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3013/9975 Surveillance Program Interim Summary Report

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3013/9975 Surveillance Program Interim Summary Report

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3013/9975 Surveillance Program Interim Summary Report (U)

I. INTRODUCTION AND CONCLUSION

The K-Area Materials Storage (KAMS) Documented Safety Analysis (DSA) requires a surveillance program to monitor the safety performance of 3013 containers and 9975 shipping packages stored in KAMS. The SRS surveillance program [Reference 1] outlines activities for field surveillance and laboratory tests that demonstrate the packages meet the functional performance requirements described in the DSA. The SRS program also supports the complex-wide Integrated Surveillance Program (ISP) [Reference 2] for 3013 containers. The purpose of this report is to provide a summary of the SRS portion of the surveillance program activities through fiscal year 2010 (FY10) and formally communicate the interpretation of these results by the Surveillance Program Authority (SPA).

Surveillance for the initial 3013 container random sampling of the Innocuous bin and the Pressure bin has been completed and there has been no indication of corrosion or significant pressurization. The maximum pressure observed was less than 50 psig, which is well below the design pressure of 699 psig for the 3013 container [Reference 3]. The data collected during surveillance of these bins has been evaluated by the Materials Identification and Surveillance (MIS) Working Group and no additional surveillance is necessary for these bins at least through FY13. A decision will be made whether additional surveillance of these bins is needed during future years of storage and as additional containers are generated. Based on the data collected to date, the SPA concludes that 3013 containers in these bins can continue to be safely stored in KAMS.

This year, 13 destructive examinations (DE) were performed on random samples from the Pressure & Corrosion bin. To date, DE has been completed for approximately 30% of the random samples from the Pressure & Corrosion bin. In addition, DE has been performed on 6 engineering judgment (EJ) containers, for a total of 17 to date. This includes one container that exceeded the 3013 Standard moisture limit which was opened at LANL. The container pieces and an oxide sample were sent to SRNL for examination in FY11. No significant pressurization has been observed for the Pressure & Corrosion bin containers. Relatively minor corrosion has been observed on some convenience containers and the inside of two inner containers. While the limited extent of corrosion does not jeopardize the integrity of the outer 3013 containers, it does highlight the importance of continuing to perform DE and the Shelf Life program to assure that the corrosion rate is not accelerating or changing to a different corrosion mechanism (e.g., stress corrosion cracking). Statistical sampling is currently scheduled to be completed in FY17, but there is a proposed reduction of the number of DE's per year for FY11 and beyond which may delay the completion date.

Since 3013 containers are stored inside 9975 containers, surveillances of 9975 containers are performed in conjunction with 3013 container surveillances. Results of 9975 container non-destructive examinations (NDEs) and DEs indicate that the containers will provide adequate protection of the 3013 containers in K-Area storage for at least 15 years [Reference 4].

II. BACKGROUND

The 3013 surveillance sampling approach is defined in the Surveillance and Monitoring Plan (S&MP) [Reference 5]. The approach combines statistical and EJ sampling to provide a powerful, cost-effective method for ensuring the safe storage of 3013 containers. To select the statistical sample, the population of containers is organized into three bins based on a container's contents and potential degradation mechanism. Using pressure and corrosion as the two potential degradation mechanisms, the three bins are: Pressure & Corrosion, Pressure, and Innocuous. The requirement of 99.9% probability of observing at least one of the worst 5% (in terms of potential degradation) is used to guide the statistical sampling process for the Pressure & Corrosion and Pressure bins [Reference 5]. The sample size for the Innocuous bin is based on evaluating the assumption that these containers will show no degradation; therefore, these containers will have almost no variability in the surveillance results. The EJ sampling uses engineering judgment and results of the MIS shelf-life studies to augment the statistical sample with additional containers that are judged to have the greatest potential for degradation.

The statistical component of the ISP includes 258 randomly selected containers from the Pressure and the Pressure & Corrosion bins that will achieve a 99.9% confidence of inspecting at least 1 of the worst 5% of the population [Reference 6]. To reach this confidence level for the Pressure bin containers, non-destructive examination (NDE) of 130 containers and destructive examination (DE) of 6 containers were performed over 5 years (FY05 – FY09). For the Pressure & Corrosion bin containers, DE of 128 containers are being performed over 11 years (FY07 – FY17) [Reference 7]. Note that NDE is also performed prior to DE. To augment the random sample, engineering judgment (EJ) is used to select containers deemed to have the highest potential for corrosion and/or pressure generation. A different approach was chosen for the Innocuous bin. Ten random items were selected to validate the assumption of no significant degradation of containers relative to containers in the other bins. NDE of randomly selected containers may be performed after the 3013 container reaches a minimum age of 3 years as determined by the weld date of the inner 3013 container. The minimum age for randomly selected DE items is 5 years. There is no age restriction for 3013 containers selected by engineering judgment or the 9975 package surveillance program. Table 1 shows a breakdown of the number of each surveillance type for each bin. Table 2 shows the number of NDE and DE surveillances performed at SRS in each fiscal year.

Surveillance is performed on 9975 packages in conjunction with the 3013 that is stored inside it. Since the 9975 packages are all essentially identical and the 3013s are selected randomly, it is assumed that the NDE surveillance of the 9975 packages is also random. DE is performed on one 9975 per year as a confirmatory test to evaluate the materials properties.

III. DISCUSSION:

The 3013/9975 surveillance program has made significant progress at SRS and achieved all major programmatic milestones planned through FY10. Complex-wide in FY05 through FY10, all of the scheduled random NDE and DE surveillances specified in the program were performed. This accounts for all of the random surveillances specified in References 6 and 7 for the Innocuous and Pressure bins and approximately 30% of the random surveillances for the Pressure & Corrosion bin specified in the program. DE has also been performed on 16 EJ containers in the Pressure and Corrosion bin. In addition, one container packaged at Hanford exceeded the allowable moisture content and it was shipped to LANL for opening. LANL opened the container and measured the moisture content and the gas pressure and composition. The can pieces and an oxide sample were shipped to SRNL for examination and analysis in FY11. Random surveillance for the Pressure bin and the Innocuous bin was completed in FY09. Since additional containers are currently being packaged at LANL and LLNL, the program will be evaluated to determine whether additional surveillance of these bins is needed. If additional surveillances for these bins are needed, they would not be performed until after FY13 so that the minimum 3 year age can be met. DE of Pressure & Corrosion bin containers is currently scheduled to continue through FY17.

The surveillance program requires a combination of field inspections and laboratory tests to validate the technical assumption regarding package performance and life expectancy. This report will provide information and data analyses for the surveillances performed in FY10 and correlate these results with the associated SRNL/LANL testing program.

Table 3 summarizes the SPA Evaluations initiated during FY10 DE surveillances for 3013 and 9975 containers. Data for the FY05 and FY06 NDE surveillances are included in References 8 and 9. Data for FY07 through FY09 NDE and DE surveillances are included in the FY07-09 Summary Report [Reference 10].

1. 3013 NDE Surveillance

Two types of surveillances are required by the program. Some containers require NDE only and some containers require NDE/DE. During FY09, 2 containers were observed to contain foreign material, one contained a glove and other contained a sampling scoopula [Reference 10]. As a result, MIS selected 16 containers for special digital radiography inspection in FY10 to evaluate whether foreign material was present in the containers. Table 2 summarizes the number of NDE and NDE/DE surveillances performed each year. Five separate NDE techniques were performed on each of the 3013 containers to evaluate their performance. The results from these evaluations provide the evidence necessary to validate that the 3013 container integrity and internal pressure meet the 3013 Standard technical assumptions for a 50 year design life and ensure the container is acceptable for continued storage in KAMS. A brief description of each NDE technique and the corresponding surveillance results are provided below.

i. Digital Radiography

Digital radiography is used to monitor the internal pressure of a 3013 container by looking for changes in the deflection of the inner 3013 container lid as compared to the baseline radiograph at the time of packaging. Inspected containers were packaged at Rocky Flats, Hanford, and SRS. The radiographs have been reviewed. A conservative correlation is used to estimate the pressure that could be present in the inner container and these results are summarized in Table 4. Data for FY05, FY06, and FY07-FY09 are documented in References 8-10, respectively. In all cases, the indicated pressure is less than 100 psig. This is consistent with the pressures estimated based on the performance of the MIS test containers at LANL and well below the container design pressure specified by the 3013 Standard (699 psig).

Baseline radiographs obtained immediately after packaging focused on only the inner container lid to allow for evaluation of pressure increase. Surveillance radiographs composite three radiographs to cover the full container. Full container radiography provides at least partial evidence of the integrity of the inner containers. Although this technique is not sensitive enough to detect small particles or thru-wall cracks or pits, a gross failure of the convenience container or inner container would be detected if plutonium bearing material were to be observed in the annulus region between convenience and inner container or between the inner and outer container. All radiographic images have been reviewed and there is no evidence of plutonium bearing material in the annular regions.

At least three sets of radiographs were performed when inspecting for foreign material. Since this inspection is most effective for the surface of the oxide bed, radiographs were taken at 0 and 90 degrees, and then the oxide was agitated by rolling the container on the table prior to each set of subsequent radiographs. Two additional containers (H001401 and H003070) were found to contain sampling scoopulas. The presence of the stainless steel scoopula was determined not to jeopardize the safe storage of the 3013 container. Therefore, they were returned to the MSA for long term storage.

ii. Contamination Survey

A contamination survey is used as one of three techniques to validate the integrity of the outer 3013 container. All containers were found to meet clean area limits for alpha and beta-gamma as defined by SRS Manual 5Q and therefore meet the programmatic acceptance criteria ($\leq$ 20 dpm/100 cm^2 alpha).

iii. Mass Measurement

Mass change is used as one of three techniques to validate the integrity of the outer 3013 container. The containers have variable loading and thereby variable container densities, so a buoyancy correction is applied to all baseline RFETS values to account for the difference in atmospheric pressure at SRS. All containers were found to be below the programmatic criteria ($\leq$ 1.0 gram difference from baseline).

iv. Visual Inspection

A visual inspection is used as one of three techniques to validate the integrity of the outer 3013 container. The visual inspection looks for signs of corrosion attack on the outside surface of the outer 3013 container or other anomalous conditions. All containers were inspected and no corrosion or other degradation was observed. Even in the case of NDE-13 where a small quantity of free standing moisture was found inside the PCV of 9975-06704 (Figure 1) and on the exterior of the 3013 (Figure 2), there was no degradation. The field observations provide evidence that there is no corrosion mechanism attacking the outside of the outer 3013 container as a result of the KAMS storage environment and outer container integrity is acceptable for continued service.

Figure 1. Moisture inside the PCV

Figure 2. Evidence of moisture on the outside of the 3013

v. Prompt Gamma

A prompt gamma (PG) measurement is performed to identify an impurity fingerprint of the material inside the 3013 container. The types of impurities present within the 3013 help determine the correct surveillance bin which is based on the potential failure mode for the container. All 3013 containers have been assigned to one of the following three bins:

1) Innocuous,
2) Pressure,
3) Pressure & Corrosion

The surveillance program requires a PG measurement with a minimum 60 minute live time count to ensure the container is designated in the correct surveillance bin. A baseline PG was not performed on some RFETS containers, and when it was, the count time was less than 15 minutes. Hanford containers were generally counted for 60 minutes. The longer count time increases the sensitivity of the measurement. The PG measurements are compared to the packaging site baseline (if available) and the binning selection criteria [Reference 6].

As reported in the previous Summary Report [Reference 10], three PG measurements for Hanford items did not match their baseline results. In FY10, two additional PG mismatches were observed (H004000 and H004309). During evaluation of these mismatches, it was discovered that there were systematic errors that occurred during the baseline PG measurements at Hanford. Reference 11 documents the cause and identifies 258 containers that were affected. In most cases a corrected PG result was available. In 13 cases, however, the correct baseline data was discarded. SRS will measure the PG for those containers in FY11. At least 70 containers will change surveillance bins. This includes 4 random samples originally thought to be in the Pressure & Corrosion bin that have already had DE performed. The binning and sample selection report [Reference 6] will be revised in FY11 to reflect these changes.

2. 3013 DE Surveillance Activities

The 3013 surveillance program completed all Data Set 1 activities for FY10. Data Set 1 activities are defined as those needed to demonstrate that there has been no impact to the integrity of the 3013 containers in the storage inventory. These activities include:

- Obtaining and transferring cans, gas samples, and oxide samples to SRNL from KIS
- Obtaining gas pressure measurement from 3013 cans in KIS
- Visual screening of 3013 cans with microscope
- Analyzing composition of gas samples
- Measuring particle density of oxide material
- Documentation of above data collected

Data Set 2 activities are the remainder of the analyses needed for complete technical evaluation for the program and to provide predictive capabilities for 3013 container lifetime. These include:

- Thermo-gravimetric analyzer (TGA)/mass spectrometer (MS) - measures moisture in the oxide
- Tapped density
- Carbon/sulfur analysis (C/S)
- Scanning Electron Microscopy (SEM) - inspects oxide at high magnification
- Energy Dispersive X-Ray Spectroscopy (EDX) - identifies elemental analysis of oxide
- Brunauer Emmet Teller (BET) analysis - determines specific surface area
- X-ray Diffraction (XRD) - analyzes compounds within oxide
- Ion Chromatography (IC) - measures anions in aqueous samples
- Inductively coupled plasma (ICP)/MS - measures plutonium isotopic composition
- ICP/Emission Spectroscopy (ES) - measures metallic impurities
- RadChem - measures radiochemical analyses of actinides in solution

All Data Set 2 activities for FY10 are completed. Additional corrosion analysis of one container (H003367) is continuing under the corrosion working group program. Additionally, the high moisture Hanford container was opened at LANL in FY10. An initial visual screening along with gas analysis and a subset of oxide powder measurements was completed at LANL. The empty containers and an oxide sample were sent to SRNL for analysis.

2.1 3013 DE Surveillance in KIS

The 3013 DE surveillance is performed in the K-Area Interim Surveillance (KIS) facility. A glovebox is setup with a can puncture device to sample the headspace gas inside the 3013, and a can cutter to open the 3013 to remove the oxide. A total of 18 DE surveillances were performed for FY10. No major findings that challenge the integrity of the 3013 container were noted. A summary is provided below for each process.

i. Can Puncture

Gas pressures and compositions of each inner 3013 container are provided in Table 5. Pressures are calculated using a software model to determine the pressure inside the 3013 prior to puncture [Reference 12]. In all cases, the pressures were less than 100 psig. The highest observed pressure was 17.6 psia. Since the container was packaged at Hanford, this equates to a pressure increase of 3.2 psig. In most cases, the pressure is less than the packaging pressure, since the oxygen has been consumed. While hydrogen has been generated in some cases, when hydrogen is present, no oxygen (< 0.1%) is present.

During DE-10 a valving error occurred after the inner can and convenience cans had been punctured. Instead of opening the valve to pull air out of the 3013, a valve was opened to allow glovebox air into the manifold. The time the CPD was exposed to atmosphere in the glovebox was 18 seconds. Unfortunately the purge cylinder was also not utilized when the inner gas sample was collected 40 minutes later. As expected, the oxygen level in the gas sample was high due to the glovebox air contribution. SRNL performed a calculation to determine the glovebox contribution in the gas sample. Two models were used (partial mixing and complete mixing) to give the bounding gas compositions. It is important to note that none of the 3013 containers that have undergone destructive evaluation since startup have had any significant level of oxygen while hydrogen is also present. Based on the processing history, prompt gamma results and moisture results of this 3013, the oxygen level is expected to be less than 0.1%, which is close to the lower bounding limit.

ii. Can Cutter

KIS utilizes a conventional pipe cutter to open the welded 3013 cans. The pipe cutter parts the metal with a rolling blade. During the second quarter of this fiscal year the can cutter guard was removed because it limited visibility and restricted working space inside the glovebox. A Hazard Risk Assessment was conducted to identify any hazards without the can cutter guard. The Assessment concluded that the removal of the guard would not constitute a violation of machine guarding requirements and would significantly reduce the likelihood of worker injury while performing necessary adjustments for cutting the 3013 containers.

iii. Oxide Inspection

Once the convenience can is removed from the 3013 package, a sample of the oxide is taken as soon as the convenience can is opened to determine the as-found moisture level in the 3013 package. Next the oxide is poured out in a pan for examination and additional sampling. A composite sample is taken from four quadrants of the pan. An additional sample is taken if there are any ‘items of interest’. The ‘items of interest’ analyzed to date include flakes of fire brick, boat corrosion products and other tramp materials due to impurities in the oxide (Table 6).

iv. Visual Can Inspection

After removing the oxide, the outer and inner cans are visually inspected for any corrosion or evidence of degradation caused by the storage of Pu oxide. In the majority of the cases, the cans appeared in excellent shape. It is not uncommon for the convenience can to have a coating either in the region of the oxide or more commonly in the head space region. In FY 10, DE 3, 4, 5, 8 and 15 had some staining or residue on the inside of the inner can also (Figure 3). No visible pitting or degradation, beside the staining or coating, was identified during the FY10 field surveillances.

Figure 3. Residue on DE #3 H003710 Inner Can

v. Packaging of Materials for SRNL

All of the samples taken during the DE process are sent to SRNL for additional examination. The convenience can, the inner and outer cut can pieces, and the gas sample vials filled during the CPD operation are packaged together in a LaCalhene 270 DPTE transfer container for shipment to SRNL. The DPTE container is filled with cut foam to protect the items during shipment. The oxide sample vials are heat sealed out in a plastic bag for transfer to SRNL in Croft Safdrums. Over 35 shipments from K-Area to SRNL were completed on time and without contamination incidents.

2.2 3013 DE Surveillance Activities in SRNL

SRNL receives the empty 3013 containers (outer, inner and convenience), gas samples from the outer/inner volume and the inner/convenience volume, and Pu oxide samples from the bulk material.

i. Can Inspections

The 3013 container system consists of nested welded 300 series stainless steel containers with the outer container credited to stay leak tight throughout a 50 year period of storage. In FY10 eighteen containers packaged at Rocky Flats Environmental Test Site (RFETS), Hanford, and SRS were examined destructively. Future examinations will include containers from Lawrence Livermore National Laboratory (LLNL) and Los Alamos National Laboratory (LANL). During destructive examination, the containers are punctured to collect gas samples, sectioned to collect Pu oxide samples, and the empty containers are metallurgically examined for damage. The results of the metallurgical examinations are provided in Table 7-8 .

Previously, the presence of pitting corrosion in the headspace region of certain 3013 inner containers as well as the presence of a dusting or coating on convenience and inner cans has been observed during destructive examination [Reference 10]. The postulated pitting mechanism requires the presence of a radiation source (alpha radiation from the plutonium material) to dissociate and ionize the gases present and form a more volatile vapor or gas containing chlorine. The chloride rich vapor or gas provided a mechanism to transport chloride to stainless steel surfaces exposed only to the headspace region and make that region susceptible to corrosion [Reference 13].

The degradation, if any, observed during destructive examination of convenience and inner containers could be correlated with the chemistry of the plutonium bearing materials stored in the convenience containers. The surveillance observations showed that none of the containers from the pressure bin displayed any evidence of corrosion on any of the surfaces. The majority of indications of incipient corrosion occurred in the headspace gas region of containers that stored plutonium bearing materials with high chloride and moisture contents. Little to no damage was observed in the plutonium oxide contact region of the convenience container. All of these observations are consistent with observations in laboratory testing of small containers [Reference 14]. Additionally, no evidence of stress corrosion cracking has been observed in any of the containers examined to date.

Perhaps the most significant observation overall was that a chloride rich gas was created under certain packaging and material conditions which provided a mechanism for chloride transport and deposition to regions of the container system that were only exposed to headspace gases. This observation emphasizes the importance of continued surveillances of the stored containers. The surveillance program will continue to evaluate containers to gain sufficient data to validate the 50 year container integrity criteria, as specified in the DOE-STD-3013. Gaseous transport of chloride and the potential for stress corrosion cracking is a strong focus of the evaluations as will pitting corrosion. The integrity of the containers is sound at this time and there is no reason to believe that the potential for a 50 year storage lifetime will be compromised, particularly given the robust nature of the 3013 container system.

ii. Gas Analysis

During destructive examination, headspace gas samples are obtained from the 3013 inner container and the annulus between the outer and inner containers. To characterize gas species, the samples are analyzed in SRNL by gas chromatography (GC), direct-inlet mass spectrometry (DIMS), and Fourier-transform infrared spectroscopy (FTIR). GC results, as well as other parameters, are utilized as input into the gas evaluation software tool (GEST) program for computation of pre-puncture gas compositions and pressures [Reference 12]. Gas composition and container pressure results obtained provide important data on the gas generation characteristics of plutonium-bearing material from actual 3013 storage inventory.

Several of the eighteen containers evaluated at SRS in FY10 contained appreciable H_2 content (some greater than 30 mol %), yet little or no oxygen was detected in any of the cans, including those exhibiting high H_2 concentrations. The maximum observed pressure was 2.9 psig (17.6 psia) which is well below the 699 psig design pressure for a 3013 outer container. Table 5 shows pressures observed within the inner containers during storage. No impact to the structural integrity of the 3013 containers was observed.

iii Oxide Analysis

Immediately after opening the container, an “initial moisture” (IM) sample is taken, sealed, and sent to SRNL. The packaging sites used several different techniques to demonstrate that the packaged material was less than the standard limit of 0.5 wt%. Many of the containers were analyzed by either Loss-On-Ignition (LOI) or Thermogravimetric Analysis (TGA). Both techniques are conservative in that they report all weight loss as water. The IM sample after DE is analyzed by TGA with Mass Spectrometry (TGA-MS), which better quantifies the actual amount of water that is present in the sample. Table 9 lists the moisture value reported by the packaging site and the IM value obtained during DE. Both values show that the moisture content is well below the 3013 Standard limit. In most cases the results reported by the packaging sites are consistent with the results from the IM sample. In three cases (DE15 – DE17) the IM results are significantly higher than the reported moisture values, but still below the Standard limit. These three containers are high salt materials that were packaged at RFETS where the sample was obtained and analyzed in dry air gloveboxes. At SRS, the DE samples are obtained and analyzed in standard air (room humidity) gloveboxes. It is likely that the samples adsorbed additional moisture prior to analysis.

Immediately after the moisture sample has been obtained, a special lid equipped with a humidity sensor and thermocouples is installed on selected convenience cans. Temperature and humidity data are collected, Table 10, and forwarded to MIS to assist in understanding the impact of humidity on corrosion. These data have proven invaluable for developing an understanding of the behavior of chloride bearing plutonium materials. A select number of future DE containers will require temperature and humidity measurements to provide additional data for understanding corrosion results.

After the container is opened, if large clumps are present, they are crushed to a point where a composite, representative sample can be obtained. The sample is shipped to SRNL for analysis. The bulk material properties (bulk density, tap density, particle density by pycnometry and specific surface area) are obtained using the entire sample. Particle density is used by the GEST model to calculate the pressure in the container prior to puncturing. The sample is then split and sent to other laboratories within SRNL to perform the remainder of the chemical characterization

As each suite of analyses was completed, the properties of, and any changes in, the Pu-bearing materials are used to interpret any package-sample interactions (corrosion) that may have occurred during storage, and could be used to deduce the chemical interactions that led to speciation of the headspace gas samples. Sample results for each container are included in Appendix A.

The characterization results are generally in agreement with the pre-surveillance data except for the two containers (H004000 and H004309) which had mismatched baseline PG as described in Section III.1.v. The predominant phases identified by X-ray diffraction are in agreement with the expected phase assemblages of the as-received materials. The measured densities are in reasonable agreement with the expected densities of materials containing the fraction of salts and actinide oxide specified by the pre-surveillance data. The radiochemical results are generally in good agreement with the pre-surveillance data for mixtures containing “weapons grade” Pu; however, the ICP-MS results from the present investigation generally produce lower concentrations of Pu than the pre-surveillance analyses. For mixtures containing “reactor grade” Pu, the ICP-MS results from the present investigation appear to be in better agreement with the pre-surveillance data than the radiochemistry results [See Appendix A].

3. 9975 NDE Surveillance

Five separate NDE techniques were performed on each of the 9975 shipping packages selected for field surveillance in FY10 (thirty-four 9975 packages). These techniques provided evidence to validate the KAMS DSA assumptions made on material and package performance over a 15 year storage period in KAMS, recently extended from the original 10 year storage period in KAMS. A summary of surveillance results is provided in Table 11 and a white paper providing a 15 year storage period justification is provided in Reference 15. Laboratory tests on selected 9975 material components are underway. Results from those laboratory tests are also summarized.

i. O-ring seal

A leak check is performed on the SCV and PCV O-ring seals prior to opening the containers in the K-Area Complex to provide evidence that the seal performance has not changed since the time of packaging. The leak check is conducted using the same 9975 post load leak check procedure, used at the time of packaging, which simultaneously tests the pressure boundary on both the upper and lower O-rings. A leak check of the PCV in 9975-06742 failed the test criteria of ≤ 1 E-3 cc/sec. The inner O-ring was determined to be leaking during the follow up bubble test. The subsequent inspection of the inner O-ring determined that a twist in the seal may have cause the leak (Figures 4 and 5). The O-rings were replaced and the shipping package passed the annual leak test.

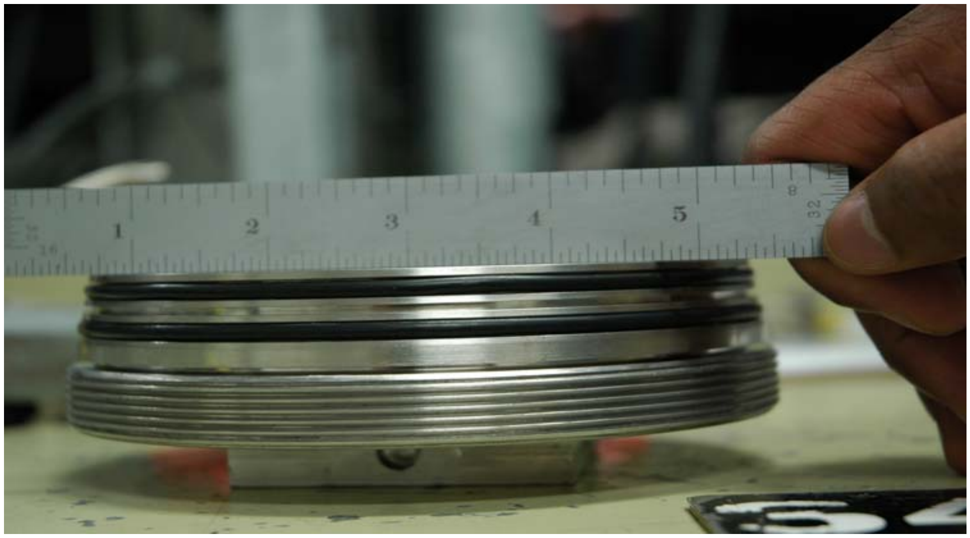

Figure 4. Cone Seal with twisted O-ring

Figure 5. O-ring does not lie flat due to deformation

O-ring dimensions are obtained in the field and again in the laboratory to monitor O-ring compression set over time. The results provide the evidence to show that there is no noticeable degradation in the 9975 O-ring seal performance due to shipping and storage operations in KAMS.

ii. Celotex® Fiberboard Density

The density of the fiberboard material inside the 9975 is determined by measuring the weight and volume of the upper subassembly. On all 34 9975 shipping packages evaluated, the weight and density was greater than the surveillance program minimum criteria of 24 lbs and 0.21 g/cc, respectively, which provides evidence that the density is above that assumed in the KAMS criticality analysis. This field result indicates that the 9975 packages are acceptable for continued service in KAMS as there is no significant change to the upper assembly density over the storage period.

iii. Celotex® Dimensions

Celotex® dimensions have been measured on the upper assembly and the lower assembly of all 34 9975 shipping packages selected for field surveillance in FY10. Only 1 package (NDE-22/DE-10) was found with an axial gap that exceeded the SARP limits. Other axial gap failures were identified during other routine activities i.e. verification measurements and during annual recertification. Verification of this axial dimension provides assurance that the Celotex® has not compressed to a greater extent than assumed in the KAMS DSA, and that a gap is not present between the upper and lower Celotex® subassemblies when assembled in a vertical orientation. In every case where the shipping package is found to exceed the SARP axial gap limit, the lead shielding is removed in order to remove the entire lower assembly from the shipping drum. In a few cases where the fiberboard has been removed for examination, the lower assembly was found to contain significantly more moisture at the bottom of the assembly. This saturated fiberboard loses its compressive strength and results in a greater than 1" axial gap inside the package. Laboratory studies have confirmed this result also. It is unclear why a small subset of the 9975 shipping packages which originated from RFETS has excessive moisture in the fiberboard. In FY10, a thermal analysis was completed to evaluate the worst case fiberboard found to date whether during a surveillance or during verification measurements, to ensure that the package can still be used for safe storage in KAMS. The analysis concluded that an axial gap up to 1.437" with an increase in moisture is safe for storage because it does not result in package component temperatures exceeding their design limit during normal conditions of storage or during a postulated facility fire event (1500 deg F for 86 min) [Reference 16]. The axial gap measurement provides confidence that significant impacts from excess moisture in the Celotex® have not occurred.

iv. Temperature Measurements

Temperature measurements have been obtained on all 34 9975 shipping packages selected for field surveillance in FY10. The difference in temperature between the interior of the lead shield and the ambient temperature is compared to a 50 degree limit. The difference in temperature between the exterior of the SCV and the ambient temperature is compared to a 65 degree F limit. All 9975 temperature measurements have been satisfactory. The temperature data provides evidence that the thermal performance of the Celotex® meets the assumptions made in the KAMS DSA.

v. Visual Inspection

A visual inspection is performed on the accessible portions of the 9975 package including all metal components, lead shielding, Celotex® and O-rings to ensure there are no signs of corrosion or other unexpected forms of degradation. The visual inspection identified lead carbonate hydroxide corrosion present on the surface of the lead shield assembly. The lead carbonate hydroxide corrosion is formed due to the presence of acetic acid within the Celotex® assembly. An experimental study investigated the formation of lead carbonate hydroxide and determined that the bounding rate of lead carbonate formation as 2 mils per year [Reference 17]. The resulting information provided the 9975 Design Authority the data necessary to ensure the Lead Shielding Body has a minimum service life of 20 years [Reference 18].

Based on the data obtained to date and projected performance of the 9975 packages in the KAMS facility, the 9975 packages are expected to maintain the minimum safety requirements for a period of 15 years in KAMS storage. This justification is established with the stipulation that surveillance activities will continue throughout this extended time to ensure the continued integrity of the 9975 materials of construction and to further understand the currently identified degradation mechanisms [Reference 15].

4. 9975 Destructive Examination

The surveillance program requires a destructive examination (DE) on one 9975 package from KAMS each year to provide a detailed assessment of the 9975 components. The scope of DE includes a thorough visual inspection of all components in the 9975 package as well as more detailed analyses of the Celotex®, O-rings, and lead shield. The results of the FY10 DE are reported in Reference 19. Measurements are taken on the upper and lower Celotex® subassembly to determine how the density, moisture content, dimensional, thermal and mechanical properties compare to baseline values and those obtained in the field. For those attributes that were measured during the field surveillance, no significant changes were observed. However, there were two conditions noted that were outside the inspection criteria as shown in the Table 12. A white layer of corrosion was observed on the lead shielding for the package, consistent with the NDE field inspection data. This phenomenon has been evaluated and determined acceptable for storage and shipping of the packages for up to 20 years [Reference 18]. Although the thermal conductivity in the axial direction exceeded the specified range for the package, this is not considered a safety concern for KAMS because the thermal conductivity test in the radial direction, which is the dominant heat transfer path, was well within the baseline values. There is no sign of corrosion (other than lead carbonate) acting on the metal surfaces, or significant change from O-ring or Celotex® material property baseline conditions. The 9975 package currently meets the material property and performance assumptions of the KAMS DSA.

5. SRNL 3013 Corrosion Project

The integrated corrosion program consists of shelf-life program experiments, both small-scale and full-scale, that provide an early response to any gas generation or corrosion concerns that might be observed in the field. These tests also help guide the selection of engineering judgment surveillance items for FY10 [Reference 20-21]. Since the shelf-life program tests focus on bounding conditions, they can produce effects such as gas generation or corrosion that are more severe and more rapid than would occur in packaged 3013 containers. As expected, field surveillance destructive examinations (DE) and non-destructive examinations (NDE) to date have shown that the pressure and corrosion inside the 3013 containers are significantly less severe than observed in the shelf-life tests. Stress corrosion cracking (SCC) was observed during small-scale 3013 corrosion tests in FY08. The SCC was observed in replicate stainless steel 304L (SS 304L) test specimens for a room temperature plutonium oxide-salt composition with bounding (0.5 wt %) water content [Reference 22]. These results have raised a concern because they show that SCC can occur under conditions allowed by DOE-STD-3013. Based on these results, the Materials Identification and Surveillance (MIS) Working Group could not ensure that SCC events were absent in 3013 packages. The integrated corrosion test plan was developed under the direction of the MIS Working Group [Reference 23] and addresses the requirements defined in the letter from the MIS Corrosion Working Group titled "Impact of stress corrosion cracking observed in shelf-life specimens for 3013 containers" [Reference 24]. These recommendations specified that a test plan be developed and implemented to address the conditions under which SCC occurs with respect to the parameters that were controlled during stabilization and packaging of 3013 containers. The major activities included:

- Determine the influence of temperature, salt composition, and moisture uptake during packaging on the resulting Relative Humidity (RH) in the packaged container.
- Determine the threshold RH of various plutonium oxide-salt mixtures for SCC to occur in direct contact and headspace exposure;
- Establish the residual stress or weld chemistry conditions for SCC;
- Evaluate the reduction of the RH over time within a sealed 3013 package including water consumption mechanisms and redistribution of the moisture within the material.

The goal of the integrated corrosion program is to resolve the stress corrosion cracking issues within 3013 storage containers. Results from tests identified in this plan will define the conditions under which stress corrosion cracking occurs with respect to the parameters that were controlled during stabilization and packaging of plutonium-bearing materials for storage in 3013 containers. The identified conditions of concern will be used to evaluate the 3013 storage inventory in conjunction with the Integrated Surveillance Program (ISP) Database to provide refined groupings of the pressure / corrosion bin. In the event that conditions are present for SCC, a more rigorous approach for the ISP would be adopted for a subset of the inventory that warrants closer scrutiny.

Although SCC has been observed in laboratory conditions, it is the technical judgment of the MIS Working Group and the MIS Corrosion Working Group that the robust design of the 3013 container system will prevent degradation of the outer 3013 containers in the existing storage inventory [Reference 25]. In the meantime the integrated corrosion program testing will proceed to validate this technical judgment.

6. ISP Database

Configuration of the 3013 ISP database for the DOE complex is managed by SRS. A configuration control plan was implemented to ensure changes to the database are tracked and documented. Fourteen modifications were requested, documented, and implemented. In conjunction with LANL, 31 queries were performed resulting in pertinent data needed for the program. Collaboration with LANL resulted in the development of a report, to be finalized in FY11, that outlines the rebinning and sample selection efforts for the packaged 3013 containers. Additionally, a Data Dictionary was developed to provide definitions of all the data fields in the ISP Field Surveillance Module (FSM) [Reference 26].

A re-baseline of the ISP database was completed and a copy was transmitted to LANL. Additionally, a copy of the FSM which contains all the data from NDE and DE surveillances to date, was provided to LANL. Surveillance data from both NDE and DE activities in KAC have been input to the 3013 FSM through FY10 while surveillance data for 3013 DE activities in SRNL is underway.

Site organizations evaluating disposition of materials also routinely use the database to evaluate the disposition paths of the currently packaged 3013 containers.

7. 9975 Laboratory Testing

The 9975 surveillance program requires material testing at bounding storage conditions in KAMS. The purpose of the tests is to obtain data to support the development of a 9975 life prediction model. This test program requires several years worth of data collection at bounding humidity, temperature, and radiation conditions in order to “catch up” to and predict the service life of 9975 packages in KAMS. The surveillance program objective is to determine the need for repackaging the 9975’s in KAMS or provide evidence for continued safe storage without repackaging. The scope of the SRNL 9975 test program is documented in [Reference 27].

<u>Cane Fiberboard</u>

Cane fiberboard material, used in the 9975 shipping package, has been tested for thermal, mechanical and physical properties following environmental conditioning for periods up to 5 years. The aging environments have included both representative conditions and bounding KAMS storage conditions, consisting of elevated temperature, up to and including 250°F (the maximum allowed service temperature for fiberboard in 9975 packages) and elevated humidity. These data show changes of some properties at bounding conditions [References 28, 29]. Development of a model to predict service life in KAMS is underway.

Analysis of data for cane fiberboard material collected in the laboratory and during field surveillance indicates that additional pertinent data needs to be collected and analyzed to fully understand the relative service life of this material in the KAMS facility. For example, the ambient conditions within KAMS can be reasonably identified and the temperature profiles within the various packages (with a range of heat loads and at varying locations within an array of packages) can be calculated. However, the humidity within the package is not well characterized. While the outer drum does not provide an air-tight seal, it does greatly restrict the gain or loss of moisture in the fiberboard. Preliminary efforts have identified a relationship between the moisture content of fiberboard samples and the relative humidity of the surrounding air [Reference 29], but further work is needed to demonstrate whether this relationship holds within an internally heated 9975 package. In FY10, field surveillance of the 9975 container added a measurement of the humidity and moisture content of the fiberboard of 9975 packages in the storage configuration. Results indicate a range of moisture content within packages and between different packages. Measured values range from ~6 to 18 %WME (wood moisture equivalent), with variation within the accessible portions of a given package varying by ~2 to 8 %WME. The higher moisture gradients within a package would be expected with the higher internal heat loads. Humidity measurements have been less useful due to the practice of temporarily relocating the packages shortly before field surveillance activities. Because of a different ambient temperature in the two locations, the humidity within the package is changed and does not have sufficient time to return to a condition in equilibrium with the fiberboard.

Softwood Fiberboard

Cane fiberboard, the material specified for thermal insulation and impact resistance in 9975 shipping packages is no longer available because the production factory was closed. In 2008 the softwood fiberboard, manufactured by Knight-Celotex® was approved as an acceptable substitute for transportation [Reference 30]. Data in the literature showed a consistent trend in thermal properties of fiberboard as a function of temperature, density and/or moisture content regardless of material source. With no obvious overall variation among all the material sources, it was reasonable to conclude that the thermal properties of un-aged cane and softwood fiberboard were comparable. This conclusion was further supported by the similarity of the SRNL thermal data for the two materials and the material was deemed equivalent to the cane fiberboard [Reference 31].

The literature data also suggested a similarity among fiberboard from the major material sources for properties relating to tensile and shear strength. However, no relevant data were found for the mechanical properties of interest for long term storage of 9975 packages in KAMS – compression strength and energy absorption. Limited SRNL data comparing un-aged cane and softwood fiberboard showed the softwood fiberboard has lower average and minimum values in these two areas. However, the available margin in compression strength exceeded the measured differences. Results from environmental conditioning and testing indicate that similar aging trends are observed for softwood and cane fiberboard samples, with a few differences. On the positive side, the softwood fiberboard data to date shows less sample-to-sample variation in physical properties than cane fiberboard, and the thermal conductivity decreases at a slower rate at 250°F for softwood fiberboard than for cane fiberboard. On the other hand, the softwood fiberboard physical property samples generally show degradation rates greater than cane fiberboard samples in the 185°F 30%RH environment [Reference 32]. Testing following additional conditioning will continue and the addition of samples in other elevated humidity environment(s) will be pursued to identify the extent of these trends and validate the continued acceptability of aged softwood fiberboard to meet KAMS storage requirements [Reference 33]. Post-conditioning data have been measured on samples from a single softwood fiberboard assembly, and baseline data are also available from a limited number of vendor-provided samples. This provides minimal information on the possible sample-to-sample variation exhibited by softwood fiberboard. Data to date are generally consistent with the range seen in cane fiberboard, but some portions of the data trends are skewed toward the lower end of that range. Further understanding of the variability of softwood fiberboard properties will require testing of additional material.

<u>GLT O-Rings</u>

A series of experiments to monitor the performance of Viton® GLT O-rings used in the 9975 package under simulated KAMS conditions has been ongoing at the Savannah River National Laboratory for five years. Sixty-two mock-ups of 9975 Primary Containment Vessels (PCVs) were assembled and exposed to nominal and bounding conditions expected in the KAMS facility, including conditions of temperature, irradiation, and internal gas atmosphere. They were leak-tested initially and have been tested every six months thereafter to determine if they meet the criterion of leak-tightness outlined in ANSI standard N14.5-97 [Reference 34]. To date 12 O-rings from 7 of the original fixtures aging at 300ºF have failed to pass the leak test requirement at room temperature following up to 4.5 years exposure at temperature. All of the fixtures aging at 200F continue to pass the leak test requirement at room temperature. There are 31 of the original 62 remaining in the testing configuration [References 35] with 26 having been removed for reasons other than room temperature leak test failure. An additional five mock-up fixtures were more recently assembled and exposed to higher temperatures (350 - 450°F) in an attempt to produce failures within a reasonable timeframe to improve the predictive capability of the leak tests. Nine of the ten O-rings in these higher temperature fixtures have failed to remain leaktight. Predictive models (below) developed using compression stress relaxation tests will attempt to quantify the expected equivalent storage lifetime at actual storage conditions.

Accelerated aging work has been underway for ~5 years. O-rings of the V0835-75 compound are being aged at temperatures of 175 - 350°F and periodically tested for compression stress-relaxation (CSR) behavior. CSR is an industry standard method for evaluating seal performance. Target levels of compression stress relaxation (90% and near-100%) have been reached at an aging temperature of 350 °F after ~2800 and 3500 hours, respectively. These data are comparable to values predicted from short-term baseline CSR tests [Reference 36]. Stress relaxation levels are less severe at lower test temperatures. The 350°F temperature is above normal conditions expected during shipping or storage in KAMS and it is above the design limit for the containment vessels (300°F). However, it is below the continuous service temperature of the O-rings (400°F) as provided by the vendor and is not considered an excessive temperature for aging purposes. These levels of stress-relaxation or retained sealing force do not inherently correlate to seal failure as defined by leak rate. The minimum sealing force required to maintain a leaktight seal in the 9975 design is not a measured value, though preliminary efforts suggest that the value is likely minimal, possibly as low as 1 N/cm, particularly for very static conditions. In absence of such data, stress-relaxation or retained sealing force values are often used to define mechanical lifetime in elastomer aging studies. CSR tests will continue until target values are reached at all temperatures [Reference 37].

A preliminary life prediction model has been developed for the O-rings in KAMS. The conservative model is based primarily on long-term compression stress relaxation (CSR) experiments and Arrhenius accelerated-aging methodology. For model development purposes, seal lifetime is defined as a 90% loss of measurable sealing force. Using time-temperature superposition principles, the conservative model predicts a service life of approximately 25 years at a constant seal temperature of 175 °F. This represents a maximum payload (19W) package at a constant ambient temperature of 106 °F, the highest recorded in KAMS to date. This is considered a highly conservative value as such ambient temperatures are only reached on occasion and for short durations. The presence of fiberboard in the package minimizes the impact of such temperature swings, with several days required for seal temperatures to respond proportionately based on full-size instrumented package experiments.

At 85°F ambient, a more realistic but still conservative value, bounding seal temperatures are reduced to ~158°F, with an estimated seal lifetime of ~35 - 45 years. These lifetimes are still conservative, as the average ambient temperature is less than 85 °F and the time at which the O-ring temperatures are below 100°F (which depends on actual payload) is likely to have far less impact on elastomer seal aging. For a lower ambient average of 70 °F, bounding seal temperatures of approximately 140 °F are estimated, increasing service life based on CSR to approximately 60-80 years. The actual service life for O-rings in a maximum wattage package likely lies higher than the estimates due to the conservative assumptions used for the model. For lower heat loads at similar ambient temperatures, seal lifetime is further increased.

The preliminary model is based on several assumptions that require validation with additional experiments and longer exposures at more realistic conditions. The assumption of constant exposure at peak temperature is believed to be conservative. Cumulative damage at more realistic conditions will likely be less severe but is more difficult to assess based on available data. Arrhenius aging behavior is expected, but non-Arrhenius behavior is possible. Validation of Arrhenius behavior is ideally determined from longer tests at temperatures closer to actual service conditions. CSR experiments will therefore continue at lower temperatures to validate the model. Ultrasensitive oxygen consumption analysis has been shown to be useful in identifying non-Arrhenius behavior within reasonable test periods [Reference 38]. Early baseline oxygen consumption analysis results have been obtained, with consumption rates of approximately 1E-10 to 4.4E-13 mol O_2/g determined.

Additional work is in progress to validate the technique and to ensure repeatable and reliable results.

GLT-S O-Rings

In 2008, DuPont Performance Elastomers ceased production of Viton® GLT, the base polymer for the V035-75 O-ring compound. Viton® GLT-S had been developed several years earlier, but dual production of both polymer types was no longer deemed viable by the polymer manufacturer. As a result, Parker Seals transitioned many customers, including SRS, to the Viton® GLT-S based VM835-75 compound. The new compound was included in the 9975 shipping package SARP and baseline testing was performed to determine suitability for interim storage in KAMS, similar to work previously done for GLT O-rings [Reference 39]. The baseline characterization work indicates that the new GLT-S compound is comparable to the GLT material, with some minor variations noted [Reference 40]. Based on these results and anticipated storage conditions, GLT-S O-rings were judged acceptable for the interim (10 year) storage period in KAMS. A longer-term scope of activities to better characterize the aging behavior of the GLT-S compound was developed and is underway [Reference 41]. The testing is commensurate with that performed for the GLT O-ring compound. A summary of alternative materials characterization and performance data is provided in Reference 42.

8. 9975 Database Development

Much like the 3013 ISP database, unclassified shipping container surveillance modules (SCSM) were developed for the 9975 surveillance data. Two modifications were requested and implemented. The data obtained from NDE field surveillance, supplemental data from NDE surveillance performed in the laboratory and supporting laboratory testing for longer term properties evaluations have all been incorporated into a database for the 9975 shipping/storage package. Additionally, data from the 9975 destructive evaluation is captured in the 9975 SCSM .

IV. SUMMARY

3013 Surveillance

Surveillance for the initial 3013 container random sampling of the Innocuous bin and the Pressure bin has been completed and there has been no indication of corrosion or significant pressurization. The maximum pressure observed was less than 50 psig, which is well below the design pressure of 699 psig for the 3013 container [Reference 3]. The data collected during surveillance of these bins has been evaluated by the Materials Identification and Surveillance (MIS) Working Group and no additional surveillance is necessary for these bins at least through FY13. A decision will be made whether additional surveillance of these bins is needed during future years of storage and as additional containers are generated. Based on the data collected to date, the SPA concludes that 3013 containers in these bins can continue to be safely stored in KAMS.

In FY10, 13 destructive examinations (DE) were performed on random samples from the Pressure & Corrosion bin. To date, DE has been completed for approximately 30% of the random samples from the Pressure & Corrosion bin. In addition, DE has been performed on 6 engineering judgment (EJ) containers, for a total of 17 to date. This includes one container that exceeded the 3013 Standard moisture limit which was opened at LANL. The container pieces and an oxide sample were sent to SRNL for examination in FY11. No significant pressurization has been observed for the Pressure & Corrosion bin containers. Relatively minor corrosion has been observed on some convenience containers and the inside of two inner containers. While the limited extent of corrosion does not jeopardize the integrity of the outer 3013 containers, it does highlight the importance of continuing to perform DE and the Shelf Life program to assure that the corrosion rate is not accelerating or changing to a different corrosion mechanism (e.g., stress corrosion cracking). Statistical sampling is currently scheduled to be completed in FY17, but there is a proposed reduction of the number of DE's per year for FY11 and beyond which may delay the completion date.

Additional 3013 Activities

Stress corrosion cracking (SCC) has been observed during small-scale 3013 corrosion tests. The SCC was observed in replicate stainless steel 304L (SS 304L) test specimens for one plutonium oxide-salt composition with bounding (slightly greater than 0.5 wt.%) water content. These results have raised a concern because they show that SCC can occur under conditions allowed by DOE-STD-3013. As a result, the 3013 corrosion working group developed an integrated corrosion plan that provides a scope of work to define the conditions under which stress corrosion cracking occurs with respect to the parameters that were controlled during stabilization and packaging of 3013 containers. The potential for SCC is seen as the most deleterious form of corrosion expected within the 3013 containers which explains the emphasis within the corrosion plan.

SRS continues to manage the ISP database and has implemented a configuration control program in compliance with site requirements. There have been 38 change requests implemented and 73 queries run for data mining. Additionally, the data dictionary has been updated to reflect the fields represented in the database.

9975 Surveillance

In FY10, SRS completed nondestructive examination (NDE) on 34 9975 packages. As in the past, multiple 9975 containers confirmed the presence of lead carbonate hydroxide formation on the lead shield and the lead shielding body was determined to have a minimum service life of 20 years and is therefore considered to have a sufficient lifetime for storage. One package was found with an axial gap that exceeded the SARP limits, however, other axial gap failures were identified during other routine activities. An analysis was completed in FY10 to evaluate the worst case fiberboard found to date to ensure the 9975 packages can still be used for safe storage in KAMS. The analysis concluded that an axial gap up to 1.437" with an increase in moisture is safe for storage. The studies also determined that the axial gap measurement provides confidence that significant impacts from excess moisture in the fiberboard have not occurred.

Compression set for the 9975 O-rings in the field has been compared with the compression set measured in the laboratory and the performance of the O-rings in the field packages is acceptable. Long term age acceleration testing for O-rings and Celotex® in the laboratory are also ongoing. Additionally, the destructive examinations (DE) of one 9975 packages were completed.

The compilation of field and laboratory data currently indicates that the functional performance of the 9975 package materials meet all DSA storage requirements and assumptions and there is no evidence to suggest a need for repackaging within a 15 year storage period. The surveillance program will develop 9975 life prediction models over the next few years to provide a basis for continued storage or need for repackaging. Additionally, an ongoing investigation into recent axial gap findings will be finalized and recommendations regarding 9975 package life in KAMS will be assembled.

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VI. APPENDICES

Table 1: 3013 Statistical Sample Sizes

Bin	**# NDE**	**# DE**
Innocuous	10	0
Pressure	130	6
Pressure and Corrosion	128	128

Table 2: 3013 Surveillances Performed By Fiscal Year and Site

Fiscal Year	**Innocuous**	**Pressure**		**Pressure and Corrosion**	**Engineering Judgment**
	NDE	**NDE**	**NDE/DE**	**NDE/DE**	**NDE/DE**
2005	3 (1)[a]	11 (14)[a]		5 (5) – NDE Only	8 (5) – NDE Only
2006	0 (1)[a]	14 (13)[a]		3 (5) – NDE Only	3 (6) – NDE Only
2007	2 (1)[a]	12 (13)[a]	2	3	2
2008	1	24	3	12	2
2009	2	24	1	11	7
2010	2[b]	8[b]	0	13 NDE/DE 6 NDE Only[b]	6[c]

[a] Numbers in parentheses indicate additional surveillances performed at Hanford and/or LLNL
[b] NDEs performed to inspect for foreign material
[c] One container was opened at LANL

Table 3: SPA Evaluations for FY10

Eval. No.	DE Run No.	Description
SRNS-SRV-2010-015-9975-E1	11-05	Excessive corrosion of the lead shielding may diminish the wall thickness of the shield container. However in this case the shielding still met the dimensional requirements as prescribed in the 9975 SARP. In addition, the thickness of the corrosion layer was measured to ensure that the bounding rate of 2mils/yr was not exceeded. Report SRNL-L4400-2010-00001 (See report for full details) documented that this package experienced a corrosion rate of only 1mil/yr.
SRNS-SRV-2010-016-9975-E1	11-02	Excessive corrosion of the lead shielding may diminish the wall thickness of the shield container. However in this case the shielding still met the dimensional requirements as prescribed in the 9975 SARP. In addition, the thickness of the corrosion layer was measured to ensure that the bounding rate of 2mils/yr was not exceeded. Report SRNL-L4400-2010-00001 (See report for full details) documented that this package experienced a corrosion rate of only 1mil/yr.
SRNS-SRV-2011-011-9975-E1	11-11	During the performance of SOP-CSS-206-K, the SCV failed the post load leak test. The calculated leak rate was 0.003 cc/sec. Per SOP-CSS-207-K, the acceptance criterion for this measurement <0.001 cc/sec. When the SCV cone seal was removed, lint was observed on the inner O-ring in two places. No problems were observed with the outer O-ring.

Table 4: Pressure Results for FY10 Surveillances

FY10 RUN	3013 ID	Surv. ID (SRNS-SRV-)	BIN	Surveillance Reason	ISP Predicted P (psia)	Lid Deflection Pressure (psig)	GEST Pressure (psia)
NDE 1/ DE1	H004251	2009-035	Pressure & Corrosion	Judgmental	22.5	-6.6 ± 9.3	17.5 ± 0.5
NDE 2	H001157	2009-037	Pressure	Random	21.0	-2.0 ± 9.8	n/a
NDE 3	H001316	2009-038	Pressure	Random	21.1	-12.4 ± 8.7	n/a
NDE 4/ DE 2	H002496	2009-036	Pressure & Corrosion	Judgmental	22.0	-16.8 ± 8.3	11.6 ± 0.4
NDE 5	H004000	2009-039	Pressure & Corrosion	Random	21.6	1.1 ± 10.1	n/a
NDE 6 / DE 3	H003710	2009-040	Pressure & Corrosion	Random	22.5	7.3 ± 10.7	16.6 ± 0.4
NDE 7	H004331	2009-041	Pressure	Judgmental	21.0	-13.0 ± 8.7	n/a
NDE 8	H004322	2009-042	Pressure	Judgmental	21.0	-17.8 ± 8.2	n/a
NDE 9 / DE 4	H003655	2009-045	Pressure & Corrosion	Judgmental	22.8	0.7 ± 10.1	17.6 ± 0.5
NDE 10 / DE 5	H002447	2009-046	Pressure & Corrosion	Random	21.4	5.6 ± 10.6	12.0 ± 0.4
NDE 11	H001281	2009-043	Innocuous	Judgmental	21.0	-21.7 ± 7.8	n/a
NDE 12	H001401	2009-044	Pressure & Corrosion	Judgmental	21.0	-13.6 ± 8.6	n/a
NDE 13	H002686	2009-047	Pressure & Corrosion	Judgmental	21.6	10.6 ± 11.1	n/a
NDE 14	H003070	2009-048	Pressure	Judgmental	21.1	-6.5 ± 9.3	n/a
NDE 15	H003689	2009-049	Pressure & Corrosion	Judgmental	22.0	-5.3 ± 9.5	n/a
NDE 16 / DE 6	R610627	2009-055	Pressure & Corrosion	Random	18.5	4.6 ± 7.5	14.7 ± 0.6
NDE 17	H004294	2009-051	Pressure	Judgmental	21.1	-5.9 ± 9.4	n/a
NDE 18	H004309	2009-052	Pressure & Corrosion	Judgmental	21.0	-7.8 ± 9.2	n/a
NDE 19 / DE 7	H003900	2009-053	Pressure & Corrosion	Judgmental	22.6	-6.2 ± 9.4	17.0 ± 0.5
NDE 20 / DE 8	H003650	2009-054	Pressure & Corrosion	Judgmental	22.4	-6.5 ± 9.3	12.8 ± 0.4
NDE 21 / DE 9	H002567	2009-050	Pressure & Corrosion	Random	21.2	2.7 ± 10.3	11.7 ± 0.4

Table 4: Pressure Results for FY10 Surveillances (Continued)

FY10 RUN	3013 ID	Surv. ID (SRNS-SRV-)	BIN	Surveillance Reason	ISP Predicted P (psia)	Lid Deflection Pressure (psig)	GEST Pressure (psia)
NDE 22 / DE 10	H002728	2010-005	Pressure & Corrosion	Random	22.1	1.0 ± 10.1	11.4 ± 0.4
NDE 23	H004605	2010-001	Innocuous	Judgmental	21.0	126.8 ± 14.7	n/a
NDE 24	H003947	2010-002	Pressure	Judgmental	21.1	-24.1 ± 7.6	n/a
NDE 25	H003413	2010-003	Pressure & Corrosion	Judgmental	21.6	-28.6 ± 7.1	n/a
NDE 26	H002631	2010-004	Pressure & Corrosion	Judgmental	22.0	7.6 ± 10.8	n/a
NDE 27 / DE 11	H002786	2010-006	Pressure & Corrosion	Random	22.3	-84.3 ± 1.5	12.5 ± 0.4
NDE 28 / DE 12	H003077	2010-007	Pressure & Corrosion	Random	21.5	9.0 ± 10.9	12.2 ± 0.4
NDE 29 / DE 13	H003367	2010-008	Pressure & Corrosion	Random	22.0	-19.3 ± 8.0	11.5 ± 0.4
NDE 30 / DE 14	H003704	2010-009	Pressure & Corrosion	Random	22.3	3.1 ± 10.3	15.3 ± 0.4
NDE 31 / DE 15	R610785	2010-010	Pressure & Corrosion	Random	18.4	6.0 ± 7.5	13.9 ± 0.5
NDE 32 / DE 16	R610826	2010-011	Pressure & Corrosion	Random	18.4	7.0 ± 7.5	12.0 ± 0.5
NDE 33 / DE 17	R610853	2010-012	Pressure & Corrosion	Random	18.5	4.5 ± 7.5	12.3 ± 0.5
NDE 34 / DE 18	S001721	2010-013	Pressure & Corrosion	Random	22.6	16.1 ± 10.5	12.0 ± 0.4

Table 5: FY10 Gas Results

FY10 RUN	3013 ID	Surv. ID (SRNS-SRV-)	BIN	Gases (%)								GEST 3013 P (psia)
				CH_4	CO_2	N_2O	He	H_2	O_2	N_2	CO	
NDE 1/ DE1	H004251	2009-035	Pressure & Corrosion	0.2	2.5	<0.1	53.4	27.4	<0.1	16.2	0.4	17.5 ± 0.5
NDE 4/ DE 2	H002496	2009-036	Pressure & Corrosion	ND	ND	<0.1	54.3	1.3	<0.1	44.4	ND	11.6 ± 0.4
NDE 6 / DE 3	H003710	2009-040	Pressure & Corrosion	<0.1	0.4	<0.1	39.8	25.7	<0.1	34.0	<0.1	16.6 ± 04
NDE 9 / DE 4	H003655	2009-045	Pressure & Corrosion	<0.1	0.3	<0.1	43.0	27.4	<0.1	29.3	<0.1	17.6 ± 0.5
NDE 10 / DE 5	H002447	2009-046	Pressure & Corrosion	ND	ND	0.3	52.0	<0.1	0.1	47.6	<0.1	12.0 ± 0.4
NDE 16 / DE 6	R610627	2009-055	Pressure & Corrosion	ND	<0.1	ND	85.1	Trace	0.1	14.8	<0.1	14.7 ± 0.6
NDE 19 / DE 7	H003900	2009-053	Pressure & Corrosion	0.1	0.2	0.0	44.1	30.1	0.0	25.5	0.0	17.0 ± 0.5
NDE 20 / DE 8	H003650	2009-054	Pressure & Corrosion	ND	ND	<0.1	52.2	16.9	<0.1	30.9	ND	12.8 ± 0.4
NDE 21 / DE 9	H002567	2009-050	Pressure & Corrosion	ND	ND	<0.1	57.88	<0.1	<0.1	42.07	ND	11.7 ± 0.4
NDE 22 / DE 10	H002728	2010-005	Pressure & Corrosion	ND	<0.1	<0.1	48.89 to 54.01	13.31 to 14.71	0.0 to 2.03	31.32 to 35.74	ND	11.4 ± 0.4
NDE 27 / DE 11	H002786	2010-006	Pressure & Corrosion	ND	<0.10	<0.10	51.74	10.53	<0.10	37.65	<0.10	12.5 ± 0.4
NDE 28 / DE 12	H003077	2010-007	Pressure & Corrosion	ND	ND	0.97	52.28	<0.1	<0.1	46.73	ND	12.2 ± 0.4
NDE 29 / DE 13	H003367	2010-008	Pressure & Corrosion	ND	<0.1	<0.1	61.2	0.78	<0.1	37.97	<0.1	11.5 ± 0.4

Table 5: FY10 Gas Results (Continued)

FY10 RUN	3013 ID	Surv. ID (SRNS-SRV-)	BIN	Gases (%)								GEST 3013 P (psia)
				CH_4	CO_2	N_2O	He	H_2	O_2	N_2	CO	
NDE 30 / DE 14	H003704	2010-009	Pressure & Corrosion	<0.1	<0.1	<0.1	41.3	22.5	<0.1	36.0	<0.1	15.3 ± 0.4
NDE 31 / DE 15	R610785	2010-010	Pressure & Corrosion	ND	Trace	ND	84.8	0.7	<0.1	14.5	<0.1	13.9 ± 0.5
NDE 32 / DE 16	R610826	2010-011	Pressure & Corrosion	ND	<0.1	ND	88.5	ND	<0.1	11.5	ND	12.0 ± 0.5
NDE 33 / DE 17	R610853	2010-012	Pressure & Corrosion	ND	Trace	ND	86.8	Trace	<0.1	13.2	ND	12.3 ± 0.5
NDE 34 / DE 18	S001721	2010-013	Pressure & Corrosion	ND	Trace	0.1	73.6	Trace	<0.1	26.2	ND	12.0 ± 0.4

Table 6: Items of Interest

FY10 DE Run	3013 ID	Sample of Interest	SRNL Findings	Comments
1	H004251	Oxide was dark brown with small chunks. The material was dry and uniform in appearance except for a few lighter color particles.	Spot 1 – Cl, K, O Spot 2 – Mg, O, Cl, Pu, K Spot 3 – Pu, O, Mg, Si, Cl Spot 4 – Mg, O, Cl, Pu Spot 5 – Cl, Na, Pu, O Spot 6 – Ni, O, Cl, Cr, Fe	This material was packaged at Hanford, but originated at RFETS. The MIS representation is PyroOx-HN-RF-ERScrap. These impurities are common in this type of material.
2	H002496	Oxide was dark brown with sizes ranging from fine powder to small granular. The material appeared to be dry, homogeneous with a few small pieces of dissimilar material that were broken up easily.	Inspected with optical microscope. After review it was decided not to perform SEM/EDX.	This material is Hanford material type 41 (PFP Lab Oxide). The MIS representation is ScrapOx-HN-Lo.

Table 6: Items of Interest (Continued)

FY10 DE Run	3013 ID	Sample of Interest	SRNL Findings	Comments
9	H002567	Oxide was dark brown, dry and medium granular. Larger chunks were broken up. Sample of interest was taken.	**IR1:** Spot 1 – Si, O Spot 2 – Pu, O, Si, Al, Na Spot 3 – Al, Si, K, O, Na, Fe Spot 4 – Al, Si, K, O, Na, Fe **IR2:** Spot 1 – Fe, O, Mg, Si, Al, Pu, Fe, Zn, Cu, Cr, Ni, Y, Ti Spot 2 – Mg, O, Cu Spot 3 – Ta, Na, O, Pu, Ti, Fe, Zr Spot 4 – Mg, O, Cr, Ni, Mo, Fe, Si, Pu, Cr Spot 5 – Pu, O, Mg, Si Spot 6 – Al, Mg, O, Cu, Pu, Cr, Fe Spot 7 – Cr, O, Mg, Cr, Ni, Fe Spot 8 – Mg, O, Ta, Y, V, Pu, Y, Ta Spot 9 – Cr, O, Mg, Al, Mb, Si, Pu Ni, Fe Spot 10 – Mo, Ni, O, Mg, Si, Al, Pu,	This material was packaged at Hanford, but originated at RFETS. The MIS representation is PyroOx-HN-RF-MiscOx. These impurities are not unusual in this type of material.

Table 6: Items of Interest (Continued)

FY10 DE Run	3013 ID	Sample of Interest	SRNL Findings	Comments
13	H003367	Oxide was medium granular and brown with some lighter pieces.	**IR1:** Spot 1 – Pu, O, W, Si Spot 2 – Ca, F, O Spot 3 – Ni, Cl, Fe, Ca, Cr, Pu, O, Si Spot 4 – Cl, Na Spot 5 – Cl, K, O Spot 6 – Mo, Ca, O, K Spot 7 – Mg, Ni, Cl, O, Pu, Ca, Na, Cu, Fe Spot 8 – Ca, F, K Spot 9 – Cr, O, Ni, Cr, Fe, Pu, Si **IR2:** Spot 1 – Ca, Pu, F Spot 2 – Mg, O, Ni, Pu, Ca, Fe, Ni, Cr, S, Mo, Si Spot 3 – Pu, O, Si, Fe, Ni, Cr Spot 4 – Mg, Ca, O, Ni, W, Pu, Fe, Cl, W, Cr Spot 5 – Cl, K, Fe, Cr Spot 6 – Mg, O, Ni, Pu, Cu, Fe Spot 7 – Ca, Si, O, K, Fe, Cl, Cr Spot 8 – Mg, O, Ni, Pu, Cu, Ni	This material was packaged at Hanford, but originated at RFETS. The MIS representation is PyroOx-HN-RF-MiscOx. These impurities are common in this type of material.

Table 6: Items of Interest (Continued)

FY10 DE Run	3013 ID	Sample of Interest	SRNL Findings	Comments
15	R610785	Oxide was dark brown granular with some compacted chunks.	Spot 1 – Pu, Cl, O, Na, Si, Al Spot 2 – Pu, Cl, Na, Mg, O, W, Fe, Cr Spot 3 – Cl, Pu, K, Mg, Na, Ni, O, Fe Spot 4 – Mg, O, Ni, Cl, Pu, Fe Spot 5 – Cr, Ni, Cl, O, K, Cr, Fe, Mg, Si, Ti, V, Ga Spot 6 – W, Mg, O, Pu, Cl Spot 7 – Pu, O, W, Na	The IDC-Description for the material is 061 – Non-Spec Oxide. These impurities are common in this type of material.
16	R610826	Oxide was dry, dark brown with sizes ranging from powder to approximately 3 inch clumps which were difficult to break up.	Spot 1 – Si, O, Al, Na, K, Cl Spot 2 – Mg, Si, O, Cl, F, Pu, K, Ni, Fe Spot 3 – Pu, O, Mg, Si Spot 4 – Cl, Na, K, O, Fe Spot 5 – Cl, K, Na, Mg, O Spot 6 – Ni, O, Si, Cl, K, Cr, Fe, Mg Spot 7 – Fe, O, Ni, Cr, Fe, K, Cl, Si, Mg Spot 8 – F, Mg Spot 9 – Cl, K, Mg, F, O, Na, Si, W Spot 10 – Ni, O, Na, Cl, K, Cr, Si, Fe, Mg Spot 11 – K, O, Si, Cl	The IDC-Description for the material is 159 – Screenings from Oxide. These impurities are common in this type of material.

Table 7: Can observations from thirteen randomly selected pressure/corrosion bin cans

Surv ID	Material Type and Surv. Reason**	S/N	Moisture (At packaging / DE)	% Actinide	Inner Container Analyzed	Convenience Container Analyzed	Corrosion Observations
10DE3	P&C / R	H003710	0.34 / 0.30	72.4	Y	N	Coating on inner container heavier than CC. Particulate observed – minor (<5um) pits in sidewall.
10DE5	P&C / R	H002447	0.10 / 0.04	78.4	N/A	N/A	None
10DE6	P&C / R	R610627	0.02 / 0.09	53.7	N/A	N/A	None
10DE9	P&C / R	H002567	0.12 / 0.05	55.0	N/A	N/A	None
10DE10	P&C / R	H002728	0.27 / 0.20	71.4	N/A	N	Coating on convenience container and lid – headspace
10DE11	P&C / R	H002786	0.33 / 0.25	71.7	N/A	N	Coating on convenience container and lid – mostly headspace
10DE12	P&C / R	H003077	0.13 / 0.08	81.8	N/A	N/A	None
10DE13	P&C / R	H003367	0.26 / 0.22	52.5	N/A	Y	Blotchy spots on wall of CC in headspace
10DE14	P&C / R	H003704	0.33 / 0.41	70.5	N/A	N	Corrosion pits on CC – not analyzed
10DE15	P&C / R	R610785	0.01 / 0.19	61.4	N/A	N/A	None
10DE16	P&C / R	R610826	0.01 / 0.28	55.3	N/A	N/A	None
10DE17	P&C / R	R610853	0.03 / 0.12	73.1	N/A	N/A	None
10DE18	P&C / R	S001721	0.21 / 0.14	84.3	N/A	N/A	None

**P=Pressure, P&C=Pressure & Corrosion, R=Random, EJ=Engineering Judgment

Table 8: Can observations from five engineering judgment selected pressure/corrosion bin cans

Surv ID	**Material Type and Surv. Reason****	**S/N**	**Description**	**Moisture (At packaging / DE)**	**% Act.**	**Inner Container Analyzed**	**Convenience Container Analyzed**	**Corrosion Observations**
10DE1	P&C / EJ	H004251	Highest TGA to 350°	0.27 / 0.27	58.2	N/A	N	Very slight coating
10DE2	P&C / EJ	H002496	Foreign material inspection	0.25 / 0.17	51.8	N/A	Y – Blotchy sidewall. Coating / product on lid	Pitting corrosion (~30um) on sidewall. Cl seen with EDX Blotchy pits coalescing to ~60 um in diameter – but not that deep. Cl also seen with EDX.
10DE4	P&C /EJ	H003655	High moisture and GB RH	0.38 / 0.33	71.1	Coating on entire inner can	No coating or corrosion	No SEM done. Additional photos taken at weld region. No additional analysis.

Table 8: Can observations from five engineering judgment selected pressure/corrosion bin cans (Continued)

Surv ID	Material Type and Surv. Reason**	S/N	Description	Moisture (At packaging / DE)	% Act.	Inner Container Analyzed	Convenience Container Analyzed	Corrosion Observations
10DE7	P&C / EJ	H003900	Foreign material inspection	0.34 / 0.25	60.4	N	N	Slight removable dusting on CC
10DE8	P&C / EJ	H003650	High moisture with storage weight gain	0.34 / 0.16	75.6	N	Y, Body sidewall and lid	Small (~5 um) pits on sidewall. Cl rich filament or whisker looking particulate on lid of CC
LANL	P&C	H003328	High moisture Hanford can opened at LANL	0.23 / 0.24	75.1	Y	Y	Can analysis to be done in FY11

**P=Pressure, P&C=Pressure & Corrosion, R=Random, EJ=Engineering Judgment

Table 9: Package Reported Moisture vs. Initial Moisture from DE

DE	3013	Reported Moisture (%)	DE Inital Moisture (%)
NDE 1/ DE1	H004251	0.27	0.27
NDE 4/ DE 2	H002496	0.25	0.17
NDE 6 / DE 3	H003710	0.34	0.30
NDE 9 / DE 4	H003655	0.38	0.33
NDE 10 / DE 5	H002447	0.10	0.04
NDE 16 / DE 6	R610627	0.02	0.09
NDE 19 / DE 7	H003900	0.34	0.25
NDE 20 / DE 8	H003650	0.33	0.16
NDE 21 / DE 9	H002567	0.12	0.05
NDE 22 / DE 10	H002728	0.27	0.20
NDE 27 / DE 11	H002786	0.33	0.25
NDE 28 / DE 12	H003077	0.13	0.08
NDE 29 / DE 13	H003367	0.26	0.22
NDE 30 / DE 14	H003704	0.33	0.41
NDE 31 / DE 15	R610785	0.01	0.19
NDE 32 / DE 16	R610826	0.01	0.28
NDE 33 / DE 17	R610853	0.03	0.12
NDE 34 / DE 18	S001721	0.21	0.14

Table 10 : Temparature and Humidity Collected

DE Number	3013	Final Humidity (%RH)	Temp Center (F)	Temp Side (F)	Temp Wall (F)
NDE 1/ DE1	H004251	3.7	115.1	106.5	86.0
NDE 4/ DE 2	H002496	12.1	95.2	85.1	77.4
NDE 6 / DE 3	H003710	5.3	118.0	99.5	84.2
NDE 9 / DE 4	H003655	3.1	118.4	101.3	85.5
NDE 10 / DE 5	H002447	5.3	N/A	N/A	N/A
NDE 19 / DE 7	H003900	5.7	101.4	95.2	82.6
NDE 20 / DE 8	H003650	12.9	102.5	99.1	81.8
NDE 21 / DE 9	H002567	11.8	79.2	78.8	77.5
NDE 22 / DE 10	H002728	4.2	123.8	102.0	84.1
NDE 27 / DE 11	H002786	3.2	115.7	97.4	80.9
NDE 28 / DE 12	H003077	4.1	146.4	121.7	82.4
NDE 29 / DE 13	H003367	14.4	99.5	93.6	78.8
NDE 30 / DE 14	H003704	3.1	120.8	109.9	86.9
NDE 34 / DE 18	S001721	0.7	164.2	N/A	106.1

Table 11: 9975 NDE Surveillance Findings

9975 NDEs	Number of Surveillances performed	9975 Visual Inspections	Celotex® Inspections	Temperature Inspections	SCV O-ring Inspections	PCV O-ring Inspections
FY10	34	0	1[A]	0	0	1[B]

A-NDE-31/DE15 9975-02130 Failed the Axial Gap Celotex®
B- NDE22/DE-10- 9975-06841 PCV failed leak check, Inner O-ring determined to be leaking

Table 12: 9975 DE Results

	9975-02168
Lead carbonate observed	√
Fiberboard thermal conductivity in axial direction exceeded specified range	√

√ Indicates findings outside inspection criteria.

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