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# EVALUATING CORROSION OF NUCLEAR MATERIAL STORAGE CONTAINERS AND THE IMPACT ON CONTAINER LIFETIME

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## ABSTRACT

Surveillance of vented, stainless-steel Hagan and SAVY-4000 storage containers packaged with plutonium materials has revealed evidence of corrosion inside the container. There are approximately 3,000 items stored in Hagan containers and approximately 1,200 items currently stored in SAVY-4000 containers at TA-55. The Hagan container is made from 304L steel, while the SAVY-4000 is made from 316L and should be less susceptible to corrosion. Given the relatively thin body wall of these containers (nominally 762 microns), it is essential to understand the effects of corrosion on the lifetime of the containers to ensure worker safety. Studies are being conducted on both container types to determine the relative susceptibility of the two types of containers to corrosion. This work describes the cause of corrosion, the extent of corrosion, and types of corrosion observed, along with the results of accelerated aging experiments. The combined results of the surveillance and lifetime extension studies establish a technical basis for extending the design life of the stainless steel components of the SAVY-4000 container from 5 years to 15 years.

## INTRODUCTION

Department of Energy (DOE) Manual 441.1-1, *Nuclear Material Packaging,* requires that nuclear material packages outside of an approved engineered contamination barrier meet packaging, surveillance, and testing requirements designed to protect workers from airborne contamination[1]. The SAVY-4000 containers were developed at Los Alamos National Laboratory (LANL) in conjunction with Nuclear Filter Technology as a manually-compliant container system for staging and storage of plutonium for LANL and the DOE complex. The SAVY-4000 containers have a 316L stainless steel containment barrier, which was selected based on its corrosion resistance properties. These containers were approved for use in 2014 with a five-year design life. A surveillance program for those containers was established per DOE M 441.1-1 requirements to ensure that packages continue to meet their design criteria. The surveillance program evaluates the aging of the container components during the storage lifetime. Surveillance includes a visual inspection with the objective of identifying early indications of package degradation.

Selection of containers for surveillance is documented in the field surveillance plan[2]. Container selection relies on engineering judgement to identify packages exposed to the most aggressive storage conditions with respect to radiation dose and potentially corrosive contents. The surveillance plan is updated each year based on the previous years' findings. Surveillance of

SAVY-4000 containers began in 2015, and corrosion was observed on two of ten SAVY-4000 storage containers after only one to two years of storage. Corrosion was also found on two SAVY-4000 containers used for short-term storage packaged with flasks of plutonium dissolved in hydrochloric acid solution. The visual inspection showed general corrosion covering inside surfaces and loose corrosion product deposited on the container bottom.

Concern that corrosion could affect the design life of the container components led to the formation of a corrosion working group tasked with determining the conditions that support corrosion of the stainless steel components and the impact of that corrosion on the containers. These tasks were accomplished by laboratory studies to evaluate the susceptibility of the container components to corrosion, comprehensive examinations of the corroded container components to determine the extent and severity of the corrosion, and drop testing to evaluate the container performance. This report summarizes the results of the corrosion studies and the impact of the corrosion on the design life of the container.

## CORROSION OBSERVATIONS IN SURVEILLANCE

SAVY-4000 and Hagan storage containers are the primary interim storage containers for nuclear materials at the Los Alamos National Laboratory Plutonium Facility. Both containers are packaged according to the requirements for allowed content and configuration defined in the SAVY-4000 Safety Analysis Report (SAR)[3]. A typical storage configuration consists of a metal inner container placed inside of a polyvinylchloride (PVC) bag, which is placed inside a storage container. The exterior surface of the PVC bag must be contamination free at the time of packaging. The SAVY-4000 SAR does not allow the packaging of certain challenging contents including any liquids, any gases, and corrosive, pyrophoric, or pressure generating solids. Local facility requirements do allow packaging of contents that are not allowed by the SAR for up to one year in short-term storage containers, referred to "transfer containers". These transfer containers must be unpackaged yearly for a maintenance cycle to ensure that they continue to perform their design function.

To date, 78 field surveillances have been performed on 27 SAVY-4000 and 37 Hagan storage containers. Additionally, 78 maintenance cycles have been performed on transfer containers. Corrosion was observed in 13 of the SAVY-4000 containers, in 20 of the Hagan containers, and in three of the transfer containers. Containers are assigned a group based on the overall appearance and coverage of the corrosion or coating. The group can be used to compare the corrosion behavior for various material forms and material types as well as to assess the progression of corrosion over time in containers that have yearly surveillances.

Table 1 defines the criteria for each corrosion group and gives the number of SAVY-4000 and Hagan containers in each group based on the criteria. Eight containers (five Hagan containers, one SAVY-4000 storage container, and two SAVY-4000 transfer containers) were assigned to corrosion group 3 based on the visual inspection. The conditions and packaging data for the containers assigned to corrosion group 3 are listed in Table 2 along with additional analyses recommended by the corrosion working group. Photos of select containers are shown in Figure 1.

**Table 1. Corrosion Group Descriptions and Number of Storage Containers Assigned by Group**

| Group | No. Containers SAVY | Hagan | Description | Criteria |
|---|---|---|---|---|
| **0** | 27 | 16 | No Corrosion | No corrosion, staining, spots, or coatings observed |
| **1** | 6 | 6 | Isolated General Corrosion | Corrosion, staining, spots, or coatings observed in isolated areas (e.g. corrosion found on weld only) |
| **2** | 6 | 9 | Light General Corrosion | Corrosion, staining, spots, or coatings throughout container; light in overall density; bare metal visible |
| **3** | 1* | 5 | Heavy General Corrosion | Corrosion, staining, spots, or coatings throughout container; heavy (dark) in overall density; little or no bare metal visible |
| *Does not include 2 SAVY-4000 transfer containers assigned to corrosion group 3. | | | | |

**Table 2. SAVY-4000 and Hagan Storage Containers in Corrosion Group 3 (Containers 20T and 21T are SAVY-4000 transfer containers).**

| ID / Type / Size | Material Form Description | Nuclear Material Description | Age (y) | Power (W) | Visual Inspection | Additional Analyses |
|---|---|---|---|---|---|---|
| **15H3**<br>H<br>8-Qt | Dioxide | Plutonium, >19.00% Pu-240 | 5.6 | 5 | • Sticky residue covering most inner surfaces | • None, however, the material was placed in SAVY-4000 for yearly surveillance |
| **15H4**<br>H<br>8-Qt | Dioxide | Plutonium, 16.00 thru 19.00% Pu-240 | 11.5 | 10.5 | • Corrosion and heavy coating covering most inner surfaces | • Chemical analysis of coating |
| **16H1***<br>H<br>5-Qt | MSE Salt | Plutonium, 10.00 thru 13.00% Pu-240<br>Americium; Am-241 | 8.1 | 2.9 | • White powder on filter cover and on storage compartment<br>• Wipeable coating covering inside surface | • Chemical analysis of white powder<br>• Gas generation<br>• Optical microscopy |
| **16S8**<br>S<br>8-Qt | Dioxide | Plutonium, 4.00 thru 7.00% Pu-240<br>Plutonium-238 (7%) | 3.4 | 21.3 | • Corrosion of TID wire and deposit of corrosion product on lid<br>• Wipeable coating covering inside surface | • Chemical analysis of coating |
| **18H7***<br>H<br>1-Qt | MSE Salt | Plutonium, 4.00 thru 7.00% Pu-240<br>Americium; Am-241 | 13 | 6.9 | • Corroded threads; lid fused to body<br>• Green-brown liquid covering inside surface | • Optical microscopy |
| **19H5***<br>H<br>1-Qt | MSE Salt | Plutonium, 4.00 thru 7.00% Pu-240<br>Americium; Am-241 | 12.3 | 5.7 | • Corroded threads; lid fused to body<br>• Corrosion product powder covering walls and bottom | • Optical microscopy |
| **20T****<br>S<br>5-Qt | Liquid; Chloride Solution | Plutonium; various | 1.2 | -- | • White powder on lid surrounding filter<br>• Corrosion product flaking off walls and covering bottom | • Wall thickness<br>• Laser confocal microscopy |
| **21T****<br>S<br>5-Qt | Liquid; Chloride Solution | Plutonium; various | 1.2 | -- | • White powder on lid surrounding filter<br>• Corrosion product flaking off walls and covering bottom | • Wall thickness<br>• Chemical analysis of corrosion product<br>• Drop testing |
| Abbreviations: S: SAVY-4000; H: Hagan; Qt: Quart<br>* Container introduced into glovebox line for unloading ** SAVY-4000 transfer container. | | | | | | |

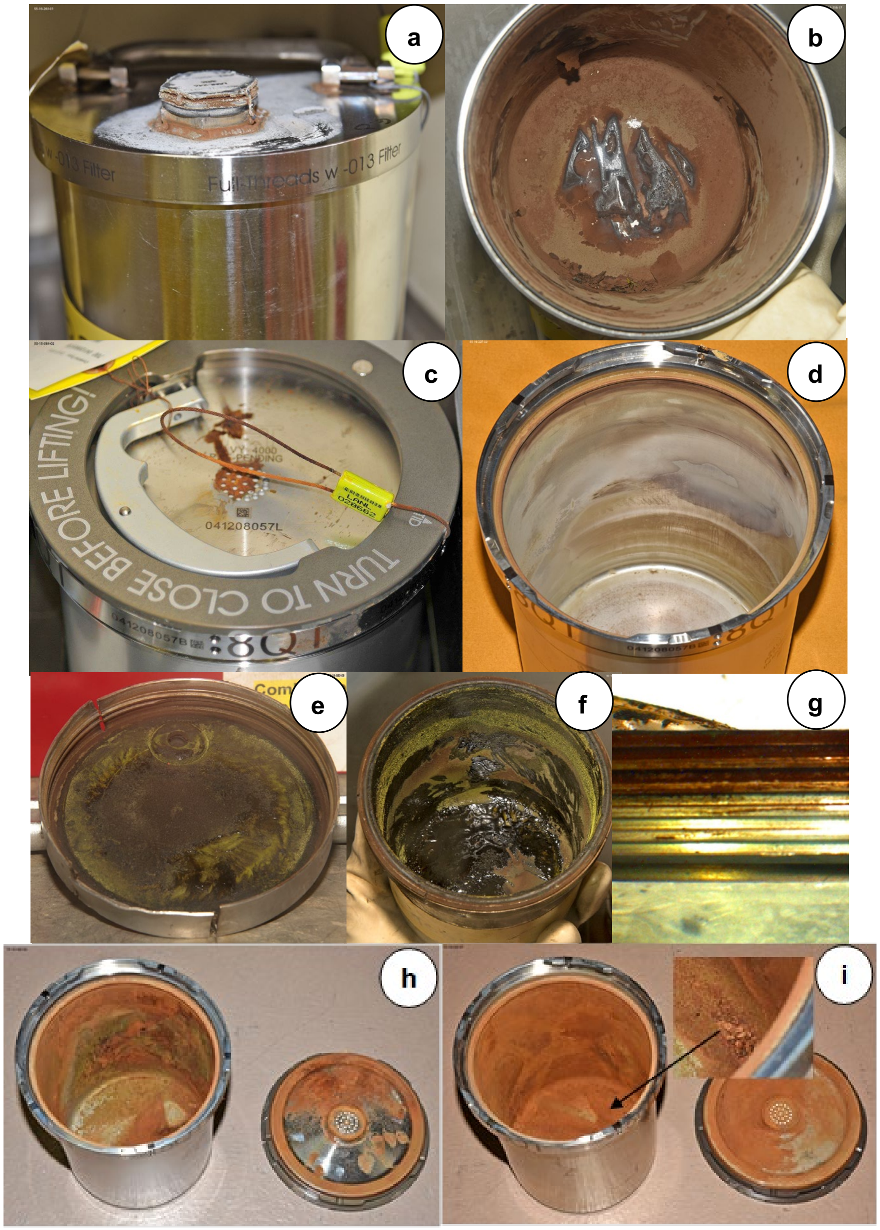

**Figure 1. Photos of select containers assigned to corrosion group 3 based on the visual inspection: (a) 16H1 lid with white powder deposit and corrosion on filter cap, (b) 16H1 inside surfaces have a brown coating, (c) 16S8 has corrosion product deposited on outside of lid from the corroded TID wire above, (d) 16S8 inside surfaces covered in brown and white powders, (e) 18H7 inner surface of lid covered in gummy, green-brown residue, (f) 18H7 inner surfaces of body with gummy, green-brown residue, (g) 18H7 corroded threads, (h) corrosion and loose brown powder on inside surfaces of 20T, (i) corrosion and loose brown powder on inside surfaces of 21T.**

All of the containers listed had coatings covering most or all of the interior surfaces. Most coatings on the inside surfaces consisted of dry powders or flakes, ranging in color from brown to white and removable by wiping with a wet cloth. Several containers had accumulated loose powder on the bottom of the containers. The powders from two containers were collected and weighed. Powder samples in the amounts of 4.2 grams and 1.9 grams were from containers 19H5 and 20T, respectively.

Several containers were found to have white powder deposits outside the container. Container 16H1 (Figures 1a and 1b) was found to have white powder covering outside surface of the filter cover and part of the Hagan container lid as shown in Figure 1a and 1b. Additionally, white powder was also deposited on the surfaces of the storage compartment and on the window. The inside surfaces of container 16S8 were found to have a mixture of brown and white powders (Figures 1c and 1d).

Analytical chemistry was performed on the white and brown powders collected from the containers. The white powder from the filter cap of 16H1 was found to be ammonium chloride. The brown powders collected from the inside surfaces were mostly a mixture of metal chlorides. The ratio of the metal cations in the powder corresponds to the ratio of the metals in stainless steel. The powder also contained small amounts of ammonium chloride. The ammonium chloride is a reaction product of hydrogen chloride (HCl) gas and ammonia, which are formed by chemical and/or radiolytic processes. Ammonia is formed by chemical and/or radiolytic processes with hydrogen and nitrogen in the presence of gamma and alpha radiation[4,5]. The possible sources of the HCl gas include the PVC bag and certain packaged materials containing chloride salts. The sources of HCl are described later in this report.

Several of the containers showed evidence that liquid had been present in the past or had liquid present at the time of opening. The inside surfaces of 18H7 (Figure 1e, 1f) were completely covered by a gummy residue that ranged in color from green to brown. The liquid was presumed to be plasticizer from the PVC bag mixed with corrosion product.

Corrosion was observed outside of the sealing surface of four containers including 16H1, 16S8, 18H7 and 19H5. Corrosion was observed on the filter cap of 16H1 and on the exterior of the lid of container16S8 (Figure 1c and 1d). The corrosion was found to be staining that resulted from corrosion of the galvanized steel tamper indicating device (TID) wire above the lid. It was presumed that the wire corroded due to exposure to the HCl gas during a humid event in the storage location. Two Hagan containers, 18H7 (Figure 1g) and 19H5 (not pictured), had corrosion on the threaded lid resulting in the lids being fused to the container. These results are consistent with corrosive gases escaping the container past the O-ring.

Additional analyses were performed on several of the corroded containers to assess the extent and severity of the corrosion. Wall thickness measurements were performed on the two SAVY-4000 containers with the heaviest corrosion (20T and 21T) to determine the effect of the general corrosion on the container body. Both containers were examined with a coordinate measuring machine and an ultrasonic wall thickness instrument. Neither instrument detected any significant change in wall thickness when compared with the variability in the wall thickness of a new, unused container.

Examination of the containers for pitting and stress corrosion cracking (SCC) was done by optical microscopy or by laser confocal microscopy (LCM). The LCM technique produces three-dimensional images of surface features and measures the height and depth of features. This technique can only be performed on container specimens approved for removal from the facility. Optical microscopy is available to examine specimens taken from containers inside the glovebox line. Estimates of the depth of features observed in optical microscopy were obtained by measuring travel of the stage when changing the focus from the top surface to the bottom of a feature.

## Optical Microscopy

Sections of the Hagan containers 16H1 and 18H7 were obtained from the lid, weld, sidewall, and bottom for examination[6]. No evidence of SCC was observed in any of the locations examined on either container. The main features were general corrosion and pitting. In the examination of 16H1, pitting was observed on the lid (5 to 15 microns in diameter) and on the sidewall in areas that were stained due to contact with the PVC bag during storage. The maximum diameter of any circular pit observed was 105 microns, and the maximum depth of the largest pit was 40 microns. The remaining areas showed general corrosion. In the examination of 18H7, mostly general corrosion was observed with occasional pitting. Many of the pits were shallow and agglomerated. Individual pits had diameters of approximately 100-160 microns and estimated depths of 20 to 40 microns (less than 5% of the total wall thickness).

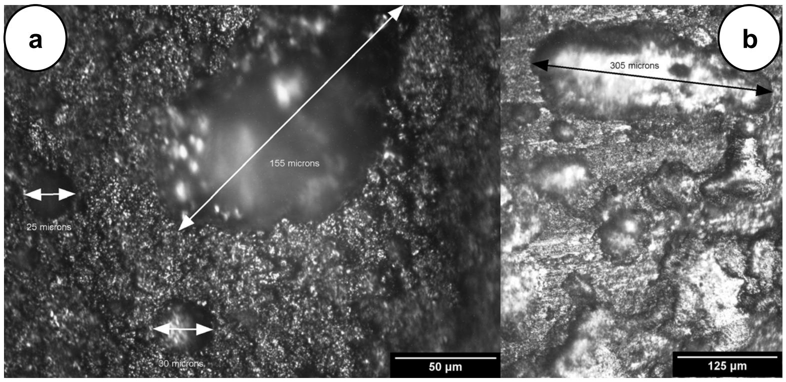

**Figure 2. Optical microscopy images of Hagan containers 16H1 and 18H7. (a) 16H1 sidewall with pits with diameters measuring (clockwise from left) 25 microns, 155 microns, and 30 microns (b) 18H7 sidewall with agglomerated pit 305 microns wide.**

## Laser Confocal Microscopy

Laser confocal microscopy was performed on SAVY-4000 transfer container 21T. Images are shown in Figure 3. Pitting was observed in the region surrounding the weld[7]. The depth of the pits was between 30 and 40 microns. The pits nearest to the weld were elongated in the vertical direction; whereas, the pits further from the weld were round in shape. A cross sectional sample was taken at the weld region and showed small, transgranular cracks originating from the bottom of the elongated pits. The crack length was approximately 30 microns. The combined depth of the pit and crack was approximately 60 to 70 microns (less than 10% of the total wall). No through-wall cracks were observed.

SAVY-4000 container 20T was cleaned and subjected to a container integrity evaluation, consisting of a series of drop tests according to the Acceptance Test Plan in the SAVY-4000 SAR[3]. The drop test was performed on container 20T four consecutive times from a height of 12 feet. A leak test was performed and showed that the helium leak rate of the container remained well below the failure criteria of $1.00 \times 10^{-5}$ atm·cc/sec with a mean leak rate of $1.65 \times 10^{-8}$ atm·cc/sec[8]. These results indicate that no through wall penetrations occurred and that these containers still perform their design function.

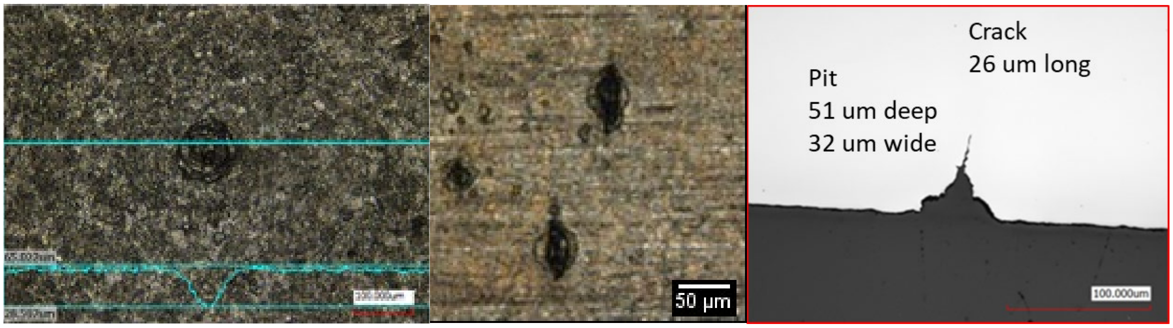

**Figure 3. LCM of SAVY-4000 container 21T showing round pit below weld area (left), elongated pits near weld (middle), and crack originating from bottom of elongated pit (right)[7].**

## CONDITIONS SUPPORTING CORROSION

Storage containers are packaged so that the nuclear material contents are not in direct contact with the inside surfaces of the outer container. Therefore, the corrosion that is observed in the storage containers is the result of reactions between corrosive gases and the surface of the containers. The primary source of the corrosive gases is the dehydrochlorination of PVC. Gamma irradiation of PVC causes degradation of the polymer backbone (double-bond formation, polymer cross-linking and chain scission). This results in PVC discoloration, brittleness and loss of strength as well production of HCl gas[9]. Alpha irradiation also causes dehydrochlorination of PVC, especially in combination with high temperature[10]. Production of HCl gas increases with increasing beta/gamma dose. This is consistent with observations of highly degraded bags in packages high in Am-241.

A second source of HCl gas is the packaged material, particularly those that contain chloride salt impurities. A gas generation study was performed on the material originally packaged in 16H1 to determine whether corrosive gases are produced and how the packaged material affects the environment inside the container[11]. In the first phase, the material was sealed in a glass container for 50 days with a compound that captures chlorine-containing gases, such as HCl and $Cl_2$, by chemical reaction. Analysis of the product compound after exposure showed that chlorine-containing gases were not detected in the original material. However, HCl gas was detected in small amounts after the material was hydrated to 0.5 wt.% moisture (and later to 2.0 wt.% moisture) by exposure to 30% relative humidity. These results show that the material in its packaged form was not (or no longer) generating corrosive gases, and the corrosion observed in the container likely resulted from PVC degradation. In the second phase of the experiment, the material, hydrated to 2.0 wt.% moisture was loaded into a, stainless-steel conflat container instrumented with sensors that measure pressure, temperature and relative humidity for a period of 56 days[12]. The container parts were inspected upon completion, and no corrosion was observed. Although it was shown that the material can generate HCl in its hydrated form, the relative humidity in the sealed container, which was less than 1% for the duration of the experiment, was too low to support corrosion. These results show that packaged materials are not a significant source of HCl gas. Further, the atmosphere of LANL gloveboxes are maintained at less than 1% relative humidity, mitigating the potential for moisture adsorption.

Hydrogen chloride gas causes uniform or general corrosion on both 304L and 316L stainless steels at room temperature. Under dry conditions, the resistance to general corrosion on both 304L and 316L stainless steels is considered good at room temperature and poor at 400 °C[13]. The presence of water vapor increases the severity of the corrosion by HCl at lower temperature[14]. Accelerated aging studies with stainless steel type 304L teardrop specimens exposed to HCl gas at 0.15 Torr and 31, 48, and 68% relative humidity at room temperature have demonstrated that corrosion is more aggressive at higher relative humidity causing pitting as well as general corrosion. The relative humidity and temperature in the storage rooms at the LANL plutonium facility is typically between 20 to 25 °C and below 35% relative humidity. Additionally, the radiative self-heating of the container due to the plutonium isotopes would lower the relative humidity inside the container with respect to the room, further mitigating the effect of the moisture. SAVY-4000 transfer containers 20T and 21T were an unusual condition in that solutions were packaged in a closed container. In a closed container, the relative humidity would reach a maximum of 80% with a maximum HCl partial pressure of 0.15 Torr. This condition is considered to be outside the scope of the SAVY-4000 SAR.

## SUSCEPTIBILITY OF CONTAINERS TO SCC

The greatest concern with respect to the corrosion of SAVY-4000 and Hagan containers is a through wall crack resulting from SCC of stainless steel. Based on the recommendations of the corrosion working group, a series of tests were performed to determine the susceptibility of SAVY-4000 and Hagan containers to SCC. The boiling magnesium chloride test (ASTM G36) determines relative susceptibility of metal objects to SCC, by exposure to a solution of magnesium chloride that boils at 155 °C until failure is observed [15]. One SAVY-4000 container and one Hagan container were tested, and failure of the Hagan occurred between 22-24 hours due to through wall cracks in the region where the body is welded to the collar. Failure of the SAVY-4000 container occurred between 44-46 hours and resulted from a through wall pit at the bottom of the container, but no through wall cracks were observed[8]. These results indicate the region of concern for the Hagan container is the weld region and that the Hagan container is more susceptible to SCC than the SAVY-4000. Based on the cracking observed in the Hagan container near the weld, residual stress measurements were performed to compare the residual stresses in the weld region of the SAVY-4000 and the Hagan containers. The measurements were performed according to the hole drilling technique described in ASTM E837[16]. The measured tensile stress for the Hagan container was a hoop stress of 194 MPa; whereas, the measured tensile stress for the SAVY-4000 was an axial stress of 25 MPa[8]. The residual stresses in the SAVY-4000 container are lower than the residual stresses measured in the Hagan due to annealing. These results are consistent with the boiling magnesium chloride test that show that Hagan containers are more susceptible to SCC in the weld region than SAVY-4000 containers.

Accelerated aging studies are being conducted under similar conditions with actual SAVY-4000 and Hagan containers and specimens. Preliminary results have shown cracking in a Hagan container specimen after 126 days of exposure to the vapor above a 6M HCl solution[17]. A SAVY-4000 container and specimens exposed to the same conditions show pitting corrosion. These results show the higher susceptibility of Hagan containers to SCC.

## IMPACT OF CORROSON ON CONTAINER LIFETIME

As of 2019, the SAVY-4000 containers have reached their 5-year design life in the SAVY-4000 SAR, and LANL has proposed a lifetime extension from 5 to 15 years based on observations from surveillance and accelerated aging studies[18]. Surveillance observations of general corrosion and pitting in storage containers packaged with some of the most challenging contents allowed by the SAR indicate that the rate of corrosion would not lead to through-wall penetration within a 40-year period. The surveillance observations include Hagan containers that have been packaged for up to 17 years. The corrosion observations in Hagan containers is expected to be more aggressive based on the corrosion rates for 316L and 304L stainless steel in the literature. Although observations of containers exposed to HCl gas and high water vapor suggest that SCC could be a concern under extreme conditions, evidence of SCC has not been observed in the surveillance of storage containers either through visual inspection or through helium leak testing.

The surveillance program supports the lifetime extension through engineering judgement, which targets SAVY-4000 and Hagan containers expected to have the most challenging contents to gain insight on the progression of corrosion in storage containers and to identify early indications of container failure. The surveillance program is guided by observations and modified as necessary to target the appropriate materials. Future consideration of a 40-year lifetime depends on the results of the surveillance program, accelerated aging studies, and possible replacement of the PVC bags used in storage containers. Aromatic polyurethane (APU) materials, both APU-ether and APU-ester, have undergone accelerated aging studies by exposure to heat and gamma radiation, and the toughness of the APU was found to be twice that of PVC in both the pristine and aged conditions[19]. Replacement of PVC with APU would remove the largest source of HCl and drastically reduce the potential for container failure due to SCC. Operational testing of these bags is currently underway.

## CONCLUSION

General corrosion and pitting has been observed in SAVY-4000 and Hagan storage containers in the surveillance program at LANL. Wall thinning due to general corrosion was shown to be insignificant. Based on the depth of the largest pit observed (40 microns) in a Hagan storage container, the maximum penetration of the sidewall was approximately 5% after 8 years and would not result in a through wall penetration in 40 years. The 316L stainless steel SAVY-4000 container bodies have lower rates of corrosion under the same conditions and are less susceptible to SCC due to lower residual stresses. Cracking observations from Hagan containers in accelerated aging studies and in containers with material contents outside of the scope of the SAVY-4000 SAR have resulted in a conservative recommendation to extend the lifetime of the metal container components from 5 to 15 years. Future consideration of a 40-year lifetime for the container will depend on data collected from the surveillance program, accelerated aging studies, and a possible replacement for the PVC bag material.

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