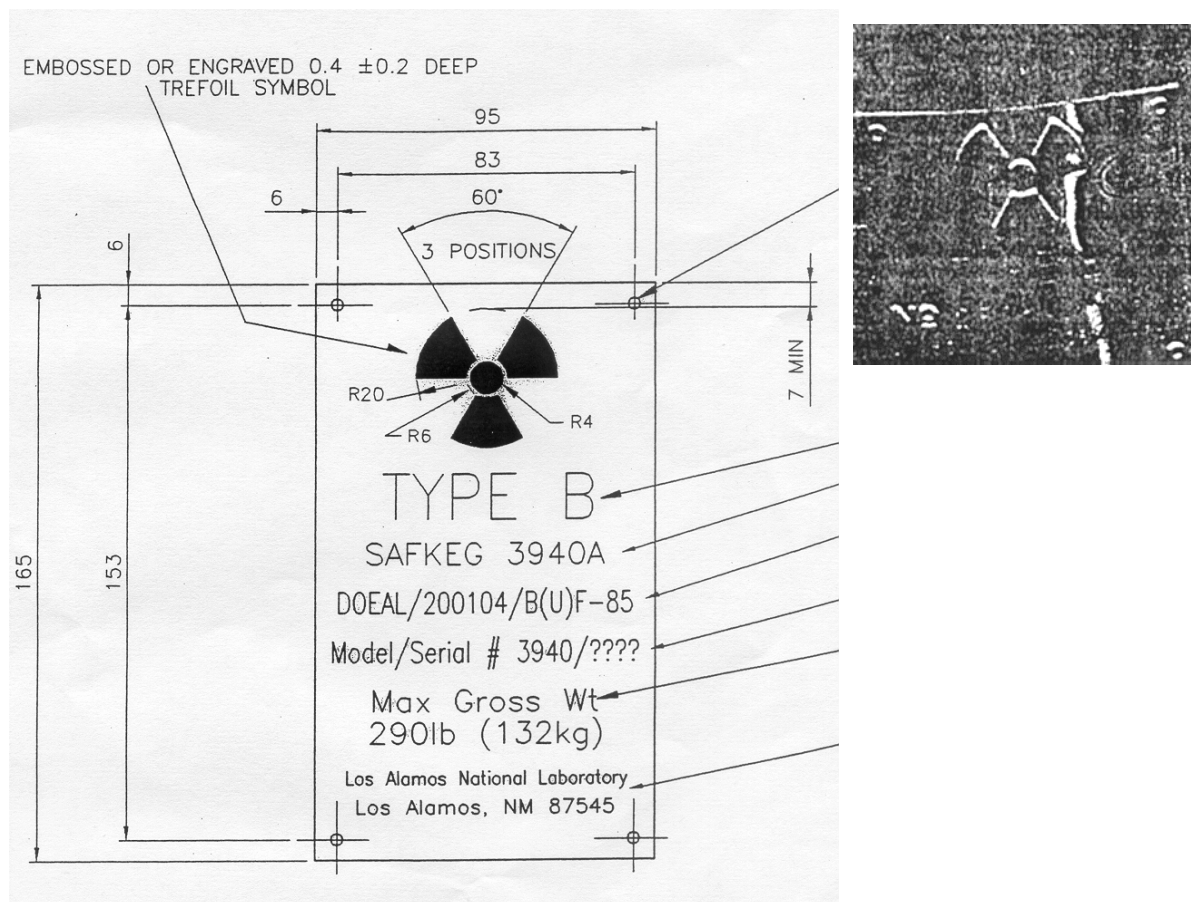
Figure 9. Schematic Drawing of the identification nameplate for the SAFKEG 3940A package (inset is an image of nameplate on a SAFKEG showing rivets).[3]

Table 1. Plutonium Metal Feed Specifications for the Weapons Program[5]

| Impurity	Upper Limit 1982	Upper Limit 1985
Aluminum + Silicon + Galium[1]	10 x (Al ppm) + 10 x (Si ppm) + 4 x (Ga ppm) $\leq$ 1300 ppm	10 x (Al ppm) + 10 x Si ppm) + 4 x (Ga ppm) $\leq$ 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 5) – Values are in ppm.

Element	Am	Al	Be	C	Ca	Cr	Cu	Fe	Ga	K	Mg	Mn	Ni	Pb	Si	Sn	Ta
Group 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 5) – Values are in ppm.

Element	Al	B	Be	Ca	Cd	Cr	Cu	Fe	Ga	K	Mg	Mn	Mo	Ni	P	Pb	Si	Sn	V
Group 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	-

Element	Barney, 1996 μg/mL B	Jones, 2001 SPF Solution C μg/mL
Iron	15700	15200
Sodium	12800	12960
Aluminum	3500	4608
Managanese	NR	2800
Chromium	750	2464
Potassium	6500	2440
Nickel	510	2016
Phosphorus	NR	1976
Calcium	600	912
Magnesium	480	295
Zinc	NR	816
Plutonium	31400	24000
Americium	61	NR
Uranium	180	NR
Hydrogen Ion	3.37M	7.0M
Nitrate Ion	210000	NR
Chloride Ion	26600	NR
Fluoride Ion	1100	NR
NR – Not Reported		

Table 4. PuO_2 composition of Hanford single-pass and double-pass filtrates (Ref. A)

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TABLE 5. SAFKEG Maximum Temperatures for Normal Conditions[8]

Ambient Temperature (°F)	SAFKEG Temperature (°F)				SAFKEG Limit* (°F)
	ICV	OCV	Cork (Limit)*	TISAF (Limit)*	
120	266	232	225(356)*	164(284)*	320

* Cork and TISAF Limits from SAFKEG SARP

Table 6. 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
1.39	25	2.78	156	50	6.2	52

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

Table 7. Corrosion of AISI 300-Series Stainless Steels in a marine atmosphere[41]
(Based on 15–year exposures 250 m (800 ft.) from the ocean at Kure Beach, NC)

AISI Type	Average Corrosion Rate		Average Depth of Pits		Appearance(a)
	mm/yr	mils/yr	mm	mils	
301	<2.5 x 10-5	<0.001	0.04	1.6	Light rust and rust stain on 20% of surface
302	<2.5 x 10-5	<0.001	0.03	1.2	Spotted with rust stain on 10 % of surface
304	<2.5 x 10-5	<0.001	0.028	1.1	Spotted with slight rust stain on 15 % of surface
308	<2.5 x 10-5	<0.001	0.04	1.6	spotted by rust stain on 25 % of surface
316	<2.5 x 10-5	<0.001	0.025	1.0	Extremely slight rust stain on 15 % of surface
317	<2.5 x 10-5	<0.001	0.028	1.1	Extremely slight rust stain on 20 % of surface

(a) All stains easily removed to reveal bright surface

Table 8. Atmospheric Corrosion of Stainless Steels at Two Industrial Sites[41]

Type(a)	New York City(industrial)		Niagara Falls (industrial-chemical)	
	Exposure time, years	Specimen surface evaluation	Exposure time, years	Specimen surface evaluation
302	5	Free from rust stains	<2/3	Rust stains
302	26	Free from rust stains		
304	26	Free from rust stains	<1	Rust stains
304			6	Covered with rust spots and pitted
316	23	Free from rust stains	<2/3	Slight stains
316			6	Slight rust spots, slightly pitted
317			<2/3	Slight stains

(a) Solution-annealed sheet, 1.6 mm (1/16 in.) thick