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MODEL 9975 LIFE EXTENSION TEST PACKAGE 3 – INTERIM REPORT

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Model 9975 Life Extension Test Package 3 – Interim Report

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Summary

Life extension package LE3 (9975-03203) has been instrumented and subjected to an elevated temperature environment for approximately 8 years. During this time, the cane fiberboard has been maintained at a maximum temperature of ~160 - 165 °F, which was established by a combination of internal (19 watts) and external heat sources.

Several tests and parameters were used to characterize the package components. Results from these tests generally indicate agreement between this full-scale shipping package and small-scale laboratory tests on fiberboard samples, including the degradation models based on the laboratory tests. These areas of agreement include the rate of change of fiberboard weight, dimensions and density, and change in fiberboard thermal conductivity. Corrosion of the lead shield occurred at a high rate during the first several weeks of aging, but dropped significantly after most of the moisture in the fiberboard migrated away from the lead shield. Dimensional measurements of the lead shield indicate that no significant creep deformation has occurred. This is consistent with literature data that predict a very small creep deformation for the time at temperature experienced by this package. The SCV O-rings were verified to remain leak-tight after ~5 years aging at an average temperature of ~170 °F.

This package provides an example of the extent to which moisture within a typical fiberboard assembly can redistribute in the presence of a temperature gradient such as might be created by a 19 watt internal heat load. The majority of water within the fiberboard migrated to the bottom layers of fiberboard, with approximately 2 kg of water (2 liters) eventually escaping from the package.

Two conditions have developed that are not consistent with package certification requirements. The axial gap at the top of the package increased to a maximum value of 1.549 inches, exceeding the 1 inch criterion. In addition, staining and/or corrosion have formed in a few spots on the drum. However, the package remains capable of performing its function. Aging of this package continues.

Background

This report summarizes information on a 9975 package tested per Reference 1, which is part of the comprehensive 9975 package surveillance program [2]. This task provides an integrated assessment of the package response to environmental extremes, and demonstrates the extent to which data from small laboratory samples scale up to a full package. The primary goal of this task is to validate aging models based on lab scale testing of the cane fiberboard overpack and containment vessel O-rings. A secondary goal is to examine the behavior of the lead shielding and other components under bounding conditions.

Three 9975 packages were modified to provide instrumentation for monitoring package performance and response to environmental aging. Each package has a different environmental exposure history. Testing of the first two packages has been previously terminated and the results documented [3, 4]. The third package (LE3, or 9975-03203) has been in test for 418 weeks (as of November 30, 2016) and remains in test. This report summarizes and analyzes the test data available to date for test package LE3.

The primary focus of LE3 was to age the fiberboard at a temperature bounding to KAC storage conditions, but not excessively so. Reference 5 identifies a conservatively bounding average KAC ambient temperature of 95 °F, and a maximum KAC ambient temperature of 109 °F, based on RF-TID sensors. Note that this maximum temperature was not sustained for an extended period and was measured immediately adjacent to a package (not the average room temperature). For the LE3 test package, the average drum surface temperature (at ~mid-height) was 122 °F, providing a slight margin to the maximum facility ambient temperature.

Experimental Method

This life extension package is a 9975 shipping package that was modified to add instrumentation and an internal heat source. An access port for fiberboard sample removal was added to the drum side wall. Thermocouples provide the temperature at a number of locations throughout the package, including the 3013 payload, PCV, SCV, multiple locations within the fiberboard, and drum surface. A sketch showing thermocouple locations is provided in Figure 1.

Package LE3 was placed within an environment with external temperature control. A cartridge heater inside a modified 3013 container provides 19 watts internal heat. The external temperature was established by enclosing the package in a modified 55 gallon drum, and placing a drum heater around this larger drum (Figure 2). The external drum heater was adjusted to maintain a maximum fiberboard temperature of ~160 °F (actual values ranged up to ~165 °F). This was typically accomplished at a setting of ~100 – 105 ºF, which produced an average drum surface temperature of 122 ºF. This condition is slightly conservative to the maximum ambient temperature observed in KAC, and all packages in KAC contain less than 19 watts. Therefore, this test provides internal temperatures that are slightly conservative to the hottest packages in storage.

The PCV and SCV were modified to allow placement of a cartridge heater through the bottom of the containment vessels and into a well in the 3013. The 3013 was welded shut with a surrogate load of steel shot. The cartridge heater conductors and thermocouples attached to the 3013, PCV and SCV exit the package opposite the side where the fiberboard is instrumented, to minimize disruption of the measured fiberboard thermal profile.

The modifications to the PCV and SCV provided open penetrations in the bottom of each vessel. Because of this, both O-rings in each vessel can receive a sensitive helium leak test. Normally, only the outer O-ring is leak-tested with a helium detector, which provides assurance of a leak-tight seal of 1×10^{-7} std cc air/sec (or 2×10^{-7} std cc He/sec). After loading a package, the leak-tightness of both O-rings is typically confirmed at a level of $\sim 1 \times 10^{-3}$ std cc air/sec with a less sensitive rate of pressure rise technique. With the modified vessels of LE3, the more sensitive helium leak test can be performed at any time.

Thermocouple data from the package is automatically recorded at preset intervals. Additional data are collected on an occasional basis during periodic examinations. This includes:

- Weight of the entire package
- Weight and moisture content of the removable fiberboard sections
- Visual observations of the package exterior
- Weight, dimensions and moisture content of the upper and lower fiberboard assemblies
- Dimensions of the lead shield
- Visual observations of other package components

The last 3 of the above data sources require opening the drum lid and removing internal components. These steps are not performed with every examination, so these data are not collected as often as the other items listed.

Results – Periodic Data

There are several metrics which provided evidence of change in the package over time. These include package weight, fiberboard moisture levels, and the temperature profile across the fiberboard. Additional indications of change might be seen in the weight, dimensions and appearance of the fiberboard, dimensional variation of the lead shield, and from visual observations of the components.

The following observations were made of specific changes within the package:

- After 2 weeks in test, liquid condensation was observed on the underside of the drum lid and on the air shield (Figure 3). The moisture level of the bottom fiberboard layers is above saturation (>38 % wood moisture equivalent, or %WME).
- After 5 weeks in test, mold was observed on the lower layers of the bottom removable fiberboard section. The mold changed color and texture through 45 weeks in test, and gradually became dormant by week 151. Little change has been observed after that. (Figure 4)
- After 7 weeks in test, increased corrosion on the outer surface of the lead shield was observed, along with some nodular corrosion deposits. Lead shield corrosion approached its heaviest visual appearance by 26 weeks, and the upper portion of the shield darkened slightly after that (Figure 5).
- After 109 weeks in test, corrosion was observed on the drum interior at the bottom crevice (Figure 6). This corrosion is not associated with any fabrication weld, and has not grown through-wall.
- After 119 weeks in test, possible corrosion was observed on one stitch weld along the bottom edge of the drum exterior (Figure 7). After 151 weeks, additional corrosion was observed on the same stitch weld and on additional stitch welds.
- After 190 weeks in test, staining and/or corrosion was observed on the drum interior at the top flange stitch welds (Figure 8).

A plot of package weight over time is shown in Figure 9. The weights of the upper and lower fiberboard assemblies were also tracked over time, although they were recorded less frequently. These data are shown in Figure 10. Two small sections of fiberboard were cut from the bottom of the lower assembly, and are accessible to remove through a hatch on the drum side. These sections are characterized more often than the drum is opened to inspect the upper or lower fiberboard assembly. Weight data for these removable sections are shown in Figure 11.

Fiberboard dimensions change over time. Table 1 summarizes some of the fiberboard dimensions and their relative change. In general, the following changes are observed:

- Fiberboard height decreases over time
- Fiberboard thickness (in the dryer regions) decreases over time
- Fiberboard diameter at the bottom increases as that region gains moisture, then decreases as the package overall loses moisture.

Moisture was originally distributed uniformly throughout the fiberboard assembly. Upon heating, it quickly re-distributed towards the OD surface and bottom. Average moisture levels of several fiberboard regions are summarized in Table 2, along with moisture content values for the small removable fiberboard sections.

Given the relatively low melting temperature of lead (622ºF), the shield may experience creep at service temperatures. Measurements of the LE3 lead shield were collected to investigate this possibility, and are summarized in Table 3.

The corrosion of the lead shield is also of interest. Significant corrosion product was observed after 7 weeks in test, and was white and nodular (Figure 5). The corrosion product had become somewhat heavier after 26 weeks in test, but did not appear to increase significantly after that. Instead, the appearance began to change slightly in that the upper portion of the lead shield (adjacent to the upper fiberboard assembly) yellowed slightly (observed after 109 weeks in test) and retained that appearance during subsequent testing. A greater degree of discoloration was observed on the lead shield from the second life extension package. Analysis of the corrosion product from that shield showed it to be consistent with lead carbonate, with up to 10 wt% chlorine (likely from the fiberboard) [3]. The lead carbonate corrosion typically seen on a 9975 lead shield is smooth and white. Once the corrosion product on a typical lead shield is sufficiently thick (a few thousands of an inch), it will begin to loosen and flake off.

After 262 weeks at temperature, the SCV O-rings were leak tested as part of the package inspection. Both O-rings remained leak-tight at that time.

Discussion

Significant moisture redistribution was occurring as the package was initially brought to temperature. Moisture in the bottom fiberboard layer had increased past saturation (~38 %WME) before the first inspection after 2 weeks (1.4 weeks at temperature). The inner (warmest) fiberboard regions would have begun losing moisture immediately, while moisture levels in the outer regions did not change significantly until ~48 weeks at temperature. Moisture redistribution likely occurred through several mechanisms, including evaporation of water in the hotter regions, and condensation on the relatively cool drum and lid surfaces. This is seen in Figure 3, where condensation appears to have formed on the lid and rained down onto the air shield. As significant water accumulates on the air shield, it is likely that some of it might spill over the side. At that point, it could either run down the drum wall or re-absorb into the fiberboard, depending on the gap between the fiberboard and drum wall. Additional condensation might form directly on the drum sides and run down to the bottom. These mechanisms would likely remain active until the majority of water in the fiberboard re-distributes toward the bottom of the package.

Initially, the fiberboard had a uniform moisture content of ~11 %WME, which corresponds to ~10 wt%. This moisture content is typical of many fiberboard assemblies. With a total fiberboard weight of 35.041 kg (subtracting 6.623 kg nominal weight for the air shield and bearing plates from the combined fiberboard assemblies weight), the fiberboard initially contained 3.186 kg of water and the dry fiberboard weight was 31.855 kg. Figure 12 shows that the total fiberboard weight decreased at an initial high rate, and then at a lower rate after ~200 weeks at temperature. Figures 13 and 14 show the fiberboard moisture content remains ~constant during the second period (>200 weeks), indicating no net water loss/gain. Therefore the weight loss during this period is a result of fiberboard (cellulose) loss only. Figure 12 illustrates this rate of fiberboard weight loss extrapolated back to time zero, where the total weight loss can be divided into cellulose loss and water loss. From this we see a net loss of 2.012 kg water and 0.646 kg fiberboard (cellulose) after 402 weeks at temperature. With movement of this amount of water through the package, the amount of condensation on the lid and air shield shown in Figure 3 is not surprising. It is noted that significant water staining from condensation on the lid and air shield was also observed on the second life extension package [3], however, that package was not opened for inspection until after 19 weeks, at which time the moisture was no longer present.

Migration of water to the cooler regions of the package would leave the bulk of the fiberboard much drier than it was initially. This in turn would decrease the thermal conductivity for the bulk of the fiberboard, and increase the temperature gradient across the fiberboard. The eventual loss of moisture from the package would exaggerate this effect.

Twelve thermocouples monitor the temperature gradient within the fiberboard. The hottest measured temperature within the fiberboard is at the highest thermocouple elevation on the ID surface, and the coolest measured temperature within the fiberboard is at the lowest thermocouple elevation on the OD surface. The temperature vs time data at these two locations, as well as the PCV, a second fiberboard OD location and the drum exterior are summarized in Figure 15. It is possible that the fiberboard reached slightly higher temperatures at elevations above the uppermost ID thermocouple. However, the variation among the thermocouples along the ID surface in the central region adjacent to the shield shows a very small axial gradient (typically < 1 ºF). Therefore, the recorded temperatures are assumed to provide a reasonable approximation of the maximum fiberboard conditions.

Potential for lead shield creep

The data in Table 3 summarize measurements of the lead shield wall thickness for LE3. Changes in the measured wall thickness might occur from the formation and/or shedding of corrosion product, from creep of material at the top toward the bottom of the lead shield, or from measurement uncertainties. Initially, corrosion product was observed to form with apparent uniformity on the lead shield, although at later times it may have flaked off locally. A consistent trend in which the thickness at the bottom increases by more than that at the top could be indicative of creep deformation. However, this was not observed in LE3. Rather, the measured thickness varied both up and down at both the top and bottom of the lead shield.

A similar analysis for the lead shield in life extension package 2 (LE2) suggested a creep rate of 0.009 to 0.02 inch/year was active in that package [3]. That shield experienced a temperature of ~250 ºF (or 66% of the melting temperature). This estimate was bracketed by literature data [6] which describes the creep rate for two extremes of grain size (10 microns and 1 mm). These same literature data predict creep rates for the lower lead shield temperature of LE3 to be ~5% of the rates predicted for the LE2 temperature. Accordingly, it is reasonable to expect a creep rate of the LE3 lead shield of < 0.001 inch/year. Even with 8 years aging, the potential lead shield deformation would be <0.008 inch, which is small compared to measurement variability and uncertainties introduced by the corrosion layer. Therefore, while a very small amount of lead creep may have occurred, it is not enough to be readily detectable or to cause concern. Typical lead shields in a storage application will experience an average temperature even less than the LE3 lead shield, and creep rates for them should be negligible.

Lead Shield Corrosion

A heavy corrosion layer was noted on the lead shield after 26 weeks (21.3 weeks at temperature), and was not observed to change significantly during subsequent inspections. If the baseline weight of all fiberboard is subtracted from the total package baseline weight, the remaining components had a combined baseline weight of 138.9 kg. After 43.7 weeks at temperature (the next time all fiberboard was weighed), the remaining components had a combined weight of 139.1 kg. This residual weight has remained approximately constant since that time. The weight gain of ~200g is attributed to lead corrosion.

Reference 7 describes the corrosion process of lead within a 9975 environment, and reports on lead corrosion rates measured under a range of conditions relative to that environment. This reference recommended a bounding corrosion rate of 2 mils/year be used for the long-term corrosion of the lead shield in normal storage conditions. However, it also identified circumstances where the corrosion rate could be significantly higher. These circumstances include elevated temperature (122 and 167 ºF vs room temperature), high humidity (near 100%) and replenishment of the ambient moisture and CO_2 levels. Reference 7 demonstrated high corrosion rates for elevated temperature and high humidity conditions with short exposure times (2 - 6 mil loss of lead metal in 30 days and 1 – 3 mil loss of lead metal in 75 days), giving short-term corrosion rates of 25 mils/year or higher. These total amounts of lead metal loss were also seen for longer (120 day) exposures, indicating a significant decline in the corrosion rate. However, if the samples were exposed to a refreshed atmosphere to replenish the moisture and CO_2 levels, the elevated corrosion rates continued for a longer period. There is significant scatter in these data, but they show the potential for significant corrosion to occur in a short period if the right conditions are present. It is postulated that such conditions existed in the LE3 package during the initial few weeks of aging.

The primary driving force for lead corrosion is the release of acetic acid from the polyvinyl acetate glue used in the fiberboard assembly. It can release acetic acid (a catalyst for lead corrosion) as it cures. It can also release acetic acid due to a hydrolysis reaction if exposed to high humidity levels and elevated temperature. This condition likely existed within the LE3 package during the early weeks as significant amounts of moisture were moving to the cooler regions of the fiberboard assembly.

It is postulated that the lead shield gained 200g in weight during the initial aging period. If this weight gain came from the production of basic lead carbonate, $Pb_3(CO_3)_2(OH)_2$, then this weight gain resulted from the consumption of 807g lead metal. With a nominal density of 0.41 lb/in^3, this corresponds to a loss of 4.34 cubic inches of lead. The side of the lead shield (conservatively ignoring the bottom surface) has a nominal surface area of 641 square inches. The uniform loss of 4.34 cubic inches would remove a 6.8 mil layer of lead metal, corresponding to a corrosion rate of 17 mils/year in the first 21.3 weeks at temperature. Reference 7 describes that the corrosion product is actually a combination of basic lead carbonate, $Pb_3(CO_3)_2(OH)_2$, and lead carbonate, $PbCO_3$. If some of the corrosion product from LE3 is lead carbonate, less metal loss would be needed to account for the observed weight gain. In addition, analysis of the lead shield corrosion product from the second life extension package identified up to 10 wt% chlorine was present. The slight discoloration of the LE3 lead shield corrosion product suggests some impurities are also present. Such impurities would further reduce the amount of metal loss for the observed weight gain. Therefore, a 200g weight gain of the LE3 lead shield is consistent with the short-term high corrosion rates measured in Reference 7. Note, however, that these corrosion rates are temporary and long-term corrosion rates of a 9975 lead shield under storage conditions should still be bounded by the recommended 2 mils/year rate.

Other Components
While limited corrosion was observed on the LE3 drum, it is noted that corrosion also occurred on the drums of the first two life extension packages. In comparison, the LE3 drum began corroding later, and has corroded less than the other two packages. Some of this corrosion is observed in areas where the drum was modified, and may be related to the modification activities, although some is also associated with non-modified areas. Corrosion of the stainless steel drum might be expected if moisture migration within the fiberboard leads to the concentration of chlorides on the drum surface, or from biological influences. However, the corrosion observed in LE3 is less severe than that observed in the earlier packages, and is judged to not represent a challenge to the drum integrity.

The SCV O-rings were shown to be leak-tight after 262 weeks (5 years) at temperature. The O-ring region of the SCV maintained an average temperature of ~170 °F during aging. This is significantly cooler than the O-ring fixtures that have been aging at 200 °F for 8 – 10 years with no leakage failures [8]. Therefore, it is expected that the SCV O-rings will remain leak-tight for a significantly longer time. The PCV O-rings were not leak tested since that would have required opening the SCV. However, the PCV O-rings maintained an average temperature during aging of ~180F, which is also significantly less than the O-ring fixtures that have been aging at 200 °F for 8 – 10 years. Therefore, the PCV O-rings are also expected to remain leak-tight for a significantly longer time.

Validation of Laboratory Data

Variations in regional fiberboard moisture content are shown in Figures 13 and 14. As discussed elsewhere [9], moisture within the fiberboard tends to seek equilibrium with moisture in the surrounding air within the package, and with moisture in the air outside the package. The absolute humidity of the air tends to remain approximately constant throughout the package, while the relative humidity varies significantly with any temperature gradient. The equilibrium moisture concentration of the fiberboard for a given moisture concentration in the air also changes with temperature. At the same time, a slow transfer of air between the package interior and the room allows for a net transfer of moisture into or out of the package, although this tends to be a very gradual process. With this in mind, the trends in Figure 13 indicate much of the fiberboard moisture initially was driven to the bottom fiberboard layers, and a gradual net loss of moisture from the package was active through the first ~200 weeks at temperature. Since that time, the moisture levels within the package appear to have been roughly constant.

Several specific attributes of the LE3 package can be compared directly to laboratory data and the model predictions based on the laboratory data. These include fiberboard weight, dimensions, density and thermal conductivity. These comparisons are summarized in Table 4, and are discussed separately below. Comparison is not made to fiberboard specific heat capacity since there is no instrumentation to capture changes in this property within LE3. However, laboratory data indicate this property changes relatively little as the fiberboard ages.

Each prediction from the degradation models is based on a specific fiberboard environment (temperature, relative humidity). The environments within LE3 vary for each fiberboard region, and are estimated as follows:

- Bottom fiberboard layers: The average temperature recorded at the lowest fiberboard OD position is ~125 °F. Since this position is about 4 inches above the bottom, and the temperature will decrease at lower elevations, it is estimated that the average temperature of the bottom fiberboard layers is ~120 °F. Initially, the average moisture content of the bottom fiberboard layers increased from ~11 %WME to above saturation (>38 %WME or >28 wt%), as measured around the outer edge. After decreasing to about 9 %WME after 262 weeks at temperature the moisture content of the bottom fiberboard layers has remained approximately constant. An average moisture content of >20 %WME will be assumed for the initial period of ~200 weeks, corresponding to an equilibrium relative humidity (at room temperature) for these moisture levels of >80% [10]. The actual relative humidity for this period will be higher due to the elevated aging temperature. Since this relative humidity is above the range for which the degradation models were developed (up to 70%), the model predictions are not applicable to this extreme condition. An average moisture content of the bottom layers of 9 %WME will be used for the later aging period. It is recognized that the interior portions of the bottom layers might not hold this much moisture (based on observations of the second life extension package [3]) so this value is an upper bound estimate. The equilibrium relative humidity (at room temperature) for 9 %WME is 42-55% at room temperature, and 58-71% at the elevated aging temperature [10].

- Fiberboard sidewall between the bearing plates: This region includes portions of the upper and lower fiberboard assemblies. The two upper fiberboard ID and OD thermocouples are located centrally between the bearing plates, and they will be used to represent the average temperature of this region. With average temperatures of 158 and 126 °F on the ID and OD, respectively, the average temperature of this fiberboard region is 142 °F. The moisture content along both the ID and OD surfaces dropped to values near or below the moisture meter detection limit (6 %WME). The observation of occasional readings just above the detection limit suggests that they likely did not drop very far below this limit. Therefore this region can be characterized with an initial moisture content of ~11 %WME which gradually decreased to an average value of ~6 %WME after ~100 weeks, and remained approximately steady since then. It will be assumed that this region had an average moisture content of ~8.5 %WME for the first 100 weeks, and ~6 %WME subsequently. For cane fiberboard, a moisture content of 8.5 %WME is in equilibrium with ~40 – 50% relative humidity in the air at room temperature, and ~56 – 66 % at elevated temperature (e.g. ~142 °F average fiberboard temperature within this package) [10]. A fiberboard moisture content of ~6 %WME is in equilibrium with ~28 – 36% relative humidity in the air at room temperature, and ~44 – 52 % at elevated temperature [10].

- Lower fiberboard assembly: The environment for the lower assembly combines that described above for the bottom layers with that between the bearing plates. With 70% of the lower assembly volume above the lower bearing plate, the average environment can be taken as a weighted average of these two regions. This provides an average environment for the first period of 135 °F and >70% relative humidity, and an average environment for the second period of 135 °F and 52% relative humidity.

- Upper fiberboard assembly: Part of the upper fiberboard assembly is between the bearing plates, and reflects that environment. The remainder of the upper assembly is expected to have an axial thermal gradient similar to the radial gradient between the bearing plates. Therefore, the average temperature for the upper assembly should be ~142 °F. Moisture measurements were made on the

ID and OD surfaces of the upper fiberboard assembly. These closely match values for these same surfaces on the lower fiberboard assembly, so the average moisture content of the upper fiberboard assembly will be assumed to match that of the fiberboard between the bearing plates.

- All fiberboard: The average environment for the combined upper and lower fiberboard assemblies can be estimated by a weighted average of the environments for each assembly. The fiberboard weight of the upper assembly is 23% of the total fiberboard weight (after subtracting the weight of bearing plates and air shield). A weighted average of the above estimated environments indicates an average environment of 137 °F ~70%RH for the first period, and 137 °F 51 %RH for the second period.

- Removable fiberboard sections: The environment for the two removable fiberboard sections is comparable to the material surrounding them. With both of these sections located below the lower bearing plate and along the OD surface, an average temperature of 120 °F is considered reasonable. The lower removable section includes a portion of the bottom fiberboard layers, and the average moisture content was near or above saturation for the first 100 weeks. This corresponds to an equilibrium relative humidity >80% at room temperature and would be even higher at elevated temperature. Beyond ~200 weeks, the average moisture content of the lower removable section is ~8 %WME, which corresponds to an equilibrium relative humidity of 31 – 46% at room temperature, and 47 – 62 at elevated temperature [10]. The average moisture content of the upper removable section is ~ 10 %WME for the first period, and ~6 %WME for the second period. These values correspond to equilibrium relative humidity values of 43 – 58% and 28 – 36%, respectively, at room temperature [10]. At elevated temperature, the relative humidity increases to 59 – 74% and 44 – 52%, respectively.

Fiberboard weight changes

Variation in the total weight of the package is summarized in Figure 9. Variation in the weights of the fiberboard assemblies and the removable fiberboard sections are shown in Figures 10 and 11, respectively. All fiberboard within the package was removed and weighed on relatively few occasions (to minimize the opportunity for inadvertent moisture changes). Additional information regarding fiberboard weight can be obtained from the more frequently obtained total package weight, assuming the package weight has changed only as a result of fiberboard weight change and lead shield corrosion.

As discussed above, the lead shield gained 200g in weight during the initial few weeks of aging. Beyond that change, any additional change in the total package weight represents fiberboard weight change. The fiberboard weight based on this method is plotted in Figure 12 with the “x” symbols. The red squares in Figure 12 show the combined total weight of all fiberboard. There is very good agreement between the two methods in Figure 12, indicating that the greater amount of data from package weight can be used to represent the change in fiberboard weight as well.

The upper fiberboard assembly lost weight at a rate of 2.4 %/year during the first period, then dropped to a rate of 0.19 %/year. The lower fiberboard assembly lost weight at a rate of 1.4 %/year during the first period, then dropped to a rate of 0.25 %/year. The combined weight loss rate for both assemblies is intermediate to these values. The weight loss model developed in Reference 11 predicts degradation rates above or intermediate to these values, but does not take into account the significant movement of moisture which further impacts the fiberboard weight change.

Fiberboard dimension changes
Changes in several fiberboard dimensions can be tracked for comparison to laboratory data. Table 1 lists data for the radial thickness for each assembly, the OD and ID height of the lower fiberboard assembly, and the bottom diameter.

The radial thickness data for the lower and upper fiberboard assemblies are plotted in Figure 16. For both assemblies, the thickness of the two radial steps is added for the total radial thickness. For the upper assembly, the radial thickness can also be obtained from the difference between the OD and ID dimensions, and gives a slightly different result. In Figure 16(b), the radial thickness for the upper fiberboard assembly is plotted based on both of these approaches, and the trend lines are shown for the average of the two approaches. This second approach is not used on the lower fiberboard assembly since the OD and ID are not measured at the same elevation. For both assemblies, the trend lines show low degradation rates (-0.03 %/year lower assembly or 0.00 to -0.22 %/year upper assembly). The degradation model for thickness based on laboratory data [10] predicts similarly low degradation rates (Table 4).

The relative diameter change for the bottom layers of the lower fiberboard assembly is shown in Figure 17. The excess moisture initially driven to the bottom layers dominates diametral changes for up to 262 weeks. The rate then decreases significantly to a value less than the rate of radial thickness loss. With the lower temperature in the bottom layers, the models based on laboratory data predict no degradation for moisture levels below 70 %RH equivalent, and are not applicable to higher moisture levels.

As the fiberboard moisture migrates toward the bottom of the assembly, the bottom layers will tend to compress since the added moisture weakens the fiberboard and these bottom layers are under load from the package internal components. In addition, the higher fiberboard regions that have lost moisture will shrink [12]. Both of these effects contribute to the 2.6% decrease in lower assembly OD height seen at the first inspection after the package began aging. Subsequent changes in fiberboard OD height result from aging of the fiberboard, and show a rate of decrease in height of 0.38 %/year (Figure 18). A slightly smaller rate of decrease (0.25 %/year) is seen for the ID height. After 234 weeks at temperature, the degradation rate drops to 0.07 %/year for both the ID and OD heights. The model for fiberboard height [11] slightly over-predicts these rates (Table 4).

Related to the change in fiberboard height is the change in axial gap at the top of the package. The axial gap is plotted in Figure 19. The baseline axial gap is smaller than nominal (0.545 inch vs 0.8 inch). During initial assembly, the axial gap was smaller on one side (0.459 inch) and interfered with drum closure such that the closure bolts were used to compress the fiberboard somewhat and pull the lid tight. From this extreme axial gap condition, interpolation of the data indicates the axial gap exceeded 1 inch after ~20 weeks at temperature. This 1 inch threshold is taken as an action point for packages in service to investigate whether significant package degradation has occurred. The axial gap in LE3 continued to increase, with a maximum value of 1.549 inches after 262 weeks at temperature.

Fiberboard density changes
Based on the measured fiberboard assembly weight and dimensions, the density can be calculated after allowing for the bearing plates and air shield. The calculated density for each fiberboard assembly is plotted in Figure 20. A decrease in the density degradation rate is seen after ~150 weeks for each assembly, corresponding to a decrease in the average moisture content. The rates of density change are

summarized in Table 4, along with comparison to model predictions from Reference 11, showing reasonable agreement.

Thermal conductivity changes
Package LE3 data were examined for evidence of change in fiberboard thermal conductivity during the testing. The methodology follows that described in Reference 3. The sidewall region of the lower fiberboard assembly provides a region of relatively constant thermal response. Since the two lower ID thermocouples failed during the course of testing, the upper half of this sidewall region will be used to estimate change in thermal conductivity. This provides the data from 2 ID thermocouples and 2 OD thermocouples, which will be averaged. The temperature along either surface varies little within this region during steady state operation. Since some heat is also lost through the top and bottom of the package, the heat conducting through this side region is less than 19 watts, but it will be assumed constant over time. Heat conduction in this region is in the radial orientation.

Thermal gradient information was examined at several discrete times during periods of steady state operation indicated by the vertical arrows in Figure 21. The thermal conductivity will vary with changes in the temperature gradient and fiberboard thickness, as described by:

$$q/A = k * \Delta T / t$$

where, q/A = heat flux (assumed constant)
k = thermal conductivity
ΔT = radial temperature gradient
t = lower assembly radial thickness (inspection data show an initial average radial thickness of 4.758 inches, and an average rate of change in radial thickness of -0.034 %/year)

At each of the arrows in Figure 21, the radial temperature gradient and lower assembly thickness are combined per this relationship to get a value proportional to the thermal conductivity (see Table 5). Any change in this value over time is proportional to the change in actual thermal conductivity. (Since the actual heat flux through this region of fiberboard is unknown, the actual thermal conductivity cannot be calculated directly.) Thermal conductivity will vary with fiberboard temperature. Laboratory data gives a magnitude for this variation of a 1.0007 factor for each degree F. While this effect is small, it is included in the calculated change in thermal conductivity.

Estimated changes in thermal conductivity are listed in Table 5 and plotted in Figure 22, along with normalized laboratory data for samples conditioned at 125 and 185 ºF. Both laboratory samples were tested at 122ºF. In this presentation, the relative thermal conductivity values for LE3 vary, but the overall trend is a slight decrease in thermal conductivity over time (-0.35 %/yr). This degradation rate falls between those for laboratory samples aged in 125 and 185 °F dry environments.

Overall Assessment of degradation model predictions
Each of the above comparisons between rate of change in LE3 and model predictions based on laboratory data are summarized in Table 4. In many cases, the data from LE3 showed two distinct degradation rates, depending on the overall moisture condition of the package. In other cases (such as thermal conductivity and radial thickness) only one distinct trend was seen over the entire duration. Model predictions were prepared for both periods for all properties.

For most properties, the model predictions are slightly conservative to the measured degradation rates. The greatest disagreement is seen in fiberboard weight change. This is likely due to the direct impact of water movement and subsequent water loss, which is measured along with loss of fiberboard mass.

The degradation models are based on the average behavior of samples taken from several 9975 packages, and some package-to-package variation exists. Until samples from the LE3 fiberboard assembly are tested directly, it is unknown how this package compares to the range of behavior seen in the laboratory samples. It is also likely that within a full package, there will be variation in the relative "leakiness" of the package, which will impact the rate of moisture gain or loss. Given these caveats, the degradation model predictions are considered a good approximation of the behavior of the LE3 fiberboard.

Conclusions

Life extension package LE3 has been instrumented and exposed to a bounding storage condition to help identify the extent to which laboratory test results for fiberboard, O-rings and other components apply to a full-scale package. Results to date indicate the following:

- As seen with the previous life extension packages, the presence of a thermal gradient across the fiberboard assembly leads to a corresponding moisture gradient, with moisture moving preferentially to the cooler regions of the package. Following the initial re-distribution of moisture, the package eventually lost 2 kg water and remained at this equilibrium value during subsequent aging.
- With elevated temperature, the fiberboard loses mass and shrinks. Shrinkage is greatest in the axial direction. The rates of weight loss and dimensional changes are generally consistent with predictions based on laboratory samples, although variations in moisture concentration can alter the apparent degradation rates, especially in the bottom fiberboard layers.
- Estimates of the change in fiberboard thermal conductivity (in the radial direction) show a decrease over time. This observed change becomes consistent with predictions based on laboratory samples after the package moisture content was reduced to an equilibrium value.
- The lead shield experienced significant corrosion within the first 26 weeks, with relatively little subsequent change in appearance. Corrosion product on the upper portion of the shield eventually became somewhat yellow in appearance. The corrosion rate of the lead shield was very high during the initial aging period when significant moisture was moving throughout the fiberboard, but the long-term average corrosion rate is well below the previously recommended rate of 2 mils/year.
- There is no consistent trend of change in shield dimensions that would suggest any creep deformation has occurred. This is consistent with literature data that indicate a small amount of creep might be expected over the 8 years aging experienced by LE3, but such deformation could easily be masked by the corrosion buildup and measurement uncertainties.
- The SCV O-rings remained leak-tight after 5 years at temperature. Based on laboratory testing at higher temperatures, the O-rings are expected to remain leak-tight for a significantly longer period.

With the exception of the increased axial gap (to 1.549 inch), and modest staining / corrosion at some of the welds on the drum, package LE3 continues to conform to requirements and maintain its full functionality. It remains in test.

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[11] SRNL-STI-2015-00610, “Status Report – Cane Fiberboard Properties and Degradation Rates for Storage of the 9975 Shipping Package in KAC”, W. L. Daugherty, December 2015.

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Table 1. Dimensional data for LE3 fiberboard

Time at Temperature (weeks)	Upper assembly radial thickness* (in)	Change in thickness (%)	Lower assembly radial thickness* (in)	Change in thickness (%)	Diameter across bottom (in)	Change in Diameter (%)
Baseline	4.519	0.00	4.757	0.00	18.029	0.00
24.4	4.507	-0.27	**	**	**	**
39.3	4.511	-0.18	**	**	**	**
48.0	4.490	-0.64	4.735	-0.46	18.047	0.10
77.0	4.494	-0.55	4.747	-0.21	18.056	0.15
100.3	4.496	-0.51	4.752	-0.11	18.036	0.04
126.7	4.482	-0.82	4.784	0.57	18.048	0.11
179.0	4.511	-0.18	4.748	-0.19	18.022	-0.04
234.1	4.488	-0.69	4.755	-0.04	18.022	-0.04
247.3	4.502	-0.38	**	**	**	**
262.0	4.484	-0.77	4.755	-0.04	18.008	-0.12
371.7	4.498	-0.46	4.750	-0.15	18.005	-0.13
402.0	4.496	-0.51	4.728	-0.61	18.001	-0.16

* Radial thickness values are the sum of two separate radial steps

** The lower fiberboard assembly was not measured during these inspection intervals.

Time at Temperature (weeks)	Lower assy OD height (in)	Change in Height (%)	Lower assy ID height (in)	Change in Height (%)
Baseline	26.861	0.00	20.469	0
48.0	26.160	-2.61	20.228	-1.177
77.0	26.146	-2.66	20.192	-1.353
100.3	26.063	-2.97	20.169	-1.466
126.7	26.016	-3.15	20.146	-1.578
179.0	25.891	-3.61	20.090	-1.852
234.1	25.822	-3.87	20.045	-2.071
262.0	25.795	-3.97	20.035	-2.120
371.7	25.760	-4.10	19.995	-2.316
402.0	25.760	-4.10	20.003	-2.277

Table 2. Moisture content of LE3 fiberboard

Time at Temp (weeks)	Average Moisture Content (%WME)				
	Fiberboard ID between bearing plates	Fiberboard OD between bearing plates	Upper removable section	Lower removable section	Bottom of lower assembly
0.0	11.7	10.4	9.2	9.7	12.1
1.4	*	*	14.9	31.6	*
4.9	*	*	13.0	saturated	*
6.7	*	*	13.8	saturated	*
14.7	*	*	13.4	saturated	*
24.4	<6	11.7	*	*	*
26.7	*	*	12.8	saturated	*
39.3	<6	10.3	11.4	28.5	*
48.0	<6	8.8	10.8	saturated	100
59.0	*	*	9.7	saturated	*
77.0	<6	7.8	11.2	saturated	100
85.7	*	*	11	*	*
100.3	<6	<6	10.2	29.9	100
110.0	*	*	8.9	21.7	*
126.7	<6	7.0	10.0	29.2	100
142.4	*	*	10.4	18.4	*
160.6	*	*	7.5	12.1	*
169.3	*	*	8.2	14.0	*
179.0	*	*	8.4	12.8	*
195.0	*	*	7	11.5	*
215.9	<6	<6	<6	10.0	20.2
234.1	6.5	6.8	7.4	9.8	15
247.3	<6	<6	6	10.6	*
262.0	<6	<6	<6	7.0	12.6
283.3	*	*	6.35	9.8	*
328.3	*	*	6	9.0	*
371.7	<6	<6	<6	7.8	10.5
402.0	<6	<6	<6	8.0	11.7

* The moisture content at this location was not measured during this inspection interval.

Table 3. Dimensional measurements of LE3 shield to investigate the potential for lead creep.

Time at Temp (weeks)	Avg Radial Thickness at bottom of shield (inch)	Avg Radial Thickness at top of shield (inch)	Difference (Bottom – Top) (inch)
Baseline	0.547	0.544	0.003
48.0	0.558	0.560	-0.002
77.0	0.560	0.570	-0.010
100.3	0.570	0.565	0.005
126.7	0.573	0.550	0.023
234.1	0.560	0.568	-0.008
262.0	0.562	0.566	-0.004
402.0	0.560	0.571	-0.011

Table 4. Comparison of LE3 fiberboard degradation rates and model predictions based on laboratory data

		Fiberboard Degradation Rates (%/year)			Fiberboard Degradation Rates (%/year)	
Fiberboard Region & Property	Average Environment	Initial Rate	Model Prediction	Average Environment	Second Rate	Model Prediction
All fiberboard weight	137F ~70%RH	-1.61	-2.55	137F 51%RH	-0.25	-0.49
Upper assembly weight	142F 61%RH	-2.41	-1.64	142F 48%RH	-0.19	-0.67
Lower assembly weight	135F >70%RH	-1.42	>-1.9	135F 52%RH	-0.25	-0.43
Upper removable section weight	120F 66%RH	-1.05	0	120F 48%RH	-0.28	0
Lower removable section weight	120F >80%RH	-4.23	NA	120F 54%RH	-0.53	0
Lower assembly OD height	135F >70%RH	-0.38	>-0.58	135F 52%RH	-0.07	-0.09
Lower assembly ID height	142F 61%RH	-0.25	-0.48	142F 48%RH	-0.07	-0.16
Lower assembly radial thickness	142F 61%RH	-0.03	-0.08	142F 48%RH	-0.03	-0.04
Upper assembly radial thickness	142F 61%RH	-0.22	-0.08	142F 48%RH	0.00	-0.04
Lower assembly bottom layers diameter	120F >80%RH	-0.06	NA	120F 64%RH	-0.01	0
Lower assembly density	135F >70%RH	-0.80	>-0.90	135F 52%RH	-0.13	-0.24
Upper assembly density	142F 61%RH	-1.45	-0.80	142F 48%RH	-0.16	-0.36
Thermal conductivity (radial)	142F 61%RH	-0.35	-1.16	142F 48%RH	-0.35	-0.27

Table 5. LE3 data used to estimate changes in fiberboard radial thermal conductivity

Time at Temp. (weeks)	Mean Fiberboard Temperature*, T (ºF)	Avg Fiberboard Radial Temp. Gradient*, ΔT (ºF)	Fiberboard Radial Thickness*, t (inch)	ΔT / t (ºF/inch)	Normalized Thermal Conductivity*, k_i/k_1
25.6	142.5	32.9	4.756	6.917	1
33.3	142.6	32.6	4.756	6.855	1.009
35.9	143.1	32.4	4.756	6.813	1.016
64.8	142.1	31.8	4.754	6.689	1.034
74.2	145	31.6	4.753	6.648	1.042
82	142.6	32.5	4.753	6.838	1.012
106.5	137.9	32.5	4.751	6.840	1.008
120.6	141.3	32.6	4.750	6.862	1.007
131.3	142.5	33.2	4.750	6.990	0.990
136.4	143	33.5	4.749	7.053	0.981
149.5	146.6	31.7	4.749	6.676	1.039
165.6	142.1	32.8	4.748	6.909	1.001
174.6	142	33.7	4.747	7.099	0.974
189.3	142.5	33.8	4.746	7.121	0.971
203.8	144.2	32.2	4.745	6.786	1.021
212.4	144	32.1	4.745	6.765	1.023
230.7	143.6	33.1	4.744	6.978	0.992
243.2	145.3	31.8	4.743	6.705	1.034
273.6	143.7	33.1	4.741	6.982	0.992
281.6	144.2	32.7	4.740	6.898	1.004
292.5	138.7	32.8	4.740	6.920	0.997
299.6	138.4	32.7	4.739	6.900	1.000
320.4	138.7	32.4	4.738	6.838	1.009
347	140	33.1	4.736	6.988	0.988
361.3	139.4	32.9	4.735	6.948	0.993
375.4	147.9	32.8	4.735	6.928	1.002
385.9	146	33.8	4.734	7.140	0.971
399	144.1	33.2	4.733	7.014	0.987

* Mean fiberboard temp. is the average of 4 consecutive measurements, taken at ~8 hr intervals.

Temp. gradient = (avg 2 upper ID readings – avg 2 upper OD readings), averaged over 4 consecutive measurement intervals.

Radial thickness = 4.578” – (6.23 E-5 “/wk * # weeks at temp)

Normalized thermal conductivity, with correction for mean temperature, is given by:

$k_i/k_1 = (\Delta T / t)_i / (\Delta T / t)_1 * 1.0007^{(T_i - T_1)}$

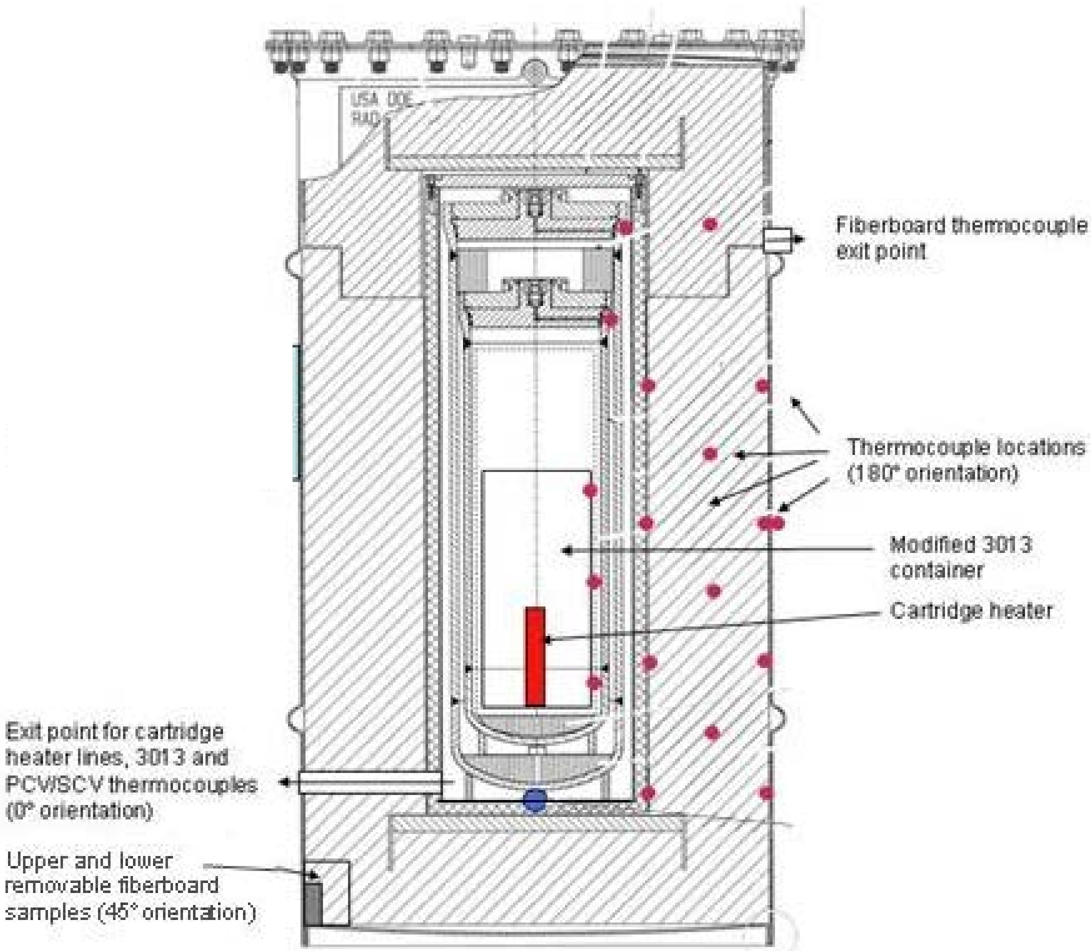
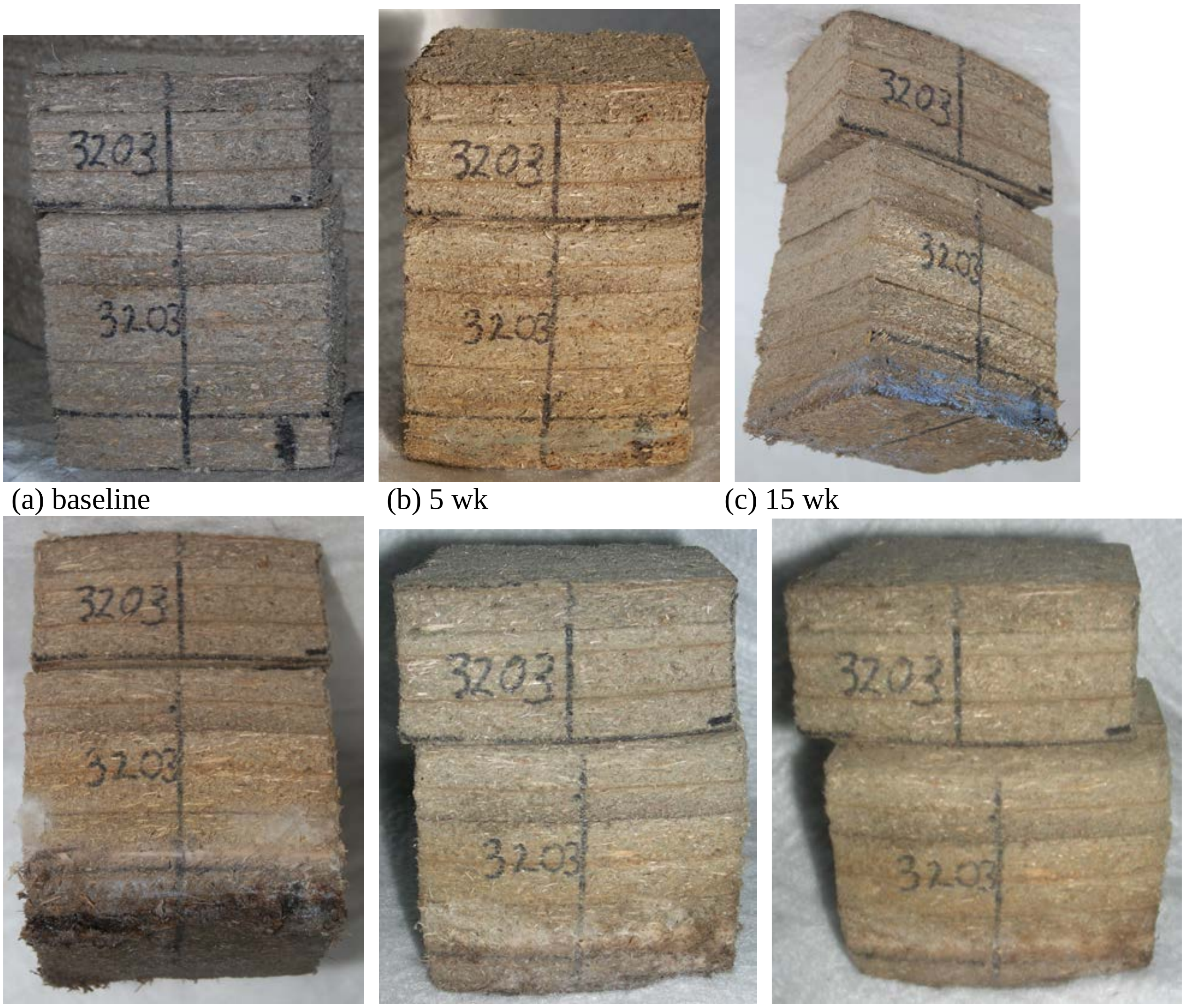


Figure 1. Cross section of 9975 package, showing added instrumentation and examination features.

(a) (b)

Figure 2. Configuration of LE3 to provide external heat source. In (a) LE3 is seen with insulation on top, inside the modified 55-gal drum. In (b), the 55-gal drum is wrapped with a drum heater and insulation.

Figure 3. Liquid condensate on the underside of the drum lid and on the air shield, observed after 2 weeks in test.



(a) baseline (b) 5 wk (c) 15 wk

(d) 45 wk (e) 151 wk (f) 418 wk

Figure 4. Sequence of photographs of small removable fiberboard sections from test LE3.

(a) Baseline

(b) 26 weeks

(c) 109 weeks

(d) 418 weeks

(e) 109 weeks

Figure 5. Progression of lead shield corrosion. Corrosion was uniformly heavy by week 26, and the upper portion (adjacent to the upper fiberboard assembly) gradually darkened somewhat by week 109. Very little change has been observed since that time. The corrosion thickness on the bottom decreases toward the center (e).

Figure 6. Corrosion of the drum interior along the bottom crevice, observed after 109 weeks in test.


(a)


(b)

Figure 7. Corrosion of the drum exterior at a stitch weld along the bottom edge, observed after 119 weeks in test (a). After 151 weeks in test, some additional corrosion is observed at this location (b).

Figure 8. Corrosion / staining of the drum interior at the upper flange stitch welds, observed after 190 weeks in test.

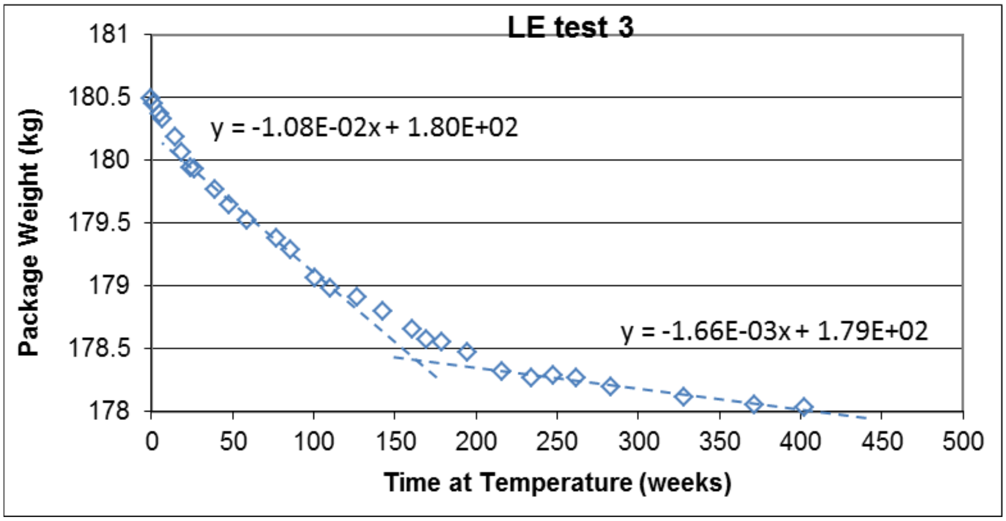


Figure 9. Package weight of LE3 over time. The trend lines illustrate two regions of relatively constant weight loss.

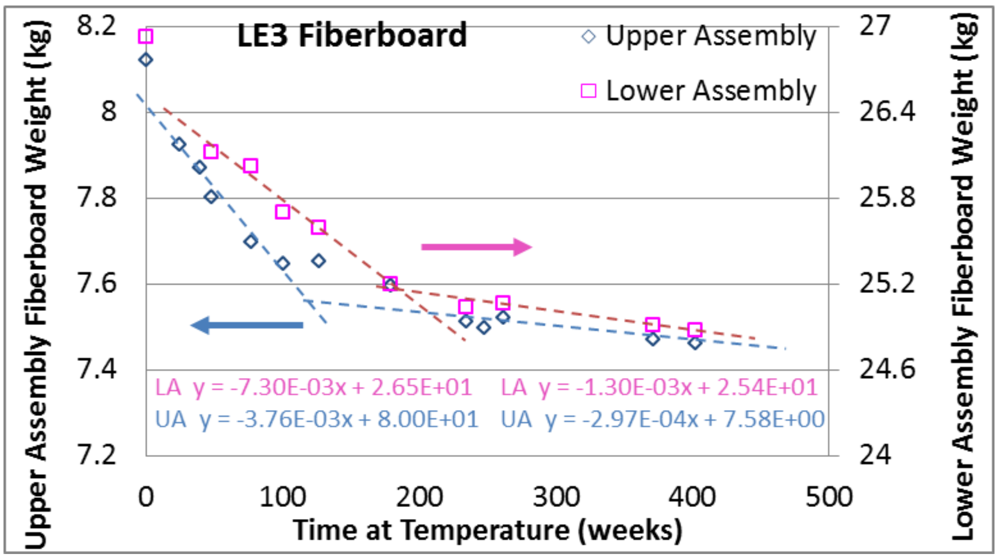


Figure 10. Weight loss of LE3 upper and lower fiberboard assemblies. The nominal weight of bearing plates and the air shield were subtracted from the recorded weight to give the weight of fiberboard only. The trend lines illustrate two regions of relatively constant weight loss for each assembly.

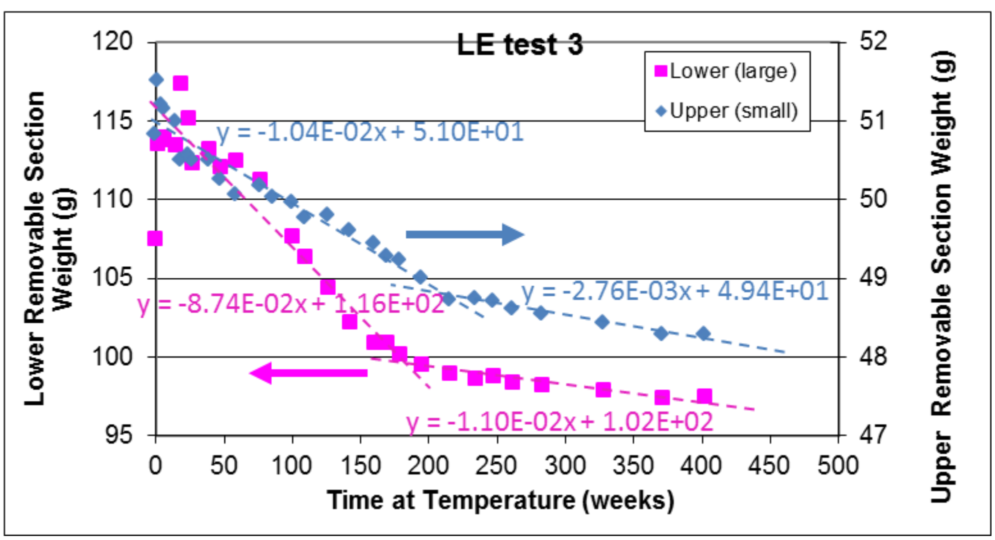


Figure 11. Normalized weight loss of LE3 removable fiberboard sections. The trend lines illustrate two regions of relatively constant weight loss for each section.

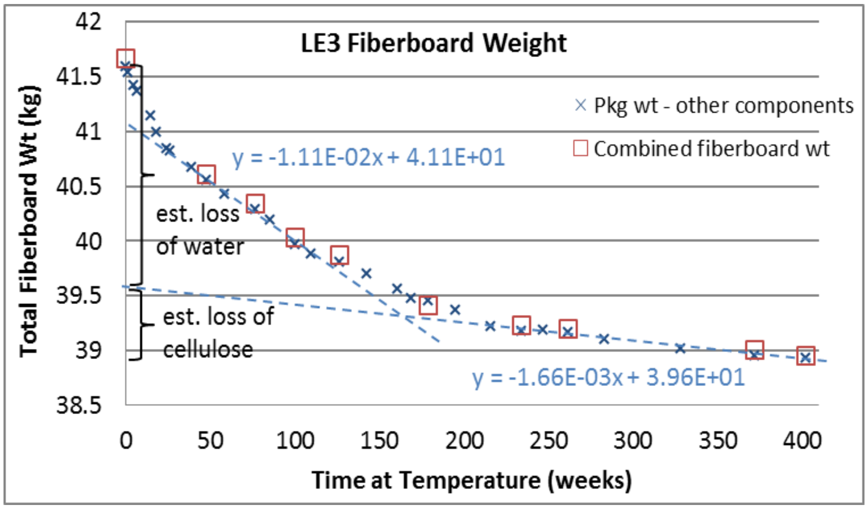


Figure 12. Variation in total fiberboard weight over time based on two methods. The combined fiberboard weight (red squares) is the total weight of all fiberboard sections. The blue "x"s show the difference between the total package weight and all non-fiberboard components.

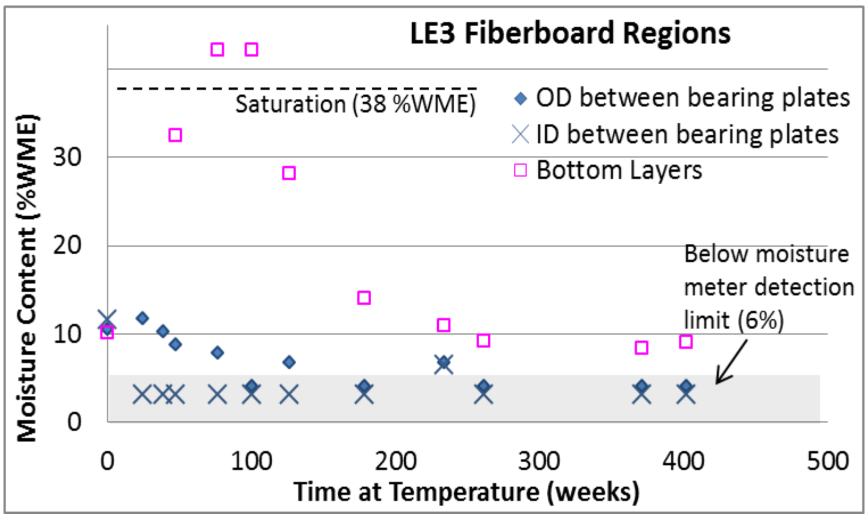


Figure 13. Moisture content history of three fiberboard regions. The regions between the bearing plates include portions of the upper and lower fiberboard assemblies. Values below the moisture meter detection limit (gray box) were assigned an arbitrarily value for illustration purposes. Similarly, the values above saturation were assigned an arbitrary value since the WME scale becomes non-linear above saturation.

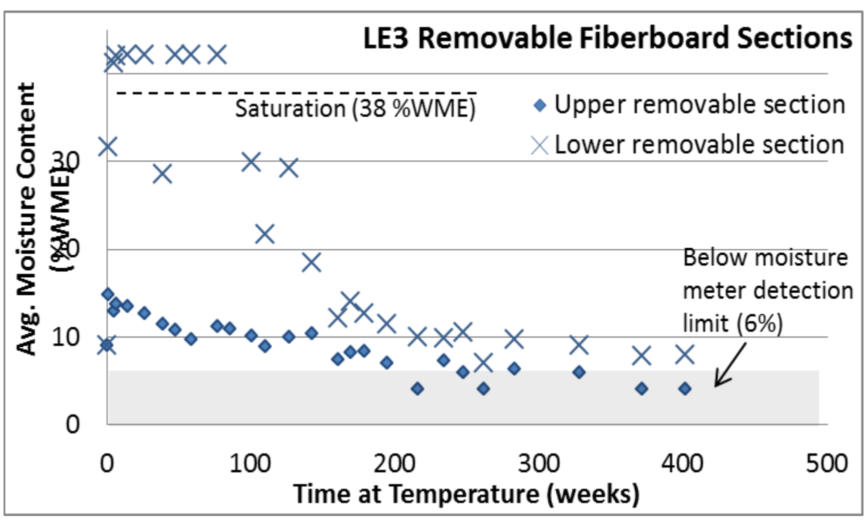


Figure 14. Average moisture content history of the two removable sections within the lower fiberboard assembly. Values below the moisture meter detection limit (gray box) were assigned an arbitrarily value for illustration purposes. Similarly, the values above saturation were assigned an arbitrary value since the WME scale becomes non-linear above saturation.

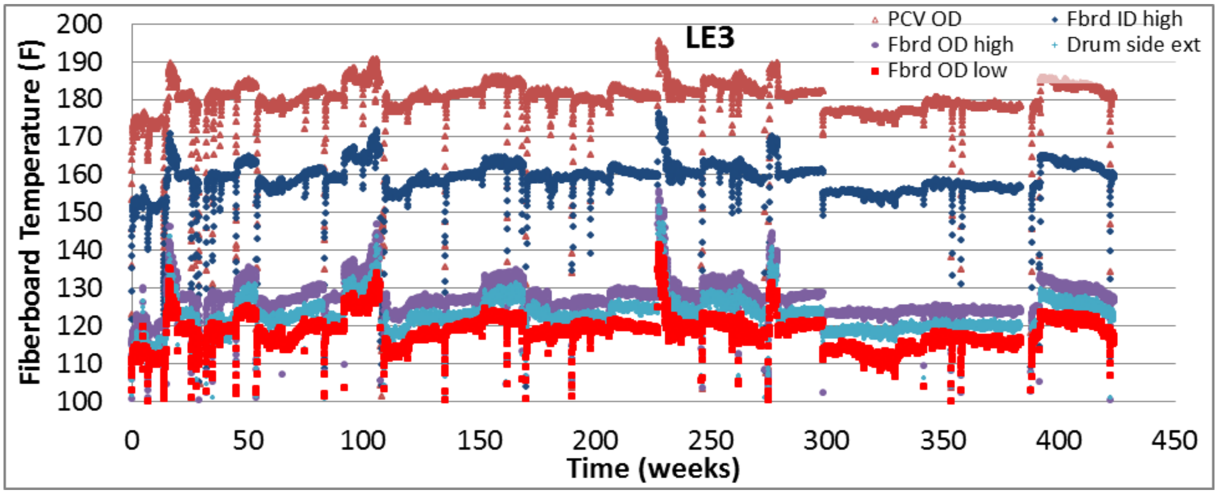


Figure 15. Temperature history for package LE3 at several locations, including the PCV OD surface, fiberboard ID and OD and the drum exterior.

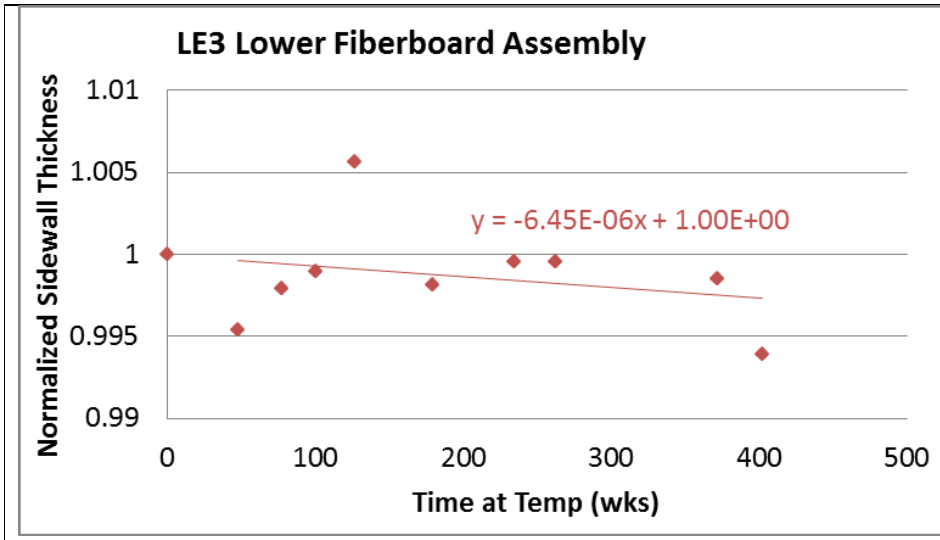
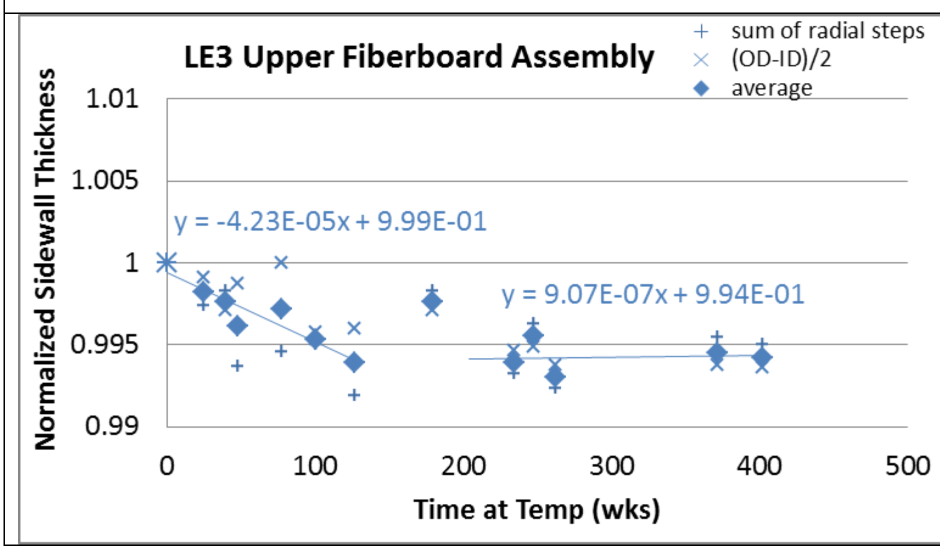


Figure 16. Normalized radial thickness of the fiberboard assemblies.

(a) LE3 lower assembly. Radial thickness is calculated by adding the thickness of the two radial steps on the assembly



(b) LE3 upper assembly. Radial thickness is calculated by adding the thickness of the two radial steps on the assembly, or by subtracting the inner and outer diameters. The trend line is shown for an average of these two calculations.

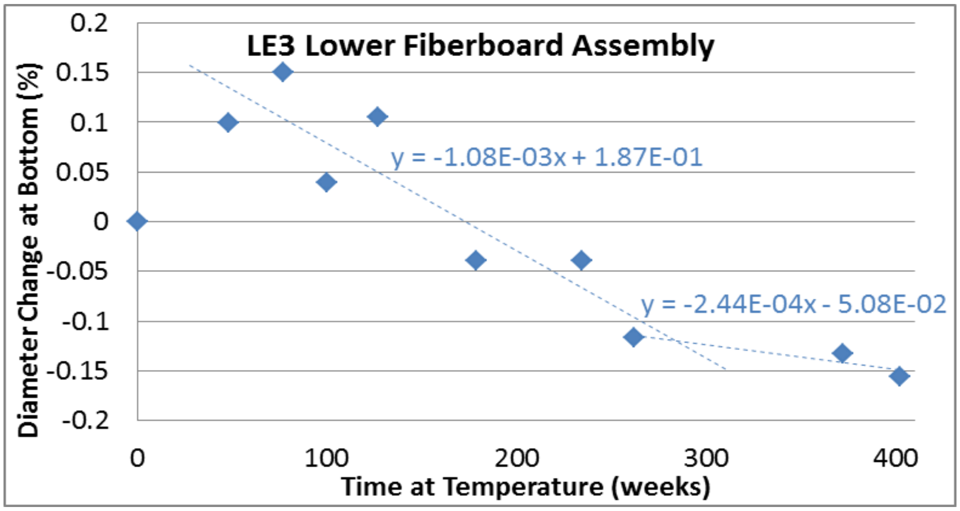


Figure 17. Change in diameter of the bottom layers of the LE3 lower fiberboard assembly. The trend lines illustrate two regions of relatively constant rate of diameter decrease.

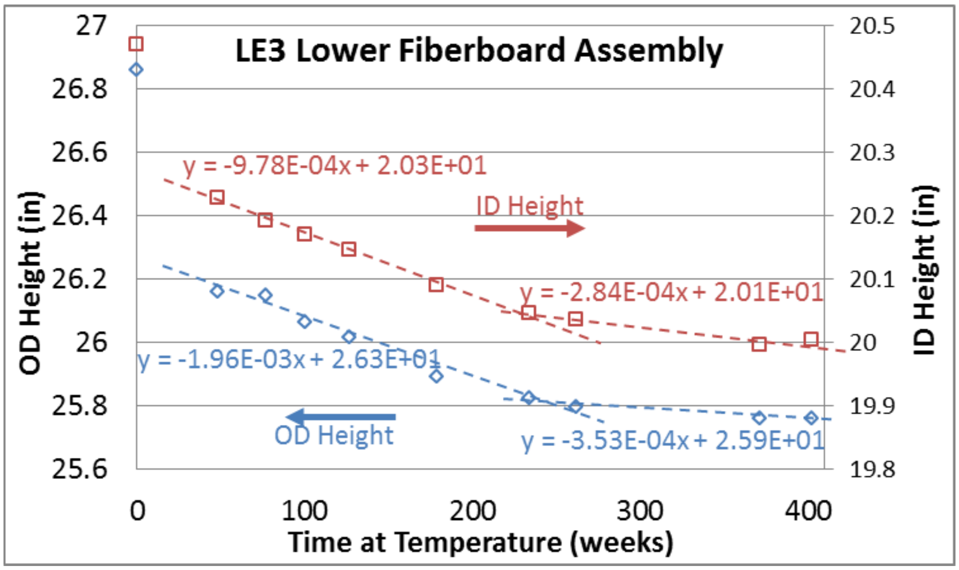


Figure 18. Change in height along the OD and ID of the LE3 lower fiberboard assembly. The trend lines illustrate two regions of relatively constant rate of height decrease for each dimension.

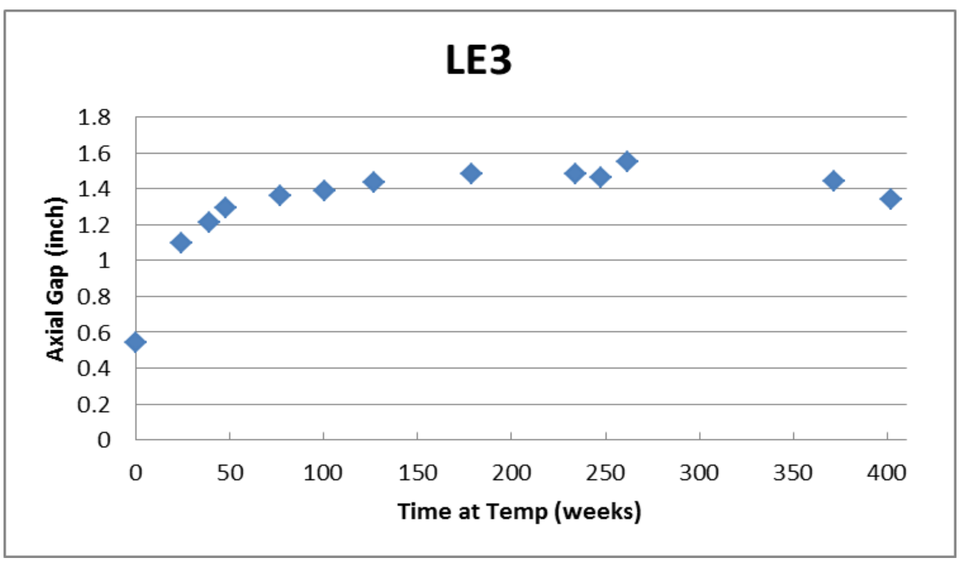


Figure 19. LE3 axial gap change over time. Interpolation indicates the axial gap exceeded 1 inch at ~20 weeks

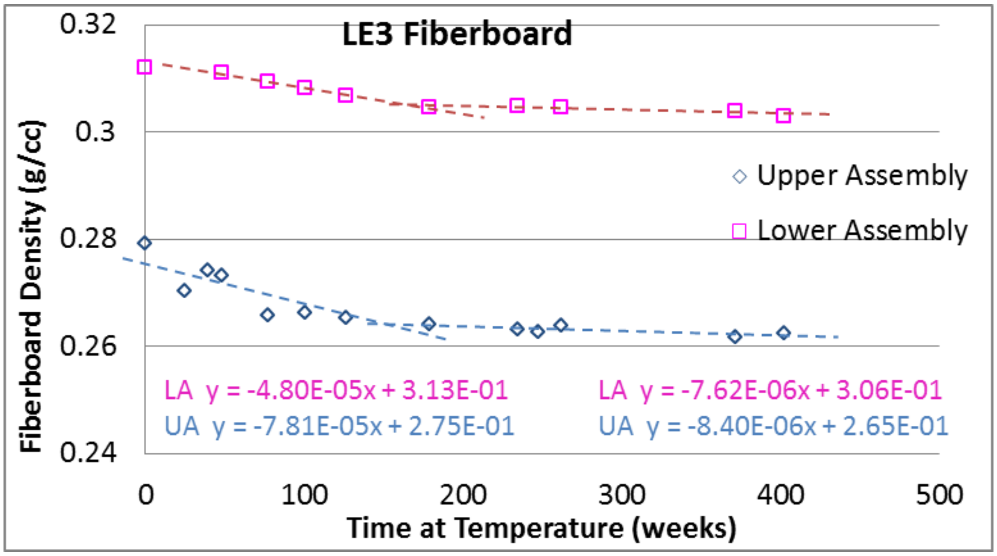


Figure 20. Density of LE3 upper and lower fiberboard assemblies. The trend lines illustrate two regions of relatively constant density change for each assembly.

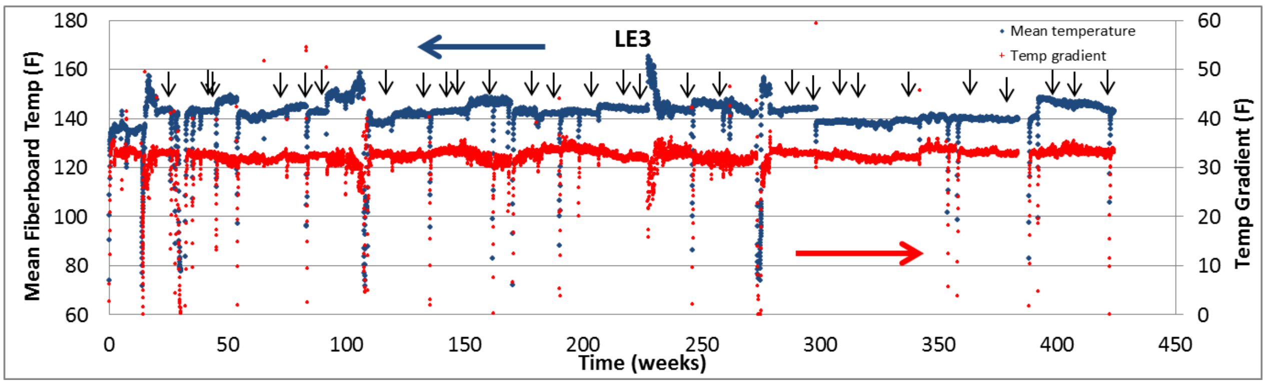


Figure 21. LE3 average radial temperature gradient (red symbols) in the fiberboard based on the upper 2 fiberboard ID thermocouples and the upper 2 fiberboard OD thermocouples, and average fiberboard temperature (blue symbols). The vertical arrows indicate times during steady state operation for which the relative thermal conductivity of the fiberboard was estimated.

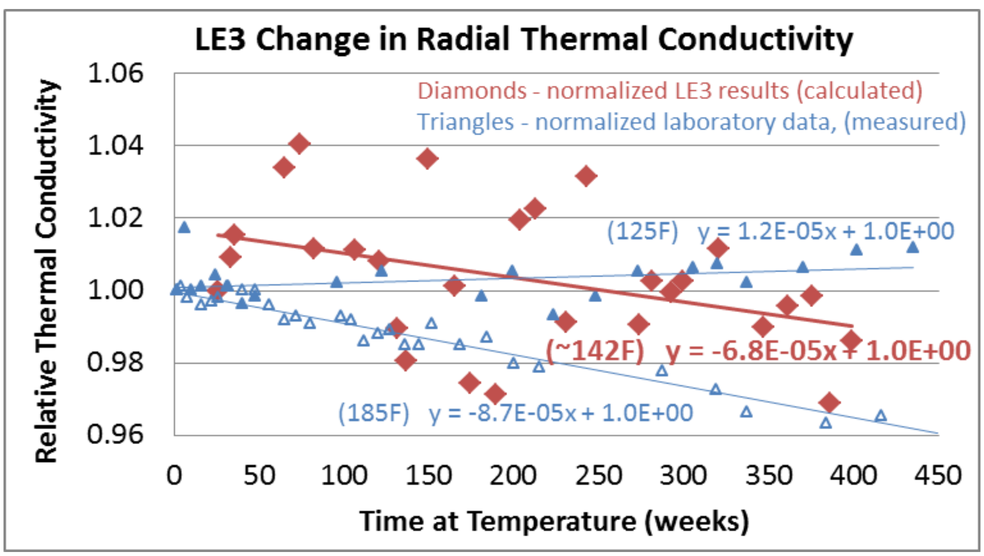


Figure 22. Relative change in radial thermal conductivity estimated from LE3 thermal gradient data. Comparable data for laboratory samples MSC-5R (conditioned at 125 ºF) and TC-3R (conditioned at 185 ºF) are also shown. The mean test temperature for the laboratory samples was 122 ºF.

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