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EXAMINATION OF SHIPPING PACKAGE 9975-02403

W. L. Daugherty March 2016 SRNL-STI-2016-00152, Revision 0



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Summary

SRNL examined shipping package 9975-02403 following storage of nuclear material in K-Area Complex (KAC). As a result of field surveillance activities in KAC, this package was identified to contain several non-conforming and other conditions, including:

- The axial gap criterion was exceeded,
- The difference between the upper fiberboard assembly inside height and the lower fiberboard assembly height from lower step to top of lead shield was less than specified,
- Stains or potential mold were observed on the lid underside, air shield and drum interior surfaces,
- Condensation was observed on internal components (drum, lid, air shield),
- The possibility of mold on the fiberboard was identified, and
- The drum contained minor corrosion along the lower flange.

Further examination of this package in SRNL confirmed significant moisture and mold in the bottom layers of the lower fiberboard assembly, and identified additional corrosion along the seam weld and on the bottom of the drum.

It was recently recommended that checking for corrosion along the bottom edge of the drum be implemented for packages that are removed from storage, as well as high wattage packages remaining in storage. The appearance of such corrosion on 9975-02403 further indicates that such corrosion may provide an indication of significant moisture concentration and related degradation within the package. This condition is more likely to develop in packages with higher internal heat loads.

Background

Package 9975-02403 was loaded with plutonium oxide material packaged at FB-Line in December 2003 in accordance with DOE-STD-3013 and received into KAC in December 2003. The contents generated approximately 14.4 watts heat load. Routine field surveillance [1] was performed on February 4 and 10, 2016. This package was among a group selected for surveillance because of its high internal heat load, and was sent to SRNL for more detailed examination based on the observation of several non-conforming conditions and other conditions of interest, including:

- The axial gap criterion (1 inch maximum from the top of the air shield to the drum flange) was exceeded. The average axial gap was 1.739 inches.
- The difference between the upper fiberboard assembly inside height and the lower fiberboard assembly height from lower step to top of lead shield should be greater than 0.425 inch to ensure any gap between the two fiberboard assemblies is less than 1/8 inch. The average difference between these two dimensions was 0.29 inch.
- Stains or potential mold were observed on the lid underside, air shield and drum interior surfaces (Figures 1 3).
- Condensation and water stains were observed on internal components (drum, lid, air shield) (Figures 1-2).
- Dark stain and slight damage to the upper fiberboard assembly were noted (Figure 4).
- The drum contained minor corrosion along the lower flange (Figure 5).

The SRNL examination was performed on February 23-24, 2016. This report documents the results of examination of 9975-02403.

Examination Results

After transfer to SRNL, the package was re-examined. In addition to the areas examined in KAC, the shield and lower fiberboard assembly were removed to provide additional access to these components and the drum interior. Dimensions and other data from the fiberboard assemblies during both examinations are summarized in Table 1. Specific observations made during the SRNL examination include:

- The axial gap remained approximately the same as measured in KAC (1.722 inch)
- The dark stains on the air shield (other than the obvious water stains) resulted from rubbing against the encapsulated blanket, as evidenced by matching patterns between the two components. The dark stains on the lid and drum do not appear to be mold, but were not identified. They have no discernible height, and do not rub off easily. On the drum, these stains occur at 3 elevations at the top, near the interface between fiberboard assemblies, and near the bottom.
- In addition to several areas of corrosion along the drum lower flange, there are multiple small corrosion spots in one area on the drum bottom, and a few small spots along the vertical seam weld (Figures 6-7).
- The dark spot on the upper fiberboard assembly is smeared glue. A fainter water stain forms an irregular ring around the step on both fiberboard assemblies (Figure 8)
- The bottom layers of the lower fiberboard assembly were saturated, with significant mold present (Figure 9). The bottom layers were very fragile and separated during handling. Portions of the bottom layer remained stuck to the drum (Figure 10). After scraping this material off the drum, its weight was 48 grams. Additional regions of mold were growing on the side of the lower fiberboard assembly (Figure 11).

The axial fiberboard dimensions are generally less than nominal drawing dimensions. While some variation in fiberboard dimensions is normal, the overall height of the lower assembly is significantly reduced (26.7 inches nominal, 25.774 actual). Compared to nominal dimensions, two-thirds of this reduction has occurred in the bottom 3.8 inches (below the bearing plate).

The drum is constructed in a manner that creates a tight crevice along the bottom edge, with the possibility of a very narrow leak path through this crevice. Given the appearance of corrosion on the outer drum surface in this area, a small amount of water was placed along the crevice inside the drum to see if a significant leak path existed. No water was observed along the outer surface after 30 minutes.

After identifying several corrosion spots along the vertical seam weld of the drum, the drum interior was re-examined for evidence of similar corrosion on the corresponding inner surface (Figures 12-13). Several elongated dark spots were observed adjacent to the seam weld inner surface, but these generally did not match the locations of the corrosion spots on the outside surface. It is therefore concluded that the corrosion spots did not originate from the inside.

Dimensional measurements were taken for the lead shield to compare to drawing requirements (Table 2). The inside diameter at the top of the shield is specified to be 7.25 - 7.26 inches. The average of 4 measurements for this dimension was 7.266 inches. The other shield dimensions meet drawing requirements. The thickness of the shield (0.538 inch) is the same at the top and at the bottom, indicating that no significant creep of the lead has occurred. Most of the side and part of the bottom surface is covered with a white, lead carbonate corrosion layer (Figure 14).

Discussion

Package 9975-02403 was among a group of 10 high-wattage packages selected for surveillance in 2015. Two additional packages from this group (9975-02101 and 9975-02713) had similar conditions (excessive axial gap, fiberboard mold, drum corrosion, etc.) to varying degrees [2]. This constitutes a relatively high rate of incidence (30%) for high wattage packages. These conditions are consistent with laboratory observations of test packages. With relatively high internal heat loads, the moisture normally present in the fiberboard will redistribute preferentially to the cooler regions (i.e. the OD and bottom surfaces of the fiberboard). Reduced moisture in the fiberboard sidewall region will lead to shrinkage, especially in the axial direction. And increased moisture in the bottom layers will decrease the compression strength, such that the weight of the internal components will cause additional compaction of these layers [3].

The moisture level within the fiberboard can increase as a result of fiberboard degradation. At elevated temperature (above ~120 $^{\circ}$ F), cellulose will begin to gradually break down through pyrolysis (the same chemical process active during burning, but at a much slower rate). One of the reaction products from pyrolysis is water, so this process can increase the total amount of water present within the fiberboard. In addition, mold will break down / consume the cellulose and other compounds within the fiberboard. This is also a chemical process, for which water would be a reaction product. Therefore, once conditions are suitable for mold to grow, additional water will be produced.

As moisture migrates through the fiberboard, chlorides can leach out and concentrate at the bottom. This can create favorable conditions for pitting or stress corrosion cracking of the drum. Since the bottom lip of the drum is not fabricated to produce a water-tight seal, some of the moisture and chlorides can seep out. As a result, corrosion may occur in this area of the drum when significant moisture migration has occurred. Regions immediately adjacent to a stitch weld along the bottom lip are especially prone to corrosion since the residual stresses from welding help create easier leak paths at these locations. The presence of chlorides has not been confirmed in these packages, but is the most probable cause of the drum corrosion. Chlorine is added to the cane fiber prior to fiberboard production as a fungus control measure [4].

In the past, the axial gap criterion has been used as a primary indicator of the condition of the fiberboard in the package. As moisture starts to concentrate in the bottom fiberboard layers, the axial gap increases. One drawback with this indicator is that the drum has to be opened to measure the axial gap. An additional indicator– the presence of corrosion on the drum bottom edge – has been proposed [2], and provides an opportunity to screen packages for significant internal degradation without opening them.

Conclusions

SRNL has assisted in the examination of shipping package 9975-02403 following storage of nuclear material in K-Area Complex (KAC) for over 12 years. This package was among a larger group selected for surveillance as part of a specific focus on high wattage packages. Several non-conforming conditions were displayed, including excessive axial gap, mold on the fiberboard, and localized corrosion on the drum bottom flange. A new screening check for drum corrosion has been recently recommended for packages that are removed from storage, as well as high wattage packages remaining in storage. The presence of corrosion could signal the need to remove the lower fiberboard assembly for further inspection of the fiberboard and drum.

References

- 1. Procedure SOP-CSS-232-K Rev. 1, Attachment 8.5 completed for 9975-02403, February 10, 2016
- 2. SRNL-STI-2016-00014, "Examination of Shipping Packages 9975-01641, 9975-01692, 9975-03373, 9975-02101 and 9975-02713", W. L. Daugherty, January 2016
- 3. SRNL-STI-2012-00429, "Analysis of the Axial Gap vs Fiberboard Moisture Content in a 9975 Shipping Package", W. L. Daugherty, September 2013
- 4. "Roof Decks Quickly Corroded by Insulation", Chuck Marvin and Bruce Byrne, *Interface*, January 2011, pp 32-36

	9975-0240	3 (in KAC) 2/4, 10/2016	9975-02403 (in SRNL) 2/23-24/2016				
Upper	Dimension	s Moisture content	Dimensions	Moisture content (%WME)			
assembly	(inch)	(%WME)	(inch)				
UD1	17.600		17.629				
UD2	8.516		8.564				
UR1	3.057 *		3.036				
UR2	1.485 *	^{0.3} 7.5	1.507	12 5			
UH1	NA		7.105				
UH2	2.006		2.036	13.0 8.5 8.3 12.5			
UH3	4.880		4.912	10.4 10.1			
Lower	Dimension	s Moisture content	Dimensions	Moisture content (%WME)			
assembly	(inch)	(%WME)	(inch)				
LD1	NA	128 67 60 112	18.040	12.8 4.89 8 9 11.6			
LD2	NA		8.492				
LR1	NA		3.236	7.6 ''			
LR2	NA		1.525	17.0 8.8 8.0 15.3			
LH1	NA		25.774	8.8			
LH2	NA		20.125	16.7			
LH3	NA		2.004	100 10.2 9.7			
Upper assy weight		25.97 lb		26.04 lb			
Upper air space RH		88.5%	39.7% at 76 F				
Axial gap		1.739 inch	1.722 inch				
Notes	Slight resis	tance felt during removal	The dark spot	ot on the upper assembly is glue			
	of upper fil	perboard assembly might	residue. Water stain left an irregular ring around				
	have cause	d some damage.	the step on both assemblies. Lower assembly				
	* Dimensio	ons UR1 and UR2 derived	bottom layers saturated, separating and very				
	from other	measurements.	fragile, some material stuck to drum. Mold				
			around bottom layers and some areas on side.				

Table 1. Detailed fiberboard data for 9975-02403

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.



Dimension	0/180 de	180 deg. 90/270 deg.		Avg.	Requirement (inch)	_	ID1	il T		
	(inch)		(inch)		(inch)		Ŕ			
OD (in)	8.332		8.336		8.334	8.252 - 8.35				
ID1 (in)	7.261		7.289		7.275*	7.25 - 7.26				
ID2 (in)	7.256		7.260		7.258	7.24 – 7.26				
	0 deg.	90 deg.	180 deg.	270 deg.						
R (in)	0.542	0.518	0.549	0.545	0.538	0.506 min		102		
H (in)	24.672	24.678	24.676	24.685	24.678	24.556 - 24.7		\leftarrow $1D_{2}$ \rightarrow	🖡	,

Table 2. Lead shield dimensions

(OD - ID2) / 2 = 0.538 inch

* ID1 re-measured at 4 locations, average value = 7.266 inch





Figure 1. Stains and liquid condensate on underside of lid. Photo taken by NMM personnel.



Figure 2. Water stains and other markings on air shield and upper drum interior. Photo taken by NMM personnel.



Figure 3. Water stains and other staining on drum interior. Light water staining is also visible on the step of the lower fiberboard assembly. Photo taken by NMM personnel.

Figure 4. Dark stain on upper fiberboard assembly. Photo taken by NMM personnel.

Figure 5. Corrosion on lower flange of drum. Photos taken by NMM personnel.

Figure 6. Region of localized corrosion on the drum bottom.

Figure 7. Localized drum corrosion along the vertical seam weld.

Figure 8. Water stain on the step of each fiberboard assembly.

(a) upper assembly

(b) lower assembly

Figure 9. Mold near bottom of lower fiberboard assembly, along with layer separation and material missing from the bottom

Figure 11. Mold on the side of the lower fiberboard assembly

(b)

Figure 12. Example of corrosion spot on the drum exterior (a) along the vertical seam weld (~19.5 inches from the drum flange), compared to the corresponding location on the interior (b) with no visible corrosion

Figure 13. Example of corrosion spot on the drum exterior (a) along the vertical seam weld (~30.8 inches from the drum flange), compared to the corresponding location on the interior (b) with an elongated dark spot.

Figure 14. Bottom and side of lead shield from 9975-02403

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