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EXAMINATION OF SHIPPING PACKAGES 9975-02694 AND 9975-02729

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November 2016

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Summary

SRNL has assisted in the examination of two 9975 shipping packages (9975-02694 and 9975-02729) following their use for storage of nuclear material in K-Area Complex (KAC). Both of these were targeted for examination because they were exposed to water from a leaking expansion joint during storage. When first opened in KAC, the axial gap was found to be in excess of the 1 inch maximum criterion, signaling the potential for degradation of the fiberboard overpack and drum. Within each package the highest fiberboard moisture levels were observed in the bottom layers, but no mold was observed in either package. The fiberboard in each package appeared to retain good integrity consistent with non-degraded material. Minor corrosion was also observed on these drums along the lower stitch welds and on several closure bolts.

Background

KAC identified in November 2015 that several 9975 shipping packages in storage were exposed to water from a leaking building expansion joint. This condition likely existed for an extended duration based on the amount of debris and staining observed on top of the drum. Two of these packages were opened in KAC for examination on September 21, 2016. Both of these packages had an axial gap (the distance from the top of the drum flange down to the top of the air shield on the upper fiberboard assembly) in excess of the 1 inch maximum criterion. This condition often accompanies elevated moisture levels in the package, with potential degradation of the fiberboard overpack assembly and/or drum. Accordingly, these two packages were sent to SRNL for more detailed examination. Additional observations made in KAC include water stains and minor corrosion on the drum exterior surfaces.

Both packages were loaded at Hanford in October 2007, and both have been in storage in KAC since receipt in December 2007. Package 9975-02694 had a 15.3 watt internal heat load, while package 9975-02729 had a 14.2 watt internal heat load.

Both packages were transferred to SRNL High Pressure Lab (Building 723-15A) on October 11, 2016, and opened and examined on October 17, 2016 by Materials Science & Technology personnel with assistance from High Pressure Lab personnel. This report documents the SRNL examination of these two packages.

Examination Results

The SRNL examination included complete removal of the shield and lower fiberboard assembly, enabling additional measurements and observations that were not possible in KAC. Measurements (dimensions and moisture content) and observations taken on the fiberboard assembly from each package are summarized, and compared to the field surveillance data collected in KAC, in Tables 1 - 2. For each package, the axial gap was slightly larger when measured in SRNL than it was in KAC. It is expected that additional compaction of the bottom fiberboard layers occurred during handling and transport.

Visual observations from 9975-02694 include:

- Water stains on one side of the drum exterior, and corrosion of several closure bolts were observed * (Figure 1a).

- Subsequent light cleaning (with a wire brush) of the closure bolts removed much of the corrosion on the threads, showing the corrosion was superficial (Figure 2a).
- Areas around the lower stitch welds on the drum OD surface were lightly corroded and/or stained (Figure 3a, b).
- An unusual yellow / brown color was mixed in with the normally white shield corrosion layer (Figure 4). There are also several nodules protruding from the side of the shield.
- The middle region of the drum bottom steps down below the outer ~2 inch wide ring. The fiberboard bottom surface was smooth where it rested on this outer ring, but not in the middle region, indicating compaction of the bottom fiberboard layer was limited (Figure 5a).
- The fiberboard had a moderate degree of mustiness, but no mold was observed. There were no significant stains / water marks on the fiberboard.

Visual observations from 9975-02729 include:

- Water stains on one side of the drum exterior, and corrosion of several closure bolts were observed * (Figure 1b).
- Subsequent light cleaning (with a wire brush) of the closure bolts removed much of the corrosion on the threads, showing the corrosion was superficial (Figure 2b).
- Areas around the lower stitch welds on the drum OD surface were lightly corroded and/or stained * (Figure 3c, d).
- The fiberboard had a very slight musty odor, but no mold was observed. Several small, dark stains were observed on the upper fiberboard assembly * (Figure 6).
- The middle region of the drum bottom steps down below the outer ~2 inch wide ring. The fiberboard bottom surface was smooth where it rested on this outer ring, but not in the middle region, indicating compaction of the bottom fiberboard layer was limited (Figure 5b).
- Several water spot stains were observed on the drum interior (Figure 7).

The observations above marked with “*” were also noted in the field surveillance.

Discussion

Given the exposure of these packages to water, and the initial observation of an axial gap exceeding the 1 inch maximum criterion, the examinations in SRNL focused on the fiberboard assembly and drum. An excessive axial gap is often associated with the concentration of water in regions of the fiberboard assembly, especially in the bottom layers, with impacts on the integrity of the fiberboard and drum.

For most packages, significant changes on the overall fiberboard moisture content appear primarily in the bottom-most fiberboard layers. Both 9975-02694 and 9975-02729 had elevated moisture content in the bottom layers (up to 26.8 and 15.4 % wood moisture equivalent, or % WME, respectively), but both were below saturation (~38 %WME for cane fiberboard) at the time of measurement. While it is expected that these peak moisture content values have decreased in the nearly 4 weeks since the packages were unloaded in KAC, it is judged unlikely that the fiberboard had previously been saturated, based on the following:

- The absence of staining on the lower assembly indicates no liquid was present at any time, and
- Had the bottom layers become saturated at any time, they would have been weakened and compacted to a greater degree than observed [1] (since the central region on the bottom of the

fiberboard assembly was not compressed, total compaction at the very bottom was less than the ~0.2 inch step in the drum bottom).

Drawing R-R2-F-0025 [2] recognizes that the axial gap dimension may vary over time due to variation in the fiberboard properties. This same caveat extends to all fiberboard dimensions. Most of the measured fiberboard dimensions for these two packages are in reasonable agreement with nominal drawing dimensions, but one falls significantly outside the drawing tolerances - the lower assembly overall height (0.26 and 0.29 inch below the minimum value). With the overall lower assembly height reduced, but the lower assembly height above the bearing plate much closer to nominal, the majority of height reduction likely occurred in the layers below the bearing plate, where the moisture levels are highest and the fiberboard experiences the greatest compressive stress from the weight of the shield and containment vessels.

The shield and lower fiberboard assembly were easily removed from both packages, indicating some gap existed between components. No physical damage to the fiberboard was observed, and the layers remained well-bonded. Aside from a few small darkened areas on the 9975-02729 upper fiberboard assembly, no stains or discoloration were noted. The fiberboard assemblies had a solid feel and appearance, consistent with non-degraded material.

The observed corrosion on the drums is limited to the stitch weld area along the bottom edge. Any moisture in the fiberboard near the bottom of the drum can migrate through the bottom fold. Residual stresses around the ends of the stitch welds would tend to make these areas slightly easier leak locations, allowing chlorides or other corrosive species to deposit on the OD surface. Combined with the altered microstructure in the weld and heat-affected zone, these areas can be susceptible to corrosion if significant moisture moves through the drum. Corrosion in this area has been observed in previous packages to similar and greater degrees [3-5]. Alternatively, given the presence of water on the drum exterior from the expansion joint leak, the bottom lip on the drum provides a crevice that can trap and retain moisture and dissolved compounds running down the side of the package, leading to corrosion.

In general, higher internal heat loads will produce higher internal temperatures and higher temperature gradients across the fiberboard. The thermal gradient in turn will create a counter-gradient in the fiberboard moisture content and relative humidity, although the absolute humidity tends to remain relatively constant [6]. Since the lower fiberboard assembly remains in the drum during field surveillance, moisture measurements are only made on the upper fiberboard assembly and the top surface of the lower fiberboard assembly. It is worth considering whether field surveillance measurements of fiberboard moisture content within the upper fiberboard assembly might indicate the moisture condition at the bottom of the package. Prior field surveillance data from KAC can be used to approximate the thermal and moisture conditions within the upper fiberboard assembly.

The thermal gradient across the fiberboard sidewall is approximated by the difference between the shield lid temperature and the drum OD temperature. The average upper fiberboard assembly moisture content is approximated by averaging the average OD moisture content and the average ID moisture content. These two parameters are compared in Figure 8 for a sampling of 30 surveillance packages with cane fiberboard.

Similarly, the moisture gradient across the upper fiberboard assembly is approximated by the difference between the average OD moisture content and the average ID moisture content. These data are shown in Figure 9 as a function of the thermal gradient.

In Figures 8 and 9, packages with an axial gap greater than the 1 inch maximum criterion are shown with solid symbols, while packages that meet this criterion are shown with open symbols. In addition, the two packages documented in this report are highlighted with red.

Several observations can be drawn from these comparisons, including:

- The average moisture content of the upper fiberboard assembly (Figure 8) does not vary with internal heat load. However, with average moisture content values ranging from 7.8 to 14.0 %WME, there is significant scatter in moisture content from package to package.
- The moisture gradient of the upper fiberboard assembly (Figure 9) varies with internal heat load, and a similar degree of scatter between packages is seen in these data.
- The average moisture content of the upper fiberboard assembly is an average of 10.4 %WME for the 17 packages with an axial gap < 1 inch, and 10.8 %WME for the 13 packages with an axial gap > 1 inch.

Moisture data are also available for six packages that were opened for reasons other than surveillance, and found to have an axial gap > 1 inch. Two of these packages (9975-01818 and -01819) experienced significant water intrusion, and had much higher upper assembly average moisture content (20.8 and 17.5 %WME). The other four packages had upper assembly average moisture content comparable to the surveillance packages (7.8 to 14.8 %WME). With the exception of the two packages with significant water intrusion, variation in the upper fiberboard assembly moisture content does not correlate well with axial gap, given the package-to-package variation in initial axial gap and overall moisture content.

Since the lower fiberboard assembly is not removed from the drum during field surveillance, there are fewer packages for which peak moisture data in the lower assembly are available. Figure 10 compares the lower assembly peak moisture values with the upper assembly OD average moisture. This shows good correlation for packages that remain below saturation. However, those packages in which the bottom fiberboard layers exceed saturation are not predicted by this correlation.

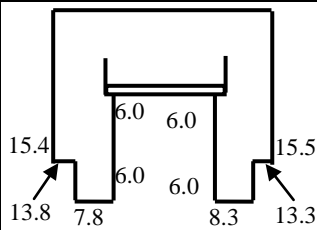
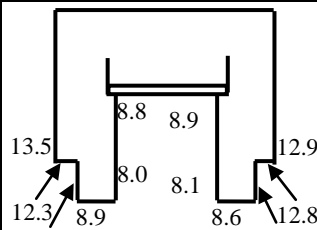
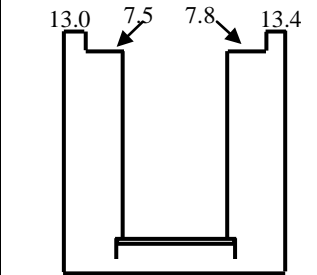
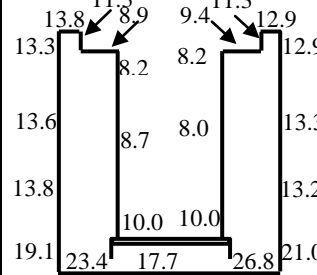
Conclusions

SRNL has assisted in the examination of two 9975 shipping packages following storage of nuclear material in K-Area Complex (KAC). These packages had been stored in a location subject to leaks from a building expansion joint. The initial observation of the axial gap exceeding the 1 inch maximum criterion signaled the potential for further degradation of the fiberboard and drum. Each package experienced a degree of compaction of the bottom fiberboard layers, and had elevated moisture levels toward the bottom, but the fiberboard appeared to retain good integrity consistent with non-degraded material. Minor corrosion was observed on these drums, and might have resulted from moisture leaking out of the drum or water running down the drum exterior..

References

1. SRNL-STI-2012-00429, "Analysis of the Axial Gap vs Fiberboard Moisture Content in a 9975 Shipping Package", W. L. Daugherty, September 2013
2. Drawing R-R2-F-0025, Rev. 2, "9975 Drum with Flange Closure Subassembly and Details", October 29, 2003
3. SRNL-STI-2016-00014, "Examination of Shipping Packages 9975-01641, 9975-01692, 9975-03373, 9975-02101 and 9975-02713", W. L. Daugherty, January 2016
4. SRNL-STI-2016-00152, "Examination of Shipping Package 9975-02403", W. L. Daugherty, March 2016
5. SRNL-STI-2016-00209, "Destructive Examination of Shipping Package 9975-02101", W. L. Daugherty, May 2016
6. SRNL-STI-2016-00254, "Humidity Data for 9975 Shipping Packages with Cane Fiberboard", W. L. Daugherty, May 2016
7. SRNL-L7200-2008-00007, "Correlation between Cane Fiberboard Moisture Content and Relative Humidity", W.L. Daugherty, December 10, 2008

Table 1. Detailed fiberboard data for packages 9975-02694 (15.3 watt internal heat load)

	9975-02694 (in KAC) 9/21/2016		9975-02694 (in SRNL) 10/17/2016	
Upper assembly	Dimensions (inch)	Moisture content (% WME)	Dimensions (inch)	Moisture content (% WME)
UD1	17.586		17.594	
UD2	8.546		8.574	
UR1			3.032	
UR2			1.474	
UH1			7.118	
UH2			2.083	
UH3	4.952		4.980	
Lower assembly	Dimensions (inch)	Moisture content (% WME)	Dimensions (inch)	Moisture content (% WME)
LD1			18.028	
LD2			8.490	
LR1			3.277	
LR2			1.509	
LH1			26.358	
LH2			20.368	
LH3	2.003		2.039	
Axial gap	1.220 inch		1.266 inch	
Notes	94.5 %RH in upper air space.		50.2 %RH at 75.3 °F in upper air space.	

Diametral dimensions were measured twice, ~180 degrees apart, other dimensions were measured 4 times, ~90 degrees apart. Average values are reported.

Dimension UH1 includes the air shield.

Fiberboard moisture content is measured with a GE Protimeter Surveymaster wood moisture meter. Conversion of its results (% WME) to wt% water has been described for cane fiberboard in Reference 7.

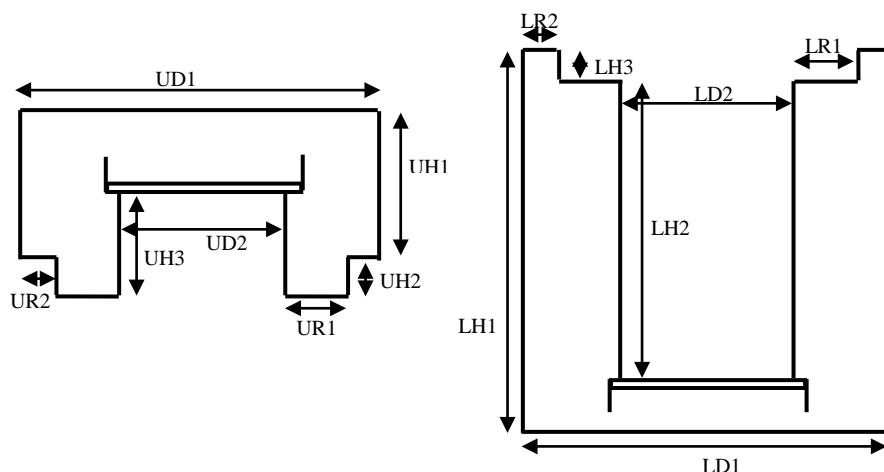
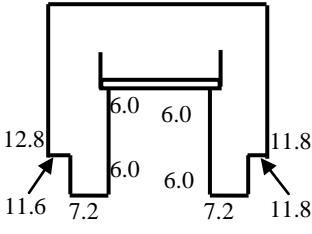
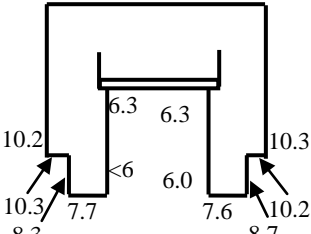
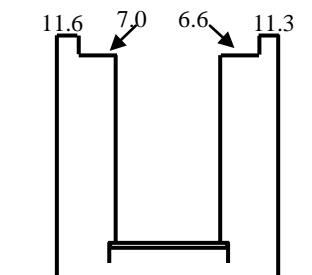
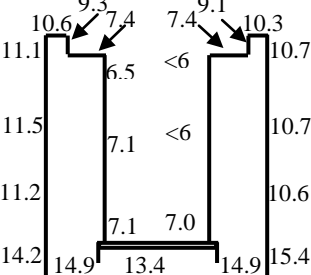


Table 2. Detailed fiberboard data for package 9975-02729 (14.2 watt internal heat load)

	9975-02729 (in KAC) 9/21/2016		9975-02729 (in SRNL) 10/17/2016	
Upper assembly	Dimensions (inch)	Moisture content (% WME)	Dimensions (inch)	Moisture content (% WME)
UD1	17.580		17.616	
UD2	8.586		8.616	
UR1			2.993	
UR2			1.550	
UH1			7.017	
UH2			2.129	
UH3	4.918		4.951	
Lower assembly	Dimensions (inch)	Moisture content (% WME)	Dimensions (inch)	Moisture content (% WME)
LD1			18.036	
LD2			8.444	
LR1			3.254	
LR2			1.530	
LH1			26.391	
LH2			20.313	
LH3	2.002		2.023	
Axial gap		1.304 inch		1.396 inch
Notes	91.8 % RH in upper air space		40.5 %RH at 74.9 °F in upper air space.	

Diametral dimensions were measured twice, ~180 degrees apart, other dimensions were measured 4 times, ~90 degrees apart. Average values are reported.

Refer to Table 1 to identify dimensions. Dimension UH1 includes the air shield.



(a) 9975-02694



(b) 9975-02729

Figure 1. Water stains on the side of the drum, and corrosion on several closure bolts.



(a) 9975-02694



(b) 9975-02729

Figure 2. Detail of several closure bolts after light cleaning (with wire brush). The superficial corrosion in the thread area has been mostly removed, but remains on the end and under the head.



(a) 9975-02694



(b) 9975-02694



(c) 9975-02729



(d) 9975-02729

Figure 3. Light corrosion / staining on drum OD surface at the stitch welds.

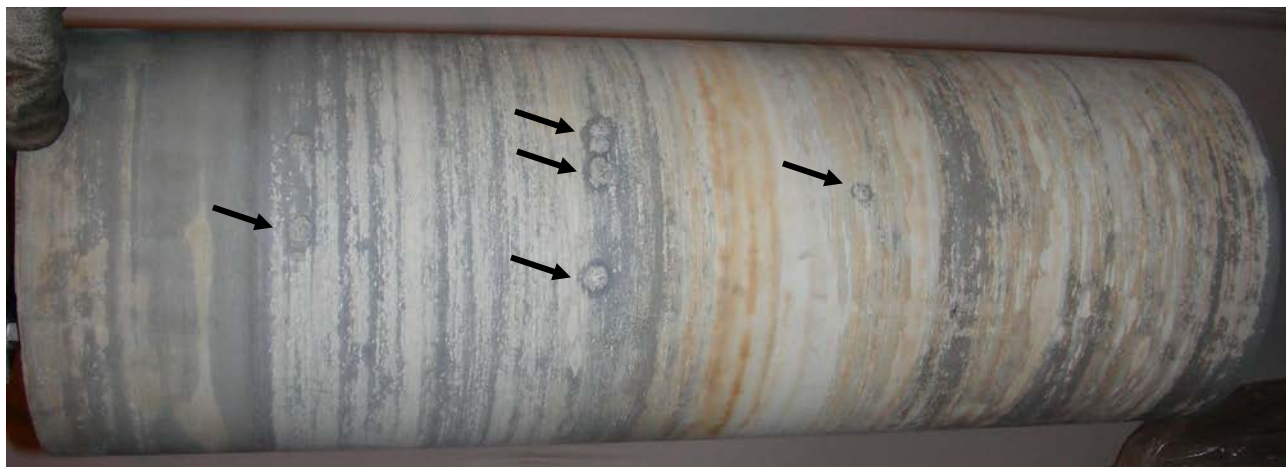


Figure 4. Unusual coloration of the shield corrosion product, and several nodules protruding from the side of the 9975-02694 shield (at arrows).



(a) 9975-02694



(b) 9975-02729

Figure 5. Lower fiberboard assemblies (upside down) showing compaction only in the outer ~2 inches.



Figure 6. Stains on the 9975-02729 upper fiberboard assembly



Figure 7. Water spot stains on the 9975-02729 drum interior.

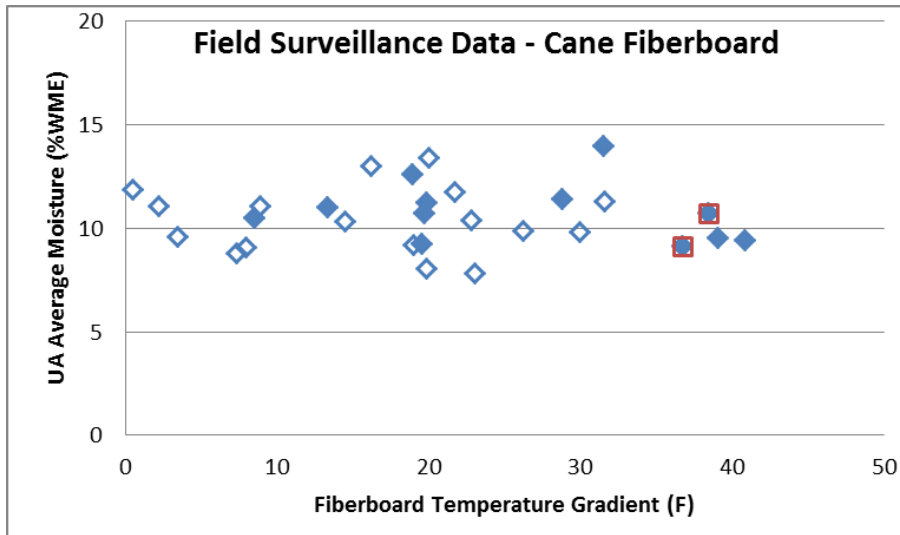


Figure 8. Comparison of average moisture content within the upper fiberboard assembly to the temperature gradient based on KAC field surveillance data of packages with cane fiberboard. The highlighted data are from 9975-02694 and 9975-02729. Packages with an axial gap > 1" are shown with solid symbols.

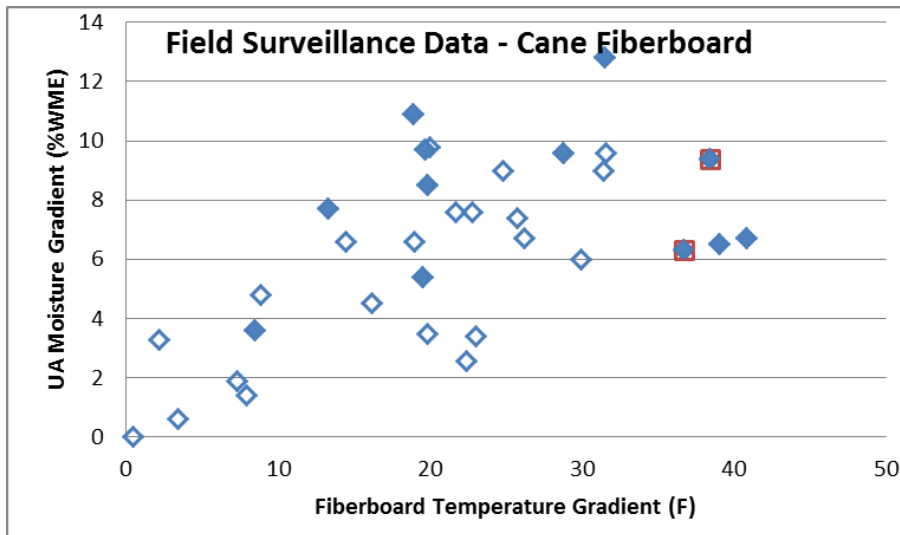


Figure 9. Comparison of moisture gradient across the upper fiberboard assembly to the temperature gradient based on KAC field surveillance data of packages with cane fiberboard. The highlighted data are from 9975-02694 and 9975-02729. Packages with an axial gap > 1" are shown with solid symbols.

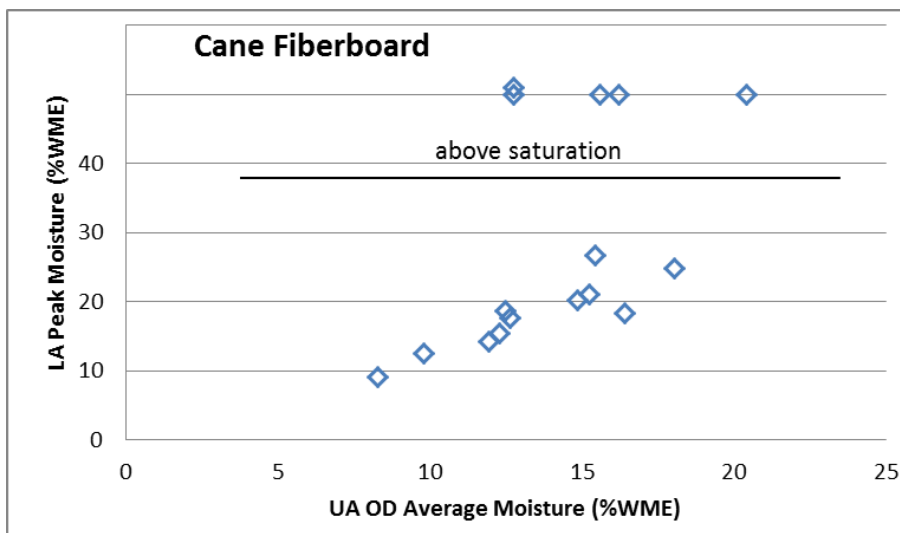


Figure 10. Peak moisture measurement in the lower fiberboard assembly vs average moisture content of the upper assembly OD surface. Data are from cane fiberboard assemblies measured in KAC and SRNL.

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