Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

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Examination of Shipping Package 9975-04985

W. L. Daugherty

August 2017

SRNL-STI-2017-00514, Revision 0

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Prepared for
U.S. Department of Energy

SRNL-STI-2017-00514
Revision 0

Keywords: *K-Area*
Fiberboard
Shield

Retention: *Permanent*

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Summary

Package 9975-04985 was examined following the identification of several unexpected conditions during surveillance activities. A heavy layer of corrosion product on the shield and the shield outer diameter being larger that allowed by drawing tolerances contributed to a very tight fit between the upper fiberboard assembly and shield. The average corrosion rate for the shield is estimated to be 0.0018 inch/year or less, which falls within the bounding rate of 0.002 inch/year that has been previously recommended for these packages.

Several apparent foreign objects were noted within the package. One object observed on the air shield was identified as tape. The other objects were comprised of mostly fine fibers from the cane fiberboard. It is postulated that the upper and lower fiberboard assemblies were able to rub against each other due to the upper fiberboard assembly being held tight to the shield, and a few stray cane chips became frayed under vibratory motions.

Background

Package 9975-04985 was packaged at Hanford. The package was leak tested prior to loading on October 26, 2007, and it was received in K-Area Complex (KAC) in November 2007. It contained an internal heat load of 16.6 watts. KAC personnel performed surveillance [1] on package 9975-04985 on June 28, 2017 following a storage period of about 9.6 years, and noted several unsatisfactory conditions [2]. These include:

- The upper fiberboard assembly did not come out smoothly - it required two operators to remove. The fit to the shield was very tight. Some effort was also required to re-install the fiberboard (Figure 1).
- There was a heavy layer of corrosion product (lead carbonate) on the shield, and a significant amount fell loose when the fiberboard assembly was removed. It was noted that a cloud of lead carbonate was generated by removing the fiberboard assembly. Additional lead carbonate was observed on the ID surface of the upper fiberboard assembly (Figure 2), on the shield lid and on the lower fiberboard assembly (Figure 3).

In an additional observation, foreign material was noted on the fiberboard assemblies and drum interior (Figures 1, 2, 4 – 6).

On July 25, 2017 this package was further examined in KAC by SRNL (Daugherty), with assistance from Sides (SRNL Packaging Technology and Pressurized Systems), Sunderland (SRNL Materials Evaluation), McEvoy (9975 Design Authority Engineer), Grimm (NMM Engineering) and Stevens (KAC Operations Support). This report documents the findings from that examination.

Results

During the July 25, 2017 examination, the upper fiberboard assembly came out and was replaced with relative ease. The upper fiberboard assembly inside diameter and shield outer diameter were measured for comparison. Similar measurements were taken during surveillance. These numbers are summarized in Table 1.

All visible lead surfaces were covered with lead carbonate. Some areas, especially along the top edge had flaked or blistered off, but additional lead carbonate remained. Most of the flaking and blistering had occurred in the upper 2 – 3 inches of the shield, which displayed an overall rougher surface (Figure 7).

The thickness of the lead carbonate remaining on the shield was measured with an Elcometer 345 FN coating thickness gage, which uses an eddy current technique. The probe was zeroed on a non-ferrous base material, and then verified with a non-metallic shim set. The gage was then used to measure the lead carbonate thickness at multiple locations, as illustrated in Figure 8. These locations included locations of previous flaking. The lead carbonate thickness ranged from 0.010 to 0.038 inch on the top edge of the shield, and from 0.004 to 0.035 inch on the side of the shield. The thinner readings were generally taken at sites of prior flaking, or more than ~2 inches from the top of the shield

Calipers were used to measure the shield thickness at four locations around the top (about ½ inch counterclockwise from each threaded insert). Attempts were then made to scrape off the lead carbonate and re-measure the shield thickness. The remaining lead carbonate thickness at each location was also measured with the coating thickness gage. These data are summarized in Table 2. Much of the lead carbonate was tightly adherent and did not scrape off easily or completely. Due to the roughness of the shield surface and the uncertainty of re-measuring the exact same location, the calculated thickness of lead carbonate removed is uncertain, as evidenced by the range of values (-0.004 to 0.042 inch). However, these surface irregularities should average out over multiple measurements, and the calculated average thickness of lead carbonate removed (0.010 inch) combined with the average remaining lead carbonate thickness (0.010 inch) provides a degree of verification in the coating thickness gage measurements (0.017 to 0.034 inch in this region).

The foreign material on the fiberboard assemblies and drum interior was visually examined. The object adherent to the upper fiberboard assembly air shield (Figure 9) was identified as tape. It lies along the top edge on the side of the air shield and has a straight edge and two irregular torn edges. One of the torn edges follows the top edge of the air shield. Based on its position, it appears a stray piece of tape (probably masking tape) became stuck to the top and side of the upper assembly. Upon removal from the top of the air shield, the portion that was stuck to the side tore off and remained in place.

Additional foreign material was observed in two locations on the outer step of the upper fiberboard assembly (Figure 10). The material in both these locations had a “fluffy” appearance. A portion of the larger object was taken for further examination. Similar “fluffy” material was observed in three locations on the outer step of the lower fiberboard assembly and adjacent drum surface (Figure 11). In addition, one additional foreign object with more of a “wispy” appearance was observed on the outer step of the lower fiberboard assembly (Figure 12). This “wispy” object was also taken for further examination.

The two foreign object samples were examined at low magnification (up to 100X). The “fluffy” material consists of a few chips of cane fiber enmeshed in a larger quantity of much finer fibers (Figure 13). In comparison, a sample of known cane fiberboard viewed at the same magnification has a similar appearance, although it contains a much higher ratio of larger chips compared to finer fibers (Figure 14). The larger cane fiber chips are composed of a large number of fine parallel fibers, which can separate under some circumstances. The "fluffy" material appears to be cane fiber which has been mostly broken down physically to its finest constituent fibers. The wispy sample contains a number of straight fibers that are intermediate in size to the cane fiber chips and the finer fibers. This sample also has some finer fibers attached to each straight section (Figure 15). It appears to be a cane fiber chip that has begun to break down physically, but has not yet been reduced to fine fibers.

Discussion

Comparing the dimensions of the shield and upper fiberboard assembly shows clearance existed at all points of measurement, although the second set of upper fiberboard assembly measurements shows a degree of eccentricity. It is unknown which fiberboard dimension would have originally aligned with which shield dimension. The diametral clearance for any combination of measured values is smaller than the minimum value based on drawing dimensions, although it is recognized that fiberboard dimensions may change in service and are not bound to original fabrication tolerances. The linear white streaks shown in Figure 2 indicate that local areas existed where the upper fiberboard assembly was tight to the shield.

It is also noted that the shield diameter is consistently smaller in the second examination than during the original surveillance. The shield was about 30 – 35 °F warmer when the package was first opened for surveillance compared to the second examination. The linear coefficient of thermal expansion of lead is 16 E-6 /°F, which would increase the shield diameter by about 0.005 inch for this temperature difference. This is not enough to account for all of the observed change. Most of the decrease in shield diameter likely results from the loss of lead carbonate each time the fiberboard was removed or replaced.

Drawing requirements specify the minimum shield ID (at the top) is 7.25 inch, and the maximum shield OD is 8.35 inch. This means the maximum shield thickness is 0.55 inch, assuming the ID and OD are concentric. However, all shield thickness measurements (Table 2) are greater than this value. After subtracting the maximum measured lead carbonate thickness, all but one thickness measurements still exceed this value. It is possible that the shield dimensions did not meet drawing requirements after fabrication. An alternate possibility suggested by the roughness around the top of the shield is that there was some degree of porosity in the top region of the shield, and that buildup of corrosion product throughout these pores has caused an overall expansion of the shield sidewall.

The upper region of the shield sidewall had lead carbonate thicknesses up to 0.038 inch. The thickness of lead lost to corrosion is approximately 45% of the corrosion product thickness if lead carbonate ($PbCO_3$) is formed, and approximately 40% of the corrosion product thickness if basic lead carbonate ($Pb_3(CO_3)_2(OH)_2$ is formed [3]. Both of these corrosion products could be formed, although basic lead carbonate has generally been identified on shields where the corrosion product was analyzed [4, for example]. The production of 0.038 inch of corrosion product would therefore consume up to 0.017 inch of lead. Package 9975-04985 was in service (following leak test) for about 9.7 years, which gives an average corrosion rate of 0.0018 inch/year based on the thickest measured corrosion, and about 0.0013 inch/year for the more average thickness of about 0.028 inch measured in the upper region of the shield. Reference 3 recommended a corrosion rate of 0.002 inch/year as a conservative estimate for 9975 packages. The observed behavior falls within this rate.

The apparent foreign material that was observed on the drum and fiberboard assemblies appears to originate from the cane fiberboard. Both incorporate a combination of larger chips and finer fibers, although the foreign material has a much higher concentration of finer fibers. The larger chips are relatively intact sections of cane, which in turn appear to be made up of numerous fine fibers. The cane fiberboard sample also had a number of small particles throughout, presumably from the binders used in its manufacture. Similar particles were observed in the collected sample, but were less concentrated among the more numerous finer fibers. The collected sample is consistent with cane fiberboard material in which the finer fibers have been selectively concentrated, or most of the coarser cane pieces have been "teased" or separated into their finer constituent fibers.

It is postulated that a very slight gap between the upper and lower fiberboard assemblies, combined with some degree of vibration, led to rubbing between the fiberboard assemblies which caused some fibers to fray and bunch up like a lint ball. The vibratory motion could lead to the fibers being pushed outward and collecting in clumps. Such rubbing would not normally be expected between fiberboard assemblies. However, it may have happened in this package because the upper assembly had become (or was originally) tight to the shield. With a small amount of fiberboard shrinkage (from moisture migration), the weight of the upper fiberboard assembly would be carried by the shield instead of the lower assembly and allow the postulated rubbing to occur. All of this material was observed on the fiberboard outer step, or the drum surface adjacent to the fiberboard outer step. When assembled, the fiberboard assemblies contact each other on the outer step, with a small gap at the inner step. Therefore, it is on this outer step that the two assemblies might rub against each other. Accumulated loose material might be expected to eventually migrate outwards and collect against the drum surface.

Conclusions

Package 9975-04985 was examined to determine the cause of a reported tight fit between the upper fiberboard assembly and shield, and to identify several apparent foreign objects that were observed during surveillance activities. Corrosion product (lead carbonate) on the shield was heavier than normal (up to 0.038 inch thick), and was the probable cause of a tight fit to the upper fiberboard assembly. Additional lead carbonate had been present at the time of surveillance, but was knocked loose by the forced removal of the fiberboard. The shield outer diameter was found larger than allowed by drawing tolerances. Even for the heavier corrosion deposits, the average corrosion rate is estimated to be 0.0018 inch/year, which is less than the bounding corrosion rate of 0.002 inch/year recommended previously.

The apparent foreign material on the air shield was identified as tape. The other objects were found to be mostly fine fibers from the cane fiberboard. It is postulated that the upper and lower fiberboard assemblies were able to rub against each other due to the upper fiberboard assembly being held tight to the shield, and a few stray cane chips became frayed under vibratory motions.

References

[1] WSRC-TR-2001-0286, Rev. 7, “The Savannah River Site Surveillance Program for the Storage of 9975/3013 Plutonium Packages in KAC”, Jan 2015.

[2] 9975-04985 Data Sheet SOP-CSS-231-K Attachment 8.5, “3013/9975 Surveillance Data Sheet”, June 28, 2017.

[3] WSRC-TR-2006-00094, “Corrosion of Lead Shielding in Model 9975 Package”, K. H. Subramanian, March 2006.

[4] WSRC-TR-2005-00273 Rev. 1, “Destructive Examination of Shipping Package 9975-02234”, W. L. Daugherty, September 2005.

Table 1. Dimensional check of shield / fiberboard interference

	Upper fiberboard assembly ID (inch)		Shield OD (inch)	Potential range of diam. clearance (inch)
6/28/2017 Surveillance Individual values (~90 deg. apart)	8.523 8.515		8.50 8 $^{15}/_{32}$	0.015 – 0.054
Average value	8.519		8.48	0.039
7/25/2017 Examination Individual values (~45 deg. apart, fiberboard measured twice)	8.517 8.522 8.496 8.510	8.540 8.521 8.525 8.470	8.44 8.45 8.41 8.45	0.020 – 0.130
Average value	8.513		8.44	0.073
Drawing requirement	8.55 +/- 0.05		8.262 - 8.35 maximum	0.15 – 0.338

Table 2. Shield thickness measurements before and after scraping to remove the lead carbonate

Location *	Shield thickness before scraping (inch)	Shield thickness after scraping (inch)	Apparent** lead carbonate thickness removed by scraping (inch)	Measured lead carbonate thickness remaining after scraping (inch)	Total apparent** lead carbonate thickness (inch)
A	0.637	0.611	0.026	0.016	0.042
B	0.598	0.579	0.019	0.005	0.024
C	0.611	0.615	-0.004	0.015	0.011
D	0.615	0.614	0.001	0.006	0.007
Average	**0.615**	**0.605**	**0.010**	**0.010**	**0.021**

* These locations are illustrated in Figure 8. Each location is approximately ½ inch counterclockwise from a threaded insert and approximately ½ inch from the top of the shield.
** Given the rough shield surface, the shield thickness can vary over a small area. The lead carbonate thickness is identified as “apparent” because minor variation in placement of the calipers on the rough shield surface can significantly change the results.

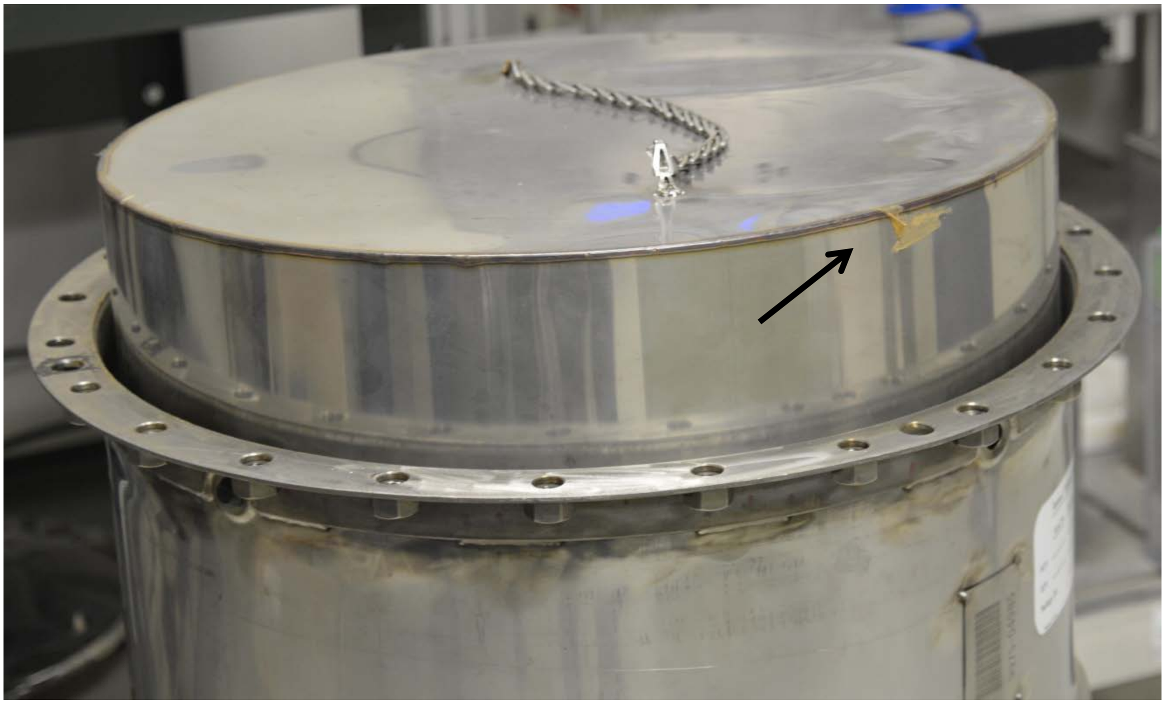


Figure 1. 9975-04985 with upper fiberboard assembly partially replaced. Note the tape on the air shield (arrow). Photo taken by KAC personnel during surveillance.

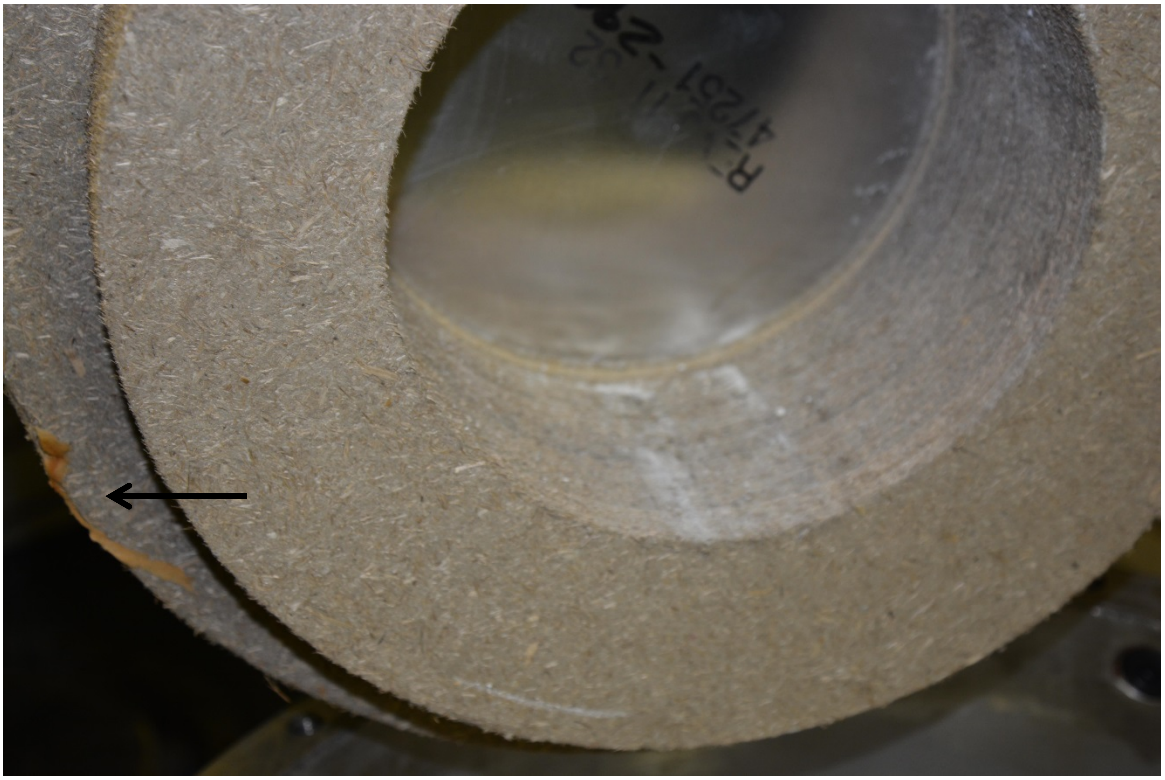


Figure 2. Lead carbonate deposits noted on the upper fiberboard assembly ID surface during surveillance. The linear orientation of the white deposits indicates a tight fit to the shield. Fibrous deposits are also shown (arrow). Photo taken by KAC personnel during surveillance.

Figure 3. Lead carbonate deposits from the shield which ended up on the lower fiberboard assembly and shield lid. Photo taken by KAC personnel during surveillance.

Figure 4. Fibrous deposit on drum interior surface.   Photo taken by KAC personnel during surveillance.

Figure 5. Fibrous deposits on lower fiberboard assembly. Photo taken by KAC personnel during surveillance.

Figure 6. Fibrous deposits on upper fiberboard assembly. Photo taken by KAC personnel during surveillance.

(a)

(b)

Figure 7. Roughness and irregularities of the shield surface, especially in the top ~2 inches.

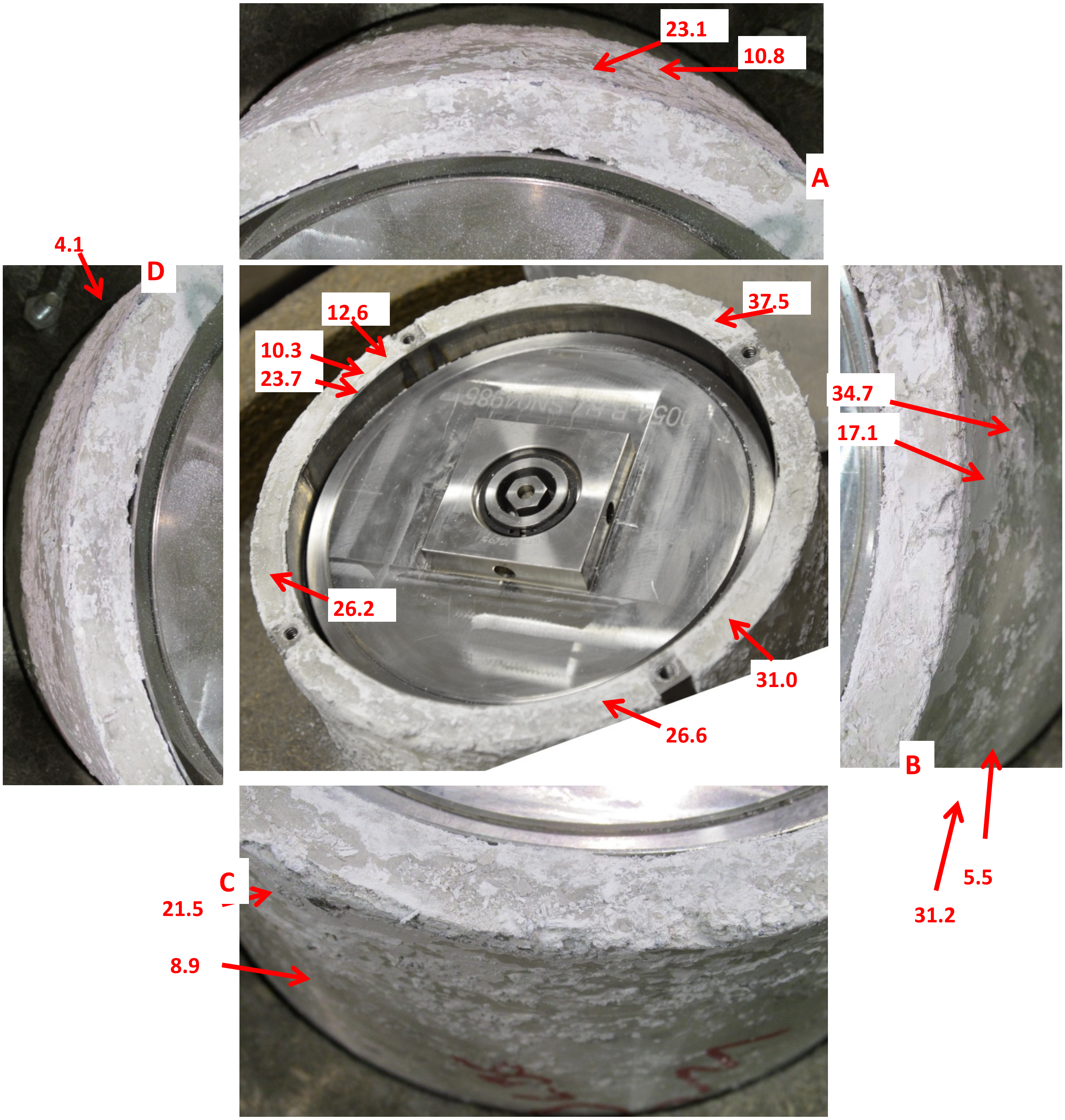


Figure 8. Lead carbonate thickness measurements taken with the coating thickness gage. All measurements are in mils. The arrows identify the approximate measurement location. The four letters (A, B, C, D) show the approximate location of shield thickness measurements and lead carbonate thickness measurements made after some of the lead carbonate was scraped off. Center photo taken by KAC personnel during surveillance.

Figure 9. Foreign material on the upper fiberboard assembly air shield, which was identified visually as tape. The upper assembly is upside down in this photo.


(a)


(b)

Figure 10. “Fluffy” foreign material observed in two locations (a, b) on the upper fiberboard assembly


(a)


(b)

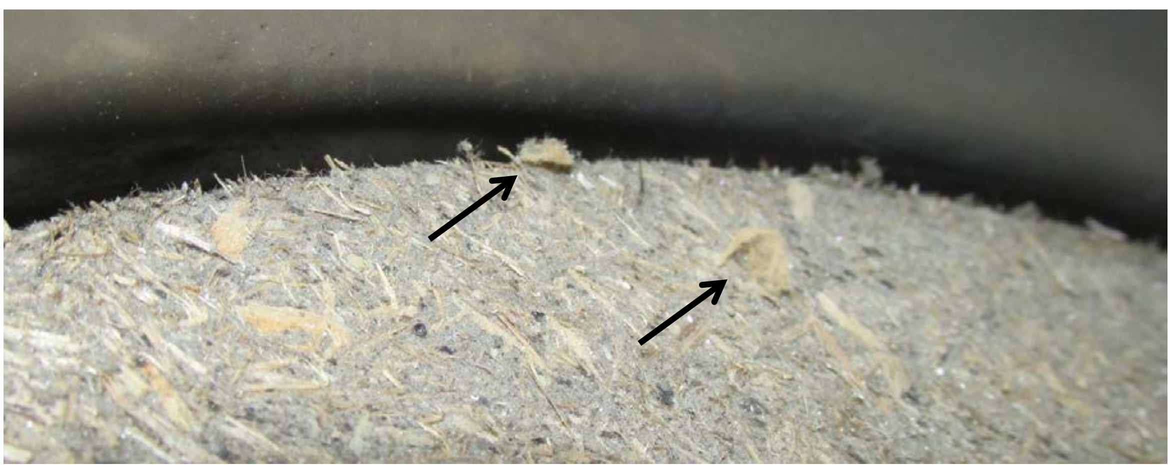

(c)

Figure 11. “Fluffy” foreign material observed on the drum (a, b) and lower fiberboard assembly (c).

Figure 12. “Wispy” foreign material observed on the lower fiberboard assembly.

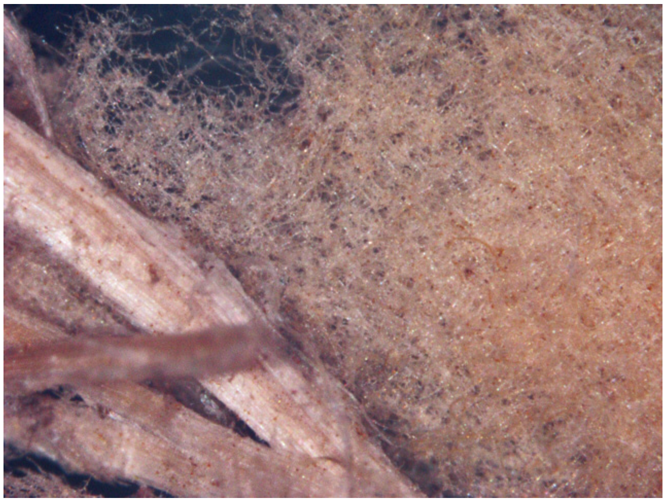
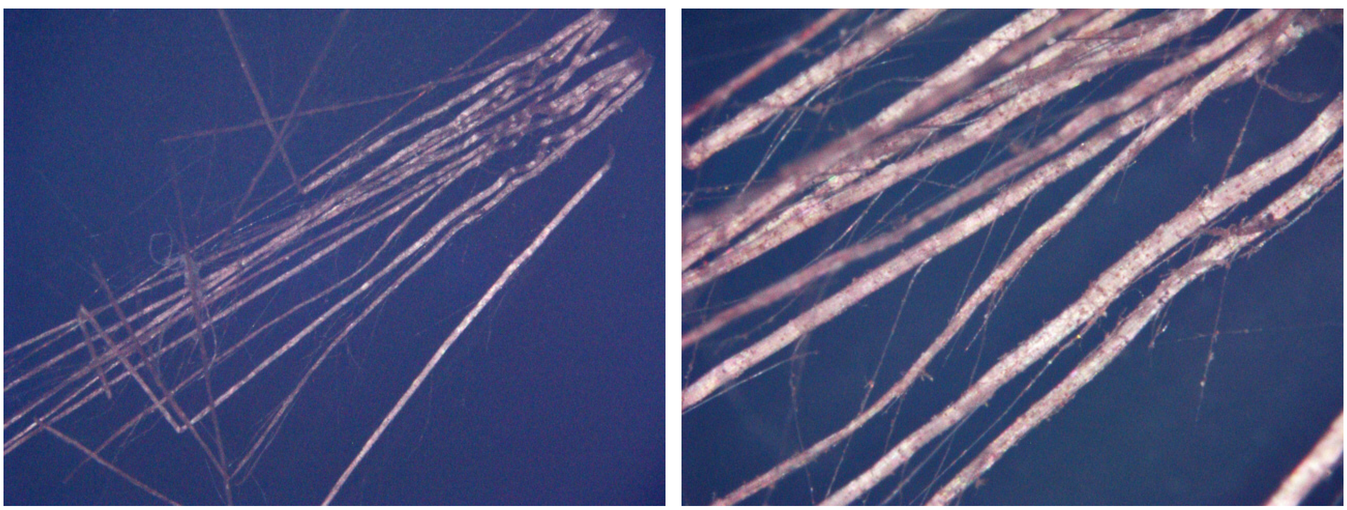


Figure 13. "Fluffy" material removed from the upper fiberboard assembly, photographed at 50X.

Figure 14. Sample of cane fiberboard photographed at 50X.



(a) (b)

Figure 15. "Wispy" material removed from the lower fiberboard assembly, photographed at 15X (a) and 50X (b).

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