### **EXAMINATION OF FIBERBOARD FROM SHIPPING PACKAGE 9975-01819**

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## **EXAMINATION OF FIBERBOARD FROM SHIPPING PACKAGE 9975-01819 (U)**

# **Revision Log**



## **Summary**

Upon opening package 9975-01819 following approximately 5.5 years storage in KAMS, it was observed that the fiberboard was moldy, and the total height of the fiberboard assemblies was less than normal. Observations and measurements have since been made on three subsequent occasions. The available information indicates that the package contained approximately 2.5 liters of water in excess of what would normally exist within the fiberboard. This excess moisture led to a significant loss of fiberboard strength, the subsequent compression of the bottom layers, and the growth of mold observed on both the upper and lower fiberboard assemblies.

In its current state, the fiberboard from this package retains a density (related to the criticality control function) within the range measured in other packages. The amount of excess moisture present is modest throughout most of the fiberboard, and its effect on thermal conductivity should be small. The thermal conductivity should increase significantly only near the bottom of the lower fiberboard assembly where the majority of excess moisture was found. The impact absorption capability is affected, and the ability of the fiberboard to perform this function in the current state must be evaluated. The longer such a condition persists, the greater the impact on fiberboard mechanical properties.

## **Background**

On January 26 2009, package 9975-01819 was opened in KIS for the first time since its receipt from RFETS in June of 2003. The package was opened for 3013 required Pu and U verification measurements. Mold was observed on the upper fiberboard assembly, and the distance from the drum flange to the top of the upper fiberboard assembly was greater than 1 inch (see Figures 1 and 2). These conditions are documented in nonconformance report 09-NCR-29-0001. Three follow-up examinations have been performed on the upper and lower fiberboard assemblies from this package. This memorandum documents the findings from those examinations.

Documentation for package 9975-01819 identifies the vendor verified fiberboard dimensions on February 1, 2003, and RFETS performed the loaded RCO survey on May 1, 2003. One additional package previously opened in KAMS (August 2007) was found to have moldy fiberboard, 9975-01710. The vendor verified the fiberboard dimensions for this previous package on December 10, 2002, and RFETS performed the loaded RCO survey on March 25, 2003. Other packages that were fabricated and loaded in this same time frame have been opened in KAMS, but did not show these same phenomena [1].

## **Examinations**

The first examination was performed on January 29, 2009. Personnel present included representatives from NMM, SRNL-SRPT, 9975 DA and SRNL-MS&T. At this time, the upper fiberboard assembly was removed from the drum for observation, but the lower fiberboard assembly remained in the drum, with the shield in place. Therefore, only the top surfaces of the lower assembly were accessible. Observations were documented by SRNL-SRPT [2], and are summarized as follows.

- Package exhibited a distinct musty, moldy odor.

- Upper fiberboard assembly has a thin coat of mold on the exterior surface.
- Accessible fiberboard (upper assembly and exposed top of lower assembly) was firm, resilient and intact.
- Moisture content of accessible fiberboard ranged from 12 to 22 %WME (wood moisture equivalent), compared with a more typical range of 8 to 16 %WME.
- Accessible dimensions are per design. The lower fiberboard assembly sits approximately 1 inch low in the drum. Suspect that this results from the bottom being too short or compressed.

A work order was initiated to remove the lower fiberboard assembly for further inspection. Both fiberboard assemblies had remained in the drum during the interim period. The second examination was performed on February 24, 2009. Personnel present included representatives from NMM, SRNL-SRPT and 9975 DA. During this examination, the lower fiberboard assembly was removed from the drum for closer examination. Observations were documented by SRNL-SRPT [3], and are summarized as follows (see Figures 3-5).

- Lower assembly was sitting 1 inch too low in the drum.
- The overall height of the lower assembly is 1 inch too short (26 vs 27 inches).
- The bottom 1.75 inches of the lower assembly has a moisture content of 100 %WME.
- At other locations that were measured, the remainder of the lower assembly has a moisture content of 20 to 26 %WME on the OD surface, and 16 %WME on the ID surface.
- Structural integrity of the bottom 2 inches of the lower assembly is very diminished, soft and wet to the touch. A liquid film / sheen would form on the surface with modest finger pressure.

The fiberboard assemblies were placed in plastic bags until further examinations could be performed. It was anticipated that they would be shipped to SRNL for more detailed testing. However, concerns that the fiberboard could be radiologically released in its condition (wet, moldy) led to performing a third examination in K Area instead. The third examination was intended to gather as much data as practical relative to the properties of the fiberboard assemblies. It was performed on March 10, 2009, with personnel from SRNL-MS&T, NMM, SRNL-SRPT and 9975 DA present. Data collected, and subsequent analyses are described below.

## **Data and Analysis from Third Examination**

It was reported shortly after the fiberboard assemblies were placed in bags (on 2/24/09) that condensation was observed inside the bags. No condensation was noted on 3/10/09. Upon removal from the bags on 3/10/2009, it was noted that the lower assembly did not appear as wet, nor did it have as significant a musty odor as before. Both the upper and lower assembly were weighed and measured. These data are summarized in Table 1. From these data, a density was calculated for the fiberboard in each assembly (correcting for the bearing plates and air shield using nominal dimensions for these components). The upper assembly fiberboard density is 0.289 g/cc, and the lower assembly fiberboard density is 0.287 g/cc. The density of fiberboard assemblies from destructively examined packages has ranged from 0.24 to 0.30 g/cc.

The moisture content of both assemblies was measured in a number of locations on 3/10/09. These values are summarized in Table 2. The height of the region at the bottom of the lower assembly with 100 %WME is smaller than noted in the 2/24/09 examination. This region extends for about 1 inch or less, whereas it was found to extend 1.75 inches in the earlier examination. At the same time, the recorded moisture content at higher elevations is about the same as previously recorded. This provides further evidence supporting the observation that the lower assembly appears somewhat drier than in the second examination. The lower fiberboard assembly, marked with results of moisture measurements, is shown in Figures 6-8.

Given the loss of moisture between the second and third examination, information from the second examination is used to estimate the total moisture content of the lower assembly. It is noted that the only significant difference between these two examinations is the moisture content of the bottom 1.75 inches. The moisture meter (which indicated 100 %WME) does not provide a linear response above ~40 %WME, and no reliable conversion exists for readings in this range. Therefore, an alternate approach was developed to estimate the actual moisture content of this region of the assembly. During the second examination, a liquid film / sheen was observed on the fiberboard under modest finger pressure. To help quantify the concentration of water this represented, a test section of fiberboard was placed in a beaker with a specific quantity of water. Upon absorption of this water, the sample contained  $~60$  wt% water. It did not release any water under finger pressure. Additional water was added to provide a total of  $\sim 83$  wt% water. In this condition, liquid was visible under modest pressure in the wetter areas of the sample, but not in the drier areas. It will be assumed based on this observation that the bottom region of the lower assembly held more than 60 wt% water, and probably held about 80 wt% water.

Details of the estimate of total moisture within the lower fiberboard assembly are provided in Attachment 1, along with the assumptions made. The total average moisture content of the lower assembly on 2/24/09 is estimated to be at least 22 wt%, with 24 wt% considered more realistic. Based on this estimate, the lower assembly contained approximately 2.5 liters of water more than would be expected in a typical assembly.

A qualitative test was performed to illustrate the extent to which the compressive strength has been compromised in the lower assembly. At several locations along the OD and bottom surfaces, each of 3 allen wrenches were pressed into the fiberboard with approximately the same force. Each impression on the side was located between glue joints. The wrenches were 4, 6 and 8 mm in size. See Figures 9 - 11. The depth of impression was recorded at each location, along with the moisture content. The same steps were performed on the OD surface of a section of fiberboard removed from a different assembly, for comparison. Results are summarized as follows:



Relative strength of lower fiberboard assembly as indicated by penetration of allen wrenches

\* At this location, the 4 mm wrench bottomed out against the handle. A longer wrench would have penetrated further.

\*\* On the bottom of the assembly, all 3 wrenches stopped at the first glue joint.

While these results cannot be converted directly into a measure of the fiberboard compressive strength, they do illustrate that significant loss of strength has occurred, especially where the moisture content is 30 %WME and greater.

## **Discussion**

Several changes in the fiberboard properties are expected as a result of increased moisture. The density will increase, the thermal conductivity and heat capacity will increase, and the compressive strength will decrease. Each of these changes should be evaluated for any impact on package performance in KAMS.

An increase in density should be favorable in terms of criticality control. The design limit for fiberboard density is 0.20 g/cc minimum [4]. The density calculated from the measured dimensions and weight (0.287 g/cc) exceeds this limit by a significant margin. In addition, Attachment 1 shows the estimated dry density (if no moisture were present) of the fiberboard in the lower assembly is 0.223 g/cc, and the estimated density of the fiberboard in the lower assembly with a typical moisture content and no compression of the lower layers is 0.251 g/cc.

The thermal properties of the fiberboard determine the internal package temperature during normal operation and accident (fire) scenarios. The variation of thermal conductivity of fiberboard with increasing moisture is expected to follow that of solid wood, which is illustrated in Figure 12 (taken from Reference 5). In normal operation (i.e. steady state), an increase in thermal conductivity (resulting from excess moisture) will decrease the internal package temperature, which will reduce the long-term aging rate of the package components (e.g. fiberboard and O-ring seals). However, during a fire scenario, an increased thermal conductivity will lead to a faster increase in internal component temperatures. This undesirable consequence will be at least partially offset by two related effects. As the fiberboard heats up, the excess moisture will be driven off. This will produce an evaporative cooling effect based on the heat capacity of the excess moisture. Once the moisture is driven off, the thermal conductivity will

decrease to more typical values. The net result of excess moisture on thermal properties may be small, but should be considered for impact on KAMS performance requirements.

Some of the potential for loss of compressive strength following exposure to excess moisture is seen in laboratory testing of samples conditioned at ambient temperature and  $\sim$ 100% relative humidity [6]. During a 2 week conditioning period, these samples gained an average of 15.8% in weight due to moisture absorption. If one assumes they started with a moisture content of 10 wt% (8 to 16% would be fairly typical), their moisture content after conditioning would be 27 wt%. A metric used to compare the compressive strength of fiberboard samples is the area under the stress-strain curve up to a strain of 40%. This metric is proportional to the energy absorbed by the sample. For three samples conditioned at ambient temperature and 100% relative humidity and compression tested in the parallel orientation (the primary direction of loading expected for a forklift impact scenario), the area under the curve to a strain of 40% ranged from 17 to 19 psi. In comparison, three samples conditioned at ambient temperature and ambient relative humidity and compression tested in the parallel orientation had an area under the curve to a strain of 40% ranging from 65 to 73 psi. This suggests that exposure to 100% relative humidity for 2 weeks led to a 75% decrease in the energy absorption capability of the fiberboard.

Calculations of the forklift impact event [7] show that the compressive strength of the fiberboard can be reduced to 20% of the nominal value before the package integrity will be compromised. For the nominal stress-strain curve used in this calculation, 20% of the area under the curve to a strain of 40% is 11 psi. While this value is exceeded for the samples tested after a 2 week conditioning period, it is unknown how much additional strength will be lost at this moisture content over longer periods and/or elevated temperatures.

The impression tests performed on the 9975-01819 lower fiberboard assembly show that significant loss of compression strength has occurred, even in the regions at  $\sim$ 20 %WME ( $\sim$ 16 wt% water). The same impressions were made on a control fiberboard section with a moisture content of 8.8 %WME. The area under the stress-strain curve to a strain of 40% for this control material was separately found to be 70 - 78 psi. The impression tests show increasing penetration (and therefore decreasing strength) with increasing moisture content.

When excess moisture is present, the highest moisture levels will tend to occur at the bottom of the package. This is due to the influence of gravity as well as the thermal gradient that will develop with an internal heat load which will tend to drive moisture to cooler regions of the package. In contrast, the failure condition for the forklift impact scenario occurs when the containment vessel seals are damaged. This is more likely with an impact closer to mid-height on the drum, away from the heaviest moisture concentration. The specific impact of reduced fiberboard strength on forklift impact and other physical challenges to the package should be evaluated by facility personnel.

An additional influence on package integrity that has not been evaluated is the effect of mold. In the earlier stages of mold growth, the presence of additional material (the mold structure itself) will tend to fill some of the available pores in the fiberboard, which may lead to modest increases in the compressive strength and thermal conductivity. At some point after significant growth occurs, the mold will begin to degrade the fiberboard fibers, and replace the cellulose structure

with decomposition by-products. The impact of this on strength and thermal properties is unknown, but it is suspected that the strength will decrease significantly (beyond the changes already described from excess moisture). Also unknown is the timeframe for such changes to occur.

## **Conclusions**

Based on measurements taken on the fiberboard assemblies from package 9975-01819, it appears that the package contained approximately 2.5 liters of water more than would normally be found within the fiberboard. This excess moisture led to a significant loss of fiberboard strength, especially in the bottom layers, and the subsequent compression of the bottom layers. This excess moisture also led to the growth of mold observed on both the upper and lower fiberboard assemblies.

In its current state, the fiberboard density and thermal conductivity are likely similar to that of other packages. The amount of excess moisture present is modest throughout most of the fiberboard, but is significant at the bottom of the lower assembly. The impact absorption capability is affected, and the ability of the fiberboard to perform this function in the current state must be evaluated. Certainly, the longer such a condition persists, the greater the impact on fiberboard mechanical properties as a result of chemical- and biological- (mold) induced degradation.

#### **References**

- 1. "Re: Fw: 9975-01819 Path Forward ", email from L. Yerger dated 3/17/2009
- 2. "9975-01819 Inspection (Jan. 29, 2009) Results", email from J. Murphy dated 1/29/2009
- 3. "9975-01819 Celotex Assemblies 02/24/09 Inspection Results", email from J. Murphy dated 2/24/2009
- 4. WSMS-CRT-03-0158, "9975 Surveillance Program Assessment of Celotex® Properties", D. Biswas, December 16, 2003
- 5. DOE / USDA-21697 / 1, "Thermal Properties of Wood and Wood Panel Products for Use in Buildings", A. TenWolde, J. D. McNatt and L. Krahn, ORNL, September 1988
- 6. WSRC-TR-2004-00523, "Baseline Mechanical Property Data for Model 9975 Package Celotex® Material", W. L. Daugherty, December 2004
- 7. M-CLC-K-00657, Rev. 1, "Structural Analysis of 9975 Package Subjected to Two Forklift Truck Impact", T. Wu, December 16, 2005

Upper assembly						
Weight	28 lb (12.70 kg)					R-R2-F-0019 Rev 5
	$0/180$ deg.		90/270 deg.		Avg.	Nominal value (inch)
$UD1$ (in)	17.650		17.696		17.673	17.7
$UD2$ (in)	8.566		8.568		8.567	8.55
	$0$ deg.	90 deg.	80 deg.	$270$ deg.	Avg.	
$UR1$ (in)	3.060	3.053	3.050	3.050	3.053	3.075
$UR2$ (in)	1.493	1.511	1.465	1.483	1.488	1.5
$UH1$ (in)	7.206	7.195	7.221	7.218	$7.209*$	7.1
$UH2$ (in)	2.061	2.034	2.068	2.082	2.061	2.1
$UH3$ (in)	4.987	4.966	4.968	4.969	4.972	5.0

Table 1. Physical measurements on 9975-01819 fiberboard assemblies on 3/10/09.

Top assembly calculated density =  $0.289$  g/cc

\* Measured values of dimension UH1 include the air shield. The average is reduced by 0.1 inch in calculating assembly density.



Bottom assembly calculated density =  $0.287$  g/cc











Figure 1. Low fiberboard height condition as first noted when the drum was opened. Photograph provided by NMM.



Figure 2. Mold on upper fiberboard assembly when first removed (January 26, 2009). Photograph provided by NMM.



Figure 3. Bottom region of lower fiberboard assembly after removal from drum on 2/24/09. Photograph provided by NMM.



Figure 4. Lower assembly after removal from drum on 2/24/09. Photograph provided by NMM.



Figure 5. Interior of drum after removal of lower fiberboard assembly on 2/24/09. Photograph provided by NMM.



Figure 6. Lower fiberboard assembly during examination on 3/10/09. The numbers are measured moisture content (%WME). Photograph provided by NMM.



Figure 7. Lower fiberboard assembly during examination on 3/10/09. The numbers are measured moisture content (%WME). Photograph provided by NMM.



Figure 8. Lower fiberboard assembly during examination on 3/10/09. The numbers are measured moisture content (%WME). Photograph provided by NMM.



Figure 9. Overview of lower assembly showing allen wrenches used to indent the fiberboard. At each set of indentations, the depth is recorded for each hole. Greater detail of the indentations is shown in Figure 10.



Figure 10. Impressions (in boxes) made in side of lower assembly by 3 allen wrenches. For each set of indentations, the depth, wrench size and moisture content is indicated.



Figure 11. Impressions made on the bottom of lower assembly (in box) by 3 allen wrenches. The depth, wrench size and moisture content is indicated for each impression.



Figure 12. Impressions made in a control fiberboard section (in box) by 3 allen wrenches. This section had 8.8 %WME moisture content. The depth is noted by each impression.



Figure 13. Calculated thermal conductivity of solid wood as a function of moisture content, for several values of dry density. Based on a model from Reference 5.

#### **Attachment 1. Estimate of water content of 9975-01819 lower fiberboard assembly**

#### **Inputs**

- Data taken on 2/24/09 using a GE Protimeter moisture meter indicate the bottom 1.75 inch has 100 %WME (wood moisture equivalent), and the remainder varies from 20 to 26 %WME on the OD surface and 16 %WME on the ID surface. Although these readings were taken at a limited number of locations, more numerous readings taken subsequently on 3/10/09 show a relatively uniform moisture level above the bottom saturated region.
- From prior destructive examination of four 9975 packages, typical moisture content of the lower fiberboard assembly is ~10.5 %WME on the ID surface and ~14 %WME on the OD surface.
- The 9975-01819 lower fiberboard assembly weighs 58 lb (26310 g). This includes an aluminum bearing plate with a nominal weight of 2189 g. The aluminum plate has a nominal volume of 807 cc, and an annular gap under the plate has a nominal volume of 175 cc.
- An approximate conversion between %WME and wt% water is given by [ref. SRNL-L7200- 2008-00007, "Correlation between Cane Fiberboard Moisture Content and Relative Humidity", W. L. Daugherty. December 10, 2008]

Wt% moisture =  $0.67 * %WME + 2.6$  (valid over a range of  $6 - 40 %WME$ )

#### **Assumptions**

- In the absence of moisture, the lower assembly is assumed to have uniform density throughout the fiberboard.
- The original lower assembly height is assumed to have been 26.7 inches (the nominal drawing value). (With this assembly, the normal tendency for significant height increase with water absorption is more than offset by the compression of the bottom layers.) Based on the fit within the drum, the other measured dimensions are assumed to approximate their original values.
- All compaction is assumed to have occurred in the bottom 1.75 inches, such that this region was originally 2.55 inches high.
- Conservatively assume that the bottom 1.75 inches has 60 %WME. This underestimates the amount of water present based on the test with a separate fiberboard sample. Assume that 80 wt% water provides a fairly realistic estimate of the actual water present.

## **Calculations**

The total fiberboard volume within the lower assembly is 83966 cc (excluding the aluminum plate and annular gap below the plate), based on the measured dimensions from Table 1, nominal dimensions for the plate and gap, and the formula given in SRNS-STI-2008-00019 "Destructive Examination of Shipping Package 9975-05128", W. L. Daugherty, August 2008.

#### *Calculate weight and volume of lower assembly:*

The volume of the bottom 1.75 inches is  $(18.104^{\circ})^2 (\pi/4) (1.75^{\circ}) (16.387 \text{ cc/cu in}) = 7382 \text{ cc}$ The remaining fiberboard has a volume of  $83966 - 7382 = 76584$  cc The weight of the lower assembly is 26310 g The nominal weight of the aluminum bearing plate is 2189 g The weight of fiberboard within the lower assembly is  $26310 - 2189 = 24121$  g

## *Calculate original dry density of lower assembly:*

The dry density of the original lower assembly is given by  $\rho_D$ .

With 60 wt% water, the current density of the bottom 1.75" is  $\rho_B = \rho_D (2.55 / 1.75) (1.60)$ 

With an average of  $\sim$ 20 %WME ( $\sim$ 16 wt% water) in the rest of the assembly, the current density of the rest of the assembly is  $\rho_R = \rho_D (1.16)$ 

Therefore,  $\rho_D$  (2.55 / 1.75) (1.60) (7382 cc) +  $\rho_D$  (1.16) (76584 cc) = 24121 g and  $\rho_D = 0.227$  g/cc

*Calculate water content of lower assembly:*

The original bottom 2.55 inches of the lower assembly had a volume of (7382 cc) (2.55 / 1.75) = 10757 cc

The original total fiberboard volume in the lower assembly was 76584 cc + 10757 cc = 87341 cc The original dry weight of the lower assembly is  $(0.227 \text{ g/cc})$   $(87341 \text{ cc}) = 19826 \text{ g}$ Actual current fiberboard weight in the lower assembly =  $24121$  g

Therefore, the current water content is  $(24121 - 19826)$  / 19826 \* 100 = 21.7 wt% water.

## *Estimate excess water in lower assembly:*

Prior destructive examination packages had an overall average moisture content in the lower assembly of  $\sim$ 12 %WME. This corresponds to  $\sim$ 10.6 wt% water.

At this moisture content, the 9975-01819 lower assembly would hold (19826 g)  $(0.106) = 2102$  g water

With the estimated moisture content, the lower assembly holds (19826 g)  $(0.217) = 4454$  g water The lower assembly therefore has approximately  $(4454 - 2102 = 2352$  g of excess water.

If the above exercise is repeated with the more realistic assumption that the bottom 1.75 inches contains 80 wt% water, one calculates a dry density of 0.223 g/cc, a moisture content of 23.8 wt%, and 2571 g of excess water.

On the basis of these 2 cases, it is likely that the lower assembly contains about 2.5 liters of excess water.

## *Nominal expectations:*

If the lower assembly had a moisture content typical of the destructive examination packages (10.6 wt%), and the lower layers had not been compressed, one might expect the following:

- Lower assembly height  $=\infty$ 26.7 inches (assumption)
- Lower assembly weight =  $(19826 \text{ g}) (1.106) + 2189 \text{ g} = 24117 \text{ g}$
- Lower assembly fiberboard weight =  $(19826 \text{ g}) (1.106) = 21928 \text{ g}$
- Lower assembly fiberboard volume = 87341 cc
- Lower assembly fiberboard density =  $21928 \text{ g} / 87341 \text{ cc} = 0.251 \text{ g/cc}$

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