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EXAMINATION OF SHIPPING PACKAGES 9975-01641, 9975-01692, 9975-03373, 9975-02101 AND 9975-02713

W. L. Daugherty
January 2016
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APPROVALS:

W. L. Daugherty  
Author, Materials Science and Technology  
___________________  Date ________________

T. E. Skidmore  
Technical Review, Materials Science and Technology  
___________________  Date ________________

K. A. Dunn  
Pu Surveillance Program Lead, Materials Science and Technology  
___________________  Date ________________

G. T. Chandler  
Manager, Materials App & Process Tech  
___________________  Date ________________

E. R. Hackney  
NMM Engineering  
___________________  Date ________________

REVIEWS:

J. W. McEvoy  
9975 Shipping Package Design Authority  
___________________  Date ________________
Summary

SRNL has assisted in the examination of five 9975 shipping packages following storage of nuclear material in K-Area Complex (KAC). Two packages (9975-01641 and -01692) with water intrusion resulting from a roof leak were selected for detailed examination after internal fiberboard degradation (mold) was observed. 9975-01692 contained regions of saturated fiberboard and significant mold, while the second package was less degraded. A third package (9975-03373) was removed from storage for routine surveillance activities, and set aside for further examination after a musty odor was noted inside. No additional degradation was noted in 9975-03373, but the lower assembly could not be removed from the drum for detailed examination.

Two additional packages (9975-02101 and -02713) identified for further examination were among a larger group selected for surveillance as part of a specific focus on high wattage packages. These two packages displayed several non-conforming conditions, including:
- The axial gap criterion was exceeded
- A significant concentration of moisture was found in the bottom fiberboard layers, with active mold in this area
- Condensation and/or water stains were observed on internal components (drum, lid, air shield)
- Both drums contained localized corrosion along the bottom lip.

It is recommended that a new screening check be implemented for packages that are removed from storage, as well as high wattage packages remaining in storage. An initial survey for corrosion along the drum bottom lip of high wattage packages could identify potential degraded packages for future surveillance focus. In addition, after packages have been removed from storage (and unloaded), the drum bottom lip and underside should be inspected for corrosion. The presence of corrosion could signal the need to remove the lower fiberboard assembly for further inspection of the fiberboard and drum prior to recertification of the package.

Background

On April 28, 2014, three 9975 packages (9975-01641, -01692 and -03373) were examined in K Area by B. Eberhard and W. Daugherty, with assistance from P. Stephens, E. Bell and additional KAC personnel. The first two of these packages (9975-01641, -01692) had experienced water intrusion from a roof leak. An additional six packages affected by the roof leak were previously found to show no evidence of water intrusion. The third package opened on April 28, 2014 (9975-03373) had been opened to perform destructive examination of the 3013 container inside, and was observed to have a musty odor. Initial observations from these three packages were reported informally on April 28, 2014 [1]. A more formal report was delayed to determine if 9975-01692 could be sent to SRNL for further examination after excessive mold was observed on the fiberboard. After this did not happen in a timely manner, it was concluded that the conditions within the package had changed sufficiently that further examinations were not warranted.

Two additional packages (9975-02101 and -02713) were removed from storage in KAC for field surveillance in August 2015. They were among a group selected because of their high internal heat load, and were sent to SRNL for more detailed examination based on the observation of several non-conforming conditions. This examination was performed August 20, 2015.
This report documents the results of examination of these five packages.

**Examination Results**

**Package 9975-01641**
Package 9975-01641 was received in KAC in April 2003 with a 3.1 watt internal heat load. It experienced significant water exposure to the drum exterior, as evidenced by stains/debris on the drum and lid (Figures 1, 2). Some water entered the drum, as evidenced by stains on the drum flange and air shield (Figure 3) and on the drum side interior (Figure 4). Local regions of mold were observed on the side and the bottom of the lower fiberboard assembly (Figures 5, 6). Water stains were observed on one side of the upper fiberboard assembly (Figure 7), but no mold was present.

Several small spots were noted on the drum side near the bottom and along the bottom lip (Figure 8). These could indicate corrosion of the drum, or could be debris that was carried to this position.

This package was first opened on September 11, 2013. At that time, the relative humidity in the upper air space was 64.7%, and the axial gap ranged from 0.67 to 0.71 inch. The upper fiberboard assembly moisture content was 11 – 19 % wood moisture equivalent (WME).

Measurements and notes taken on the fiberboard assembly on April 28, 2014 are summarized in Table 1. A modest moisture gradient is seen across the fiberboard. The minimum moisture level (on the ID) is the same as noted 7 months earlier, while the peak OD moisture level is reduced. In addition, the absence of an increase in the moisture content at the bottom suggests that the amount of water entering the drum was minimal. Otherwise, an increase in the moisture level at the bottom might be expected.

**Package 9975-01692**
Package 9975-01692 was received in KAC in April 2003 with a 1.5 watt internal heat load. It experienced significant water exposure to the drum exterior, as evidenced by stains/debris on the drum and lid (Figures 9, 10). Significant water entered the drum, as evidenced by stains on the drum flange and standing water on the air shield (Figure 11), and heavy mold on the fiberboard assemblies (Figures 12 – 15). Numerous white flakes were observed on the upper fiberboard ID surface and on top of the shield (Figures 13, 16). This is assumed to be lead carbonate from the shield, but is more than is typically observed.

Light staining and/or corrosion were noted on the drum exterior along the bottom lip (Figure 17) and on the drum bottom. With minimal staining on the drum sides in these areas, it is likely that at least some of this staining / corrosion resulted from water inside the drum seeping out through the bottom lip.

This package was initially opened on September 13, 2013 by KAC personnel to remove the contents, and it was known from that effort that significant mold was present on the fiberboard. At that time, the relative humidity in the upper air space was 66.6%, and the axial gap ranged from 0.48 to 0.50 inch. The upper fiberboard assembly moisture content was 18 – 19 %WME, with wet regions in excess of 90 %WME.
IH personnel were present when the package was reopened for inspection on April 28, 2014, and the decision was made, based on the mold present, to not proceed after the upper fiberboard assembly was removed. The upper assembly was returned to the drum without further measurement or inspection.

The relative humidity at a caplug hole was 72.8% at 79.9 F. The axial gap was 0.434 inch.

**Package 9975-03373**

9975-03373 was packaged and received in KAC in January 2005. This package contained a 10 watt internal heat load. When it was opened to remove the contents on April 22, 2014, a musty odor was noted. At that time, the relative humidity in the upper air space was 75%, and the upper fiberboard assembly had 11 – 18 %WME moisture content. When it was re-inspected on April 28, 2014, no damage or degradation was observed to the drum, fiberboard or other accessible components, although minor rust stains were observed on the lid under the bolts/washers (Figure 18). The lower fiberboard assembly was tight in the drum with very little radial gap, and could not be removed by the techniques that were readily available at the time. Measurements that could be made with the lower assembly in the drum were recorded and are summarized in Table 1.

**Package 9975-02101**

9975-02101 was selected for field surveillance due to its high internal heat load (14.5 watts). It was received in KAC in June 2003, and was opened for surveillance on August 17, 2015. Non-conforming conditions observed at that time include an axial gap exceeding 1 inch (1.659 inch average), and mold on the upper fiberboard assembly. Water staining was also observed on the upper fiberboard assembly. A significant moisture gradient was observed across the fiberboard (7 to 20 %WME). Additional fiberboard data are summarized in Table 2.

This package was sent to SRNL for additional examination, which occurred on August 20, 2015. In addition to the upper fiberboard assembly mold and stains (Figure 19), the lower fiberboard assembly was moldy and the bottom layers were saturated (Figures 20 – 22). Stains were observed on top of the air shield (Figure 23).

Several areas of corrosion were observed along the bottom lip of the drum (Figures 24 – 25).

**Package 9975-02713**

9975-02713 was selected for field surveillance due to its high internal heat load (16.8 watts). It was received in KAC in November 2007, and was opened for surveillance on August 13, 2015. A non-conforming axial gap (1.484 inch average) was observed at that time. In addition, moisture was observed on top of the air shield and drum ID surface, as well as stains on these same surfaces (Figures 26, 27). A significant moisture gradient was observed across the fiberboard (< 6 to 16 %WME). Additional fiberboard data are summarized in Table 3.

This package was sent to SRNL for additional examination, which occurred on August 20, 2015. Additional observations include mold around the bottom of the lower fiberboard assembly (Figure 28). The bottom layers of this assembly were soft, and began tearing apart during disassembly. Limited corrosion was observed on the bottom lip of the drum (Figure 29).
Discussion

Of the 8 packages opened because of the facility roof leak, packages 9975-01641 and -01692 were the only two identified by NMM as showing evidence of water intrusion into the drum. Of these two, 9975-01692 was more severely impacted with significant water intrusion, as evidenced by the significant mold and water staining. When this package was first opened, the fiberboard moisture content was ~18.5 %WME (15.0 wt%) on both ID and OD surfaces, and >90 %WME in obviously wetted areas. Moisture measurements were not taken when this package was re-examined 7 months later, but significant mold and obvious wet regions were still apparent.

When 9975-01641 was first opened, the fiberboard moisture was ~11.6 %WME (10.4 wt%) on the ID and ~19.5 %WME (15.7 wt%) on the OD. When it was examined ~7 months later, the moisture content averaged 11.1 %WME (10.0 wt%) on the ID surfaces and 12.7 %WME (11.1 wt%) on the OD surfaces. One region of higher moisture content (up to 15.8 %WME, or 13.2 wt%) was measured, but most of the fiberboard in this package appears to have experienced relatively little impact from moisture intrusion. Nevertheless, the local appearance of mold may be sufficient to compromise the package long-term integrity.

Both of the packages with water intrusion would have experienced concentration of moisture originally within the fiberboard to an extent proportional to the internal heat load. However, since both packages had very low internal heat loads (~3 watts or less), the moisture concentration from this effect would be minimal. Most of the elevated moisture in these packages likely resulted from water intrusion. A large majority of axial gap measurements taken during field surveillance range from 0.6 to 0.9 inch [2]. The axial gap in 9975-01641 falls within this range, while that of 9975-01692 is significantly less. This observation is circumstantial, but suggests that the water that leaked into 9975-01692 led to swelling of the fiberboard (primarily in the axial direction) to a greater extent than the bottom layers compacted due to reduced compressive strength.

Drawing R-R2-F-0025 [3] recognizes that the axial gap dimension may vary over time due to variation in the fiberboard properties. The primary changes observed in laboratory testing include shrinkage of the sidewall region due to moisture loss and compaction of the bottom layers. Of the several axial dimensions listed in Table 1 for 9975-01641, three values (dimensions UH3, LH1 and LH2) are greater than drawing tolerances. This provides further circumstantial evidence of swelling and increased axial dimensions from the water intrusion.

While the water intrusion into these two packages has caused non-conforming conditions (mold growth, increased fiberboard dimensions), these conditions appear to have resulted solely from the roof leak. There is no evidence to indicate these packages would have been compromised had they not been exposed to the roof leak.

Packages 9975-02101 and -02713 were among a group of 10 packages selected for field surveillance due to the high heat load they contained, as well as their time in storage. Of those examined by NMM to date, only these two were identified for additional examination. Both had an axial gap well above the acceptance criterion of 1 inch maximum. 9975-02713 had condensate on the air shield and drum interior, and mold was identified on the upper fiberboard assembly of 9975-02101.
During the subsequent SRNL examination, significant mold was found on the bottom fiberboard layers of both packages along with high moisture levels (above saturation). Fiberboard dimensions LH1 and LH2 were below drawing tolerances for both packages, with the decrease in dimension LH1 about 0.5 inch greater than the decrease in dimension LH2. This comparison, combined with the axial gaps of ~0.5 to 0.7 inch above the acceptance criterion, indicates that significant compaction of the bottom fiberboard layers has occurred. In addition, both of these packages had local corrosion along the exterior bottom lip of the drum.

The conditions that developed in 9975-02101 and -02713 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 [2]. As the 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 in these locations.

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 lip – is proposed. Consider the following range of examples:

- 9975-02101 and -02713 had axial gaps of 1.689 and 1.565 inch, respectively. Both had mold and saturated moisture levels in the bottom fiberboard layers, and both had corrosion on the drum bottom lip.
- 9975-01818 had significant water intrusion, heavy mold on the fiberboard, and an axial gap of 1.437 inch. Corrosion was noted on the drum bottom lip. [4]
- 9975-01819 was examined after identification that the axial gap exceeded 1 inch. The package experienced significant water intrusion, and significant mold was present. There was no note of drum corrosion, but this was one of the earlier packages inspected with elevated moisture and drum corrosion was not a primary focus of the examination. [5]
- 9975-02130 had an axial gap of 1.099 inch, up to 24 %WME moisture content, and small patches of mold. Some corrosion was noted on the bottom of the drum, but not on the bottom lip (side). [6]
- 9975-05050 was inspected because of its high heat load, after 6.2 years in storage. The axial gap was acceptable (0.95 inch), and no mold or drum corrosion was noted. [7]
- 9975-01903 and -02287 were identified with axial gaps of 1.152 inch and 1.008 inch, respectively. 9975-01903 had small regions of mold, while 9975-02287 did not. No drum corrosion was noted for either package. [4]
- 9975-02274 and -04769 were identified with axial gaps of 1.077 inch and 1.001 inch, respectively. 9975-02274 had a musty odor and regions of dormant mold, while 9975-04769 had no mold. No drum corrosion was noted for either package. [8]
- 9975-01968, -04353 and -06870 were examined after identification that the axial gap exceeded 1 inch. Fiberboard moisture levels were normal, and no mold or drum corrosion was noted. [9]

The above examples illustrate that a number of scenarios are possible. Elevated moisture levels can occur as a result of water intrusion or from redistribution of existing fiberboard moisture. While elevated moisture levels tend to increase the axial gap, there are other causes of an increased axial gap that do not relate to fiberboard degradation - i.e. not every instance of violating the axial gap criterion was associated with elevated fiberboard moisture levels or mold. Of the packages that developed mold on the fiberboard, not all experienced drum corrosion. However, within this small sampling, all packages on which drum corrosion was noted also had fiberboard mold and failed the axial gap criterion. In one case, the drum corrosion was on the bottom only, and not visible on the side.

For packages in storage, looking for corrosion on the drum side would provide a quick screening for potential internal issues without having to open the package. For packages removed from storage, examining the drum bottom as well would provide a further check against possible internal degradation. It is recommended that NMM consider implementing these screening checks, especially for higher wattage packages.

Conclusions

SRNL has assisted in the examination of five 9975 shipping packages following storage of nuclear material in K-Area Complex (KAC). Two of the packages (9975-01641 and -01692) had experienced water intrusion, and the extent of internal fiberboard degradation (elevated moisture and mold) was documented. A third package (9975-03373) was identified for examination after a musty odor was noted during routine surveillance activities. No additional degradation was noted in 9975-03373, but the lower assembly could not be removed from the drum for detailed examination.

Two additional packages (9975-02101 and -02713) identified for further examination were among a larger group selected for surveillance as part of a specific focus on high wattage packages. These two packages displayed several non-conforming conditions, including excessive axial gap, mold on the fiberboard, and localized corrosion on the drum bottom lip. It is recommended that a new screening check for drum corrosion be implemented 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. Email message “examination of 9975 packages”, W. Daugherty to B. Hackney et al., April 28, 2014

5. SRNL-STI-2009-00240, “Examination of Fiberboard from Shipping Package 9975-01819”, W. L. Daugherty, April 2009
6. SRNL-STI-2010-00402, “Examination of Shipping Package 9975-02130”, W. L. Daugherty and J. L. Murphy, July 2010
9. SRNL-STI-2010-00233, “Examination of Shipping Packages 9975-01968, 9975-04353 and 9975-06870”, W. L. Daugherty and J. L. Murphy, April 2010
Table 1. Detailed fiberboard data for packages 9975-01641 and -03373, examined in KAC

<table>
<thead>
<tr>
<th>Upper assembly</th>
<th>Dimensions (inch)</th>
<th>Moisture content (%WME)</th>
<th>Dimensions (inch)</th>
<th>Moisture content (%WME)</th>
</tr>
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<tbody>
<tr>
<td>UD1</td>
<td>NA</td>
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<td>UD1</td>
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<td>UH3</td>
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<td>Lower assembly</td>
<td>Dimensions (inch)</td>
<td>Moisture content (%WME)</td>
<td>Dimensions (inch)</td>
<td>Moisture content (%WME)</td>
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Additional notes

<table>
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<tr>
<th>Upper air space RH</th>
<th>68.0% at 75.5 F</th>
<th>68.2% at 75.0 F</th>
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<tbody>
<tr>
<td>Axial gap</td>
<td>0.677 inch</td>
<td>0.984 inch</td>
</tr>
</tbody>
</table>

Notes: Small section of lower assembly broke during removal. Small spot of mold on lower assembly OD near top. Water stains on 1 side of upper assembly.

Very little radial gap around lower assembly, could not remove with “normal measures”. Slight musty odor on both assemblies, but no visible damage or degradation.

Diametral dimensions were measured twice, ~180 degrees apart, other dimensions were measured 4 times, ~90 degrees apart. Average values are reported. Dimensions greater than 6 inches were read to the nearest 1/16 inch (by tape measure).

Dimension UH1 includes the air shield.
Table 2. Detailed fiberboard data for package 9975-02101

<table>
<thead>
<tr>
<th></th>
<th>9975-02101 (in KAC) 8/17/2015</th>
<th>9975-02101 (in SRNL) 8/20/2015</th>
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<td><strong>Upper assembly</strong></td>
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<td>Dimensions (inch)</td>
<td>Moisture content (%WME)</td>
<td>Dimensions (inch)</td>
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<td>UH3</td>
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<td>4.976</td>
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<tr>
<td><strong>Lower assembly</strong></td>
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<td></td>
</tr>
<tr>
<td>Dimensions (inch)</td>
<td>Moisture content (%WME)</td>
<td>Dimensions (inch)</td>
</tr>
<tr>
<td>LD1</td>
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<td>LH3</td>
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<td>Additional notes</td>
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<tr>
<td>Upper air space RH</td>
<td>95.3%</td>
<td>66.3% at 80.6 F</td>
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<tr>
<td>Axial gap</td>
<td>1.659 inch</td>
<td>1.689 inch</td>
</tr>
<tr>
<td>Notes</td>
<td>Condensate staining evident on upper celotex assembly. Evidence of water stains on upper celotex. Moldy smell evident with mold located on ¼ of upper celotex outer circumference.</td>
<td>Mold on outer edge of upper assembly and corresponding areas of drum and lower assembly. Mold on bottom ~1-2 inch of lower assembly. Odor of mold/mildew on both assemblies. Bottom layers of lower assembly very soft and separated when slid out of drum. Regions of darker spots on upper assembly as from smeared glue.</td>
</tr>
</tbody>
</table>

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.
Table 3. Detailed fiberboard data for package 9975-02713

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<tr>
<th>Upper assembly</th>
<th>Dimensions (inch)</th>
<th>Moisture content (%WME)</th>
<th>Dimensions (inch)</th>
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Additional notes

- Upper air space RH: 94.6% at 80.4 F
- Axial gap: 1.484, 1.565 inch

Notes

- Moisture observed on top of upper shield assembly and on inner side drum. Stains from moisture observed on drum inside. Celotex okay. Air gap in excess of 1” all locations.
- Water spots on air shield (upper assembly), including rust stains in 1 area. Bottom ~3/4 inch of lower assembly has mold, and bottom layer split apart when removed from drum. Bottom layer very soft and wet. No odor of mold/mildew until lower assembly was removed from drum.

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.
Figure 1. Stains on 9975-01641 drum side. Photo taken 9/11/13 by NMM personnel.

Figure 2. Stains and debris on lid of 9975-01641
Figure 3. Stains on 9975-01641 drum flange and air shield. Photo taken 9/11/13 by NMM personnel.

Figure 4. 9975-01641 drum interior stains from water running down side. Photo taken 9/11/13 by NMM personnel.
Figure 5. Mold on side of 9975-01641 lower fiberboard assembly

Figure 6. Mold on bottom of 9975-01641 lower fiberboard assembly

Figure 7. Stains on 9975-01641 upper fiberboard assembly
Figure 8. Stains, debris and/or corrosion on 9975-01641 drum exterior

Figure 9. 9975-01692 drum lid with stains and debris
Figure 10. Stains on side of 9975-01692 drum. Photo taken 9/11/13 by NMM personnel.

Figure 11. Rust stains on 9975-01692 drum flange and air shield, and water on air shield. Photo taken 9/11/13 by NMM personnel.
Figure 12. 9975-01692 upper fiberboard assembly showing wetted area and mold. Photo taken 9/11/13 by NMM personnel.

Figure 13. 9975-01692 upper fiberboard assembly with mold and lead carbonate flakes.

Figure 14. Mold on 9975-01692 upper fiberboard assembly ID surface. Photo taken 9/11/13 by NMM personnel.
Figure 15. Top of 9975-01692 lower fiberboard assembly showing wetted area and mold. Photo taken 9/11/13 by NMM personnel.

Figure 16. Lead carbonate flakes on 9975-01692 shield lid
Figure 17. Light staining or possible corrosion on 9975-01692 drum exterior.

(a) Photo taken 9/11/13 by NMM personnel.

(b)

(c)

Figure 18. Rust stains on 9975-03373 lid (under bolts/washers)
Figure 19. Mold on 9975-02101 upper fiberboard assembly

Figure 20. Mold on side of 9975-02101 lower fiberboard assembly
Figure 21. Mold on side and bottom of 9975-02101 lower fiberboard assembly

Figure 22. Mold between 9975-02101 lower fiberboard assembly and drum
Figure 23. 9975-02101 air shield with water/rust stains

Figure 24. Three areas of corrosion on 9975-02101 drum exterior bottom lip
Figure 25. Region of local corrosion on 9975-02101 drum bottom lip, showing external surface (a, b) and internal surface of the same area (c).
Figure 26. Condensate and stains observed in 9975-02713 upon opening the lid. Photo taken 8/13/15 by NMM personnel.

Figure 27. Condensate and stains on 9975-02713 drum interior. Photo taken 8/13/15 by NMM personnel.
Figure 28. Mold around the bottom of 9975-02713 lower fiberboard assembly, and damage to layers from removal

(a)

Figure 29. Corrosion on the bottom lip of 9975-02713 drum on the exterior (a) and matching region of the interior (b)
CC:  R. J. Bayer, 705-K
     J. S. Bellamy, 730-A
     G. T. Chandler, 773-A
     W. L. Daugherty, 773-A
     K. A. Dunn, 773-41A
     B. A. Eberhard, 105-K
     L. F. Gelder, 999-W
     T. W. Griffin, 705-K
     T. J. Grim, 105-K
     E. R. Hackney, 705-K
     E. V. Henderson, 705-K
     J. M. Jordan, 705-K
     D. R. Leduc, 730-A
     J. W. McEvoy, 707-C
     T. E. Skidmore, 730-A
     D. E. Welliver, 705-K
     K. E. Zeigler, 773-41A

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