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Towards establishing a service life for the SAVY-4000 nuclear material storage container at Los Alamos National Laboratory

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Abstract

The SAVY-4000 nuclear material storage container has been in use at Los Alamos National Laboratory (LANL) as a DOE Manual 441.1-1 compliant container since 2014. The most common packaging configuration for storage containers involves a nested configuration of a metal inner container, bag-out bag, and SAVY-4000 outer container. Polyvinyl chloride (PVC) bag-out bags are used to control radiological contamination when the inner container is bagged out of the glovebox. This generally prevents contamination of the inside of the SAVY-4000 container and allows them to be re-used. In the past few years, however, corrosion of the SAVY-4000 has been observed through annual container surveillance efforts. One source of this corrosion arises from the degradation of the bag-out bag resulting from thermal and radiolytic degradation pathways. The subsequent production and release of hydrogen chloride gas corrodes the stainless-steel container wall. Corrosion rates of the 316L stainless steel and, consequently, the service life of the SAVY-4000 depend heavily on this bag-out bag degradation rate, in addition to other environmental and packaging factors, such as temperature and relative humidity. Our recent efforts to establish a service life for the SAVY-4000 comprises three unique activities: 1) Surveillance of nuclear material storage containers including the SAVY-4000 and its predecessor containers with longer service lives, 2) systematic corrosion experiments on coupons to understand controlling mechanisms of corrosion, and 3) accelerated aging experiments to establish corrosion thresholds as related to container performance. Details of the projected path toward lifetime evaluation and possible extension are highlighted, as well as an overview of the latest corrosion analysis efforts.

Introduction

The SAVY-4000 series of nuclear material storage containers was developed to ensure Los Alamos National Laboratory (LANL) nuclear material storage safety and compliance to facility and regulatory requirements. The Department of Energy (DOE) Nuclear Material Packaging Manual, DOE M 441.1-1 (The Manual) prescribes a facility agnostic set of requirements for short term nuclear material storage containers. The SAVY-4000 family of containers was developed because of worker dose uptake due to release from a less-than-adequate material packaging configuration. As a more robust containerization package, the SAVY-4000 series meets or exceeds criteria outlined in The Manual in addition to those specified at facilities.

A large effort encompassed the creation and implementation of the SAVY-4000 nuclear material storage containers. With roots from the Department of Transportation's type B packages, the SAVY-4000 Safety Analysis Report (SAR) was created. This SAR analyzed possible accident scenarios to ensure that the containment boundary of the SAVY-4000 was not compromised under bounding conditions [1]. Extensive testing and documentation of the results were included within the SAR. This SAR was accepted by the Los Alamos Field Office on April 16th, 2014 [2], making the SAVY-4000 series a Manual compliant container and establishing a lifetime of 5 years for containers. Further extensions to the service life up to and beyond the initial design life intent (40 years) would require compelling justifications derived from both the surveillance of containers committed to storage conditions and appropriate accelerated aging studies.

Two main components of the SAVY-4000 container, the Viton® O-ring and the Fiberfrax™ filter with a polytetrafluoroethylene (PTFE) external scrim with a base of polyethylene (PE) for structural support, were initially determined to be the most likely area of containment boundary failure. The O-ring provides a piston seal of the container, ensuring nuclear material particulates do not escape. The filter ensures particulates do not escape while also allowing for the diffusion of gasses that are generated during radiolysis and decay of nuclear material. The PTFE scrim layer provides a one-way hydrophobic barrier to ensure liquid water does not enter the filtered container, a criticality safety requirement of some locations of PF-4. Viton® and PTFE were assumed to be the least robust materials on the container as all the containment boundary metal components consist of passivated 316L stainless steel. This material was chosen due to its corrosion resistance compared to 304L, the construction material of the previous Hagan container.

Initial Lifetime Extension and Technical Basis Generation

One aspect of lifetime determination for these components utilized accelerated aging experiments. These experiments varied temperature, time, and radiation dose on the materials in question [3,4]. These experiments characterized aging as well as significant material science, specifically the O-ring material. In addition to small scale experiments, whole container aging was conducted. These full-sized containers were tested against the fabrication test methods, mainly water penetration, filter efficiency and helium leak testing. The results of these experiments yielded a suggested a lifetime of 40 years. The data showed a minimum of 65 years, but to be conservative, a 40-year lifetime was recommended. In addition to evaluating O-ring and filter lifetime, an additional test and requirement for the O-ring during surveillance testing was added to the surveillance plan and procedure. The measurement of durometer and ensuring a compression set less than 65% is required on all surveillance containers. This compression set was determined, based on the experimental data, to still be able to seal the container according to helium leak testing.

Additional experiments examined the failure mechanism of the O-ring, specifically looking at how wear and tear and debris might cause degradation and thus leak pathways [5]. It was confirmed that a single human hair would cause a leak rate failure when wrapped around the O-ring. However, it was shown that the precise placement of the hair on the O-ring was necessary to provide a failing leak path. Debris was also considered within the study leading to evidence that larger debris particles result in larger leak paths. This was confirmed through application of silica gel (large debris particles) and NIST standardized Arizona road dust (fine particles). Only the silica gel particles caused the container to fail the helium leak criteria of 1×10^{-5} atm cm^3 s^{-1}. Wear and tear on the O-ring through 100 opening and closings of the SAVY were conducted. The leak rate was independent of the number of closures indicating that degradation through nominal use is not an issue. With this study in mind, procedural steps for O-ring examination before closure of a SAVY have been written and are in place. Procedures have been established to ensure a clean O-ring before installation and is an important step of the closure process to minimize the influence of contaminants on the seal.

Polymer degradation was examined under elevated temperature and radiological conditions, including gamma and alpha irradiation [6]. Multiple polymers were analyzed in the study, but the three chosen were polyvinyl chloride (PVC, bag-out bag material), polyethylene (base of scrim layer on the filter), and PTFE (provides hydrophobicity of the scrim layer). Thermogravimetric analysis (TGA) of PVC showed thermal degradation initiated around 180°C but the literature has also shown degradation to begin as low as 140°C. A proposed unzipping dehydrochlorination mechanism of the polymer chain is the suspected degradation mechanism which causes discoloration, brittleness, and loss of strength. Additionally, PVC degradation results in the off gassing of gaseous hydrogen chloride (HCl). HCl is reactive to the passivated 316L stainless steel and produces two readily observable corrosion products on the surface of exposed components: iron (II) chloride and iron (III) oxide.

Both polymers, PE and PTFE, showed resistance to 5 Mrad gamma irradiation based on contact angle measurement and FTIR. Contact angles allude to slightly better gamma resistance for PTFE, but not significantly enough to decipher any real degradation of the polymer. However, alpha exposure proved a different story. Samples were aged under differing fluxes of alpha irradiation in an alpha beam line experiment up to a total dose of 5×10^{14} α cm^{-2}. In these highest dose experiments, PTFE showed signs of degradation through contact angle measurement, FTIR, and SEM imaging. Severe cracking, shown in Figure 1, was observed alongside chemical changes evident in PTFE. Mechanical degradation in the irradiated PE specimens was the only measurable change. These results suggest that if the filter (external side of the containment boundary) is not exposed to alpha radiation, the filter and its hydrophobicity will not be degraded within the SAVY-4000's current lifetime; and a 40-year lifetime to match the O-ring suggested lifetime is appropriate.

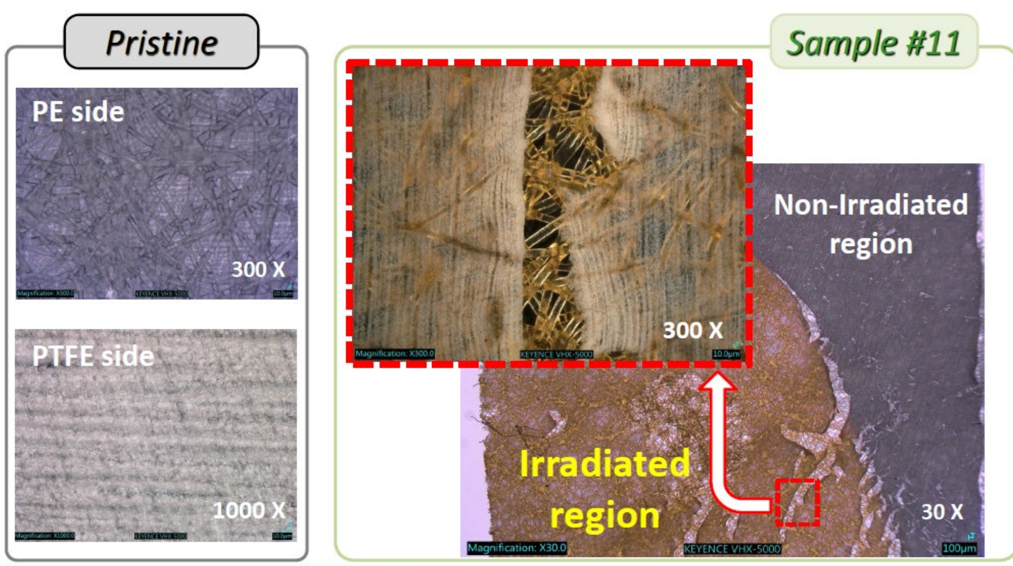

Figure 1. Images show pristine (left) to alpha irradiated filter material (right). The tear on the PTFE side is a visible indication of degradation.

In conjunction with accelerated aging studies, annual surveillance of both Hagan and SAVY-4000 provided in-situ evidence of aging conditions. Hagan containers are not a direct comparison as they are constructed out of 304L stainless steel but have been in service since 1999 and are another avenue to evaluate of potential degradation pathways of storage containers. In 2016, a Hagan was selected by engineering judgement due to the white powder collecting near the filter. When opened, the interior showed signs of corrosion, and the bag-out bag was heavily darkened and weakened, as shown in Figure 2. The presence of corrosion presented a significant challenge to the ability to justify additional design life extensions for the SAVY-4000.

Figure 2. Heavily corroded Hagan container after 8.1 years. From left to right – the inside of the Hagan lid, the interior of the Hagan, the external second bag-out bag, the internal first bag-out bag.

A corrosion working group (CWG) was established to help evaluate the effects of corrosion on SAVY-4000 containers. This team was formed of people experienced with the 3013 container corrosion issues as well as people working with SAVY-4000 and Hagan containers. Various studies on used and pristine containers were conducted through the corrosion working group [7]. Three main corrosion failure mechanisms were identified: general corrosion, pitting corrosion, and stress corrosion cracking (SCC). Material stresses influence SCC, so the residual stress was measured using through hole drilling measurements 4mm below the weld region for both the SAVY-400 and Hagan container. The Hagan showed significantly more stress, leading to the conclusion that it is more susceptible to SCC. Boiling $MgCl_2$ tests were conducted on pristine SAVY-4000 and Hagan containers. Through-wall cracks were observed after 22-24 hours of $MgCl_2$ near the weld region for Hagan containers, leading to containment failure. Failure of the SAVY-4000 container was observed by through-wall holes in the bottom of the container after 44-46 hours. Cracks observed near the weld of the SAVY were watertight, but it was not confirmed that these would pass a helium leak test. These tests support evidence that the SAVY-4000, while still susceptible to SCC is more resilient than Hagan containers.

Surveillance of Hagan and SAVY-4000 containers showed that both containers exhibited corrosion. The identified worst case for Hagan containers so far has been the "white powdered" Hagan discussed earlier. This molten salt extraction (MSE) residue was contained for 8.1 years in a Hagan and showed external signs of off-gassing in the form of a white powder observed near the filter and on the surfaces of the storage shelf. This white powder was analyzed and determined to be ammonium chloride [8]. The residue material was split to measure off-gassing utilizing an experiment designed from the Materials Identification and Surveillance (MIS) Working Group under the DOE Standard-3013 program. It was observed that off gassing of material only happens when enough water vapor is present to undergo radiolysis. Due to the presence of a filter on the

Hagan and SAVY-4000 container, there is enough water absorbed from the air in PF-4 to allow these reactions to take place.

This Hagan container underwent corrosion analysis. Unfortunately, to unpack this item the container had to be introduced into the glovebox line and precluded typical surveillance testing. However, a sectioning saw was available and 3 representative samples were cut from the Hagan. These samples were cleaned in 2M nitric acid for 30 minutes, washed in DI water and dried. This cleaning method has shown to not remove base material or affect the shape of corroded pits within the subsurface. An optical microscope was used to observe the cleaned surface and pitting of the material. The worst pitting present was 40 µm deep and 105 µm in diameter. Assuming a linear growth rate, this results in a pit penetration rate of 5 µm per year. The nominal wall thickness of a Hagan container is 508 µm, meaning that only ~8% of the wall was penetrated over an 8-year period and through-wall pitting would occur in >100 years, assuming a linear growth model. More recent efforts have been initiated to establish more physically accurate pit growth models.

The worst cases of corrosion in SAVY-4000 containers were observed in solution assay instrument (SAI) calibration solutions (plutonium in 3M hydrochloric acid), MSE residue, and a "hatch" item. Hatch is high purity plutonium dioxide mixed with 7% Pu-238. The MSE residue, hatch and SAI items passed all surveillance testing. The SAI solution container showed large amounts of general corrosion, seen in Figure 3. This corrosion product was easily wiped away with cleaning solution. The base surface showed that the laser-etched barcode and serial number were visible on the interior surface of the container. To evaluate whether the etching provides an area likely for corrosion, small samples of SAVY-4000 containers and Hagan containers underwent etching at different speeds, powers, and frequencies [9]. These samples were subjected to Iron (III) chloride to induce pitting in the samples. Based on the results, the etching did not play a role in pitting characteristics or rate.

Figure 3. On the left, the SAVY-4000 that contained the SAI solution. This solution was in a plastic Erlenmeyer flask bagged out in polyethylene bag-out bag material. This solution was containerized for 14 months. On the right is the SAVY-4000 that contained "hatch" material

After cleaning, the SAI solution container was removed from PF-4 and the wall thickness measured through a coordinate measuring machine and an ultrasonic wall thickness measurement device utilizing eddy current array flaw detection. These measurement devices did not detect any significant wall loss from the corrosion. This container was subsequently helium leak tested and underwent design qualification drop testing 4 times. The container was loaded with 32.73 pounds of tungsten shot (total weight of 40.13 pounds) and was dropped in a center of gravity over bottom corner orientation from 12 feet 2 inches. After each drop the container underwent a helium leak test. The second drop orientation was center of gravity over top corner. The third drop utilized a side drop orientation, but a minor "slap down" occurred, meaning that the horizontal inclination of the container at impact was not 0° in reference to the impact plate. The fourth test was conducted in a similar side drop orientation, but on the other side of the container, 180° from the impact from the third drop test. This test also had a slap down impact, but slightly more aggressive than the third drop. The leak test after each of these drops passed; and in fact, the helium leak rate did not depend on the number of drops. This result also shows that the containment boundary was not compromised after 4 consecutive drops on a previously corroded container.

With the accelerated aging and surveillance observation and testing, a technical basis document was drafted to extend the life of the SAVY-4000 from 5 years to 15 years [10]. This technical basis relied heavily on the data from accelerated aging studies for the O-ring and filter, and on surveillance test results for corrosion behavior. The SAVY-4000 body was identified as the life limiting component. The main area of concern was corrosion, but specifically a through wall crack from SCC. The fact that Hagans are more susceptible to SCC and no SCC has been observed, even in 17-year-old Hagan containers, was the basis for proposing a 15-year lifetime for SAVY-4000. This technical basis was submitted to the Los Alamos Field Office and sent to experts on containers from other sites other than LANL for comment. After comments were received, a comment resolution was generated [11], and a final submission to the field office was made. On June 11th, 2019, the DOE accepted the lifetime extension of the SAVY-4000 series of containers from 5 years to 15 years [12].

Second Lifetime Extension

Based on the information gleaned from the initial lifetime extension; a second lifetime extension effort focusing on SAVY-4000 containers older than 15 years has begun. The main priorities of this effort are to further characterize the susceptibility to corrosion, focusing heavily on pit growth rates and SCC initiation. Additional efforts in bag-out bag degradation measurement, analysis of a loaded SAVY-4000's headspace gas, and material characterization methods are underway. The goal of the second lifetime extension is to be able to claim the longest life possible from the data gathered. Therefore, high confidence in the data gathered is paramount.

In addition to corrosion characterization, containment boundary failure testing is planned. The primary containment boundary failure concern is a corroded container that is dropped. Understanding the extent of wall loss or pitting required to penetrate the containment boundary in this scenario is key. If this parameter can be determined, a bounding scenario can be generated and tested against. This also provides a condition in which surveillance containers can be monitored and evaluated, allowing for either confirmation or rejection of the proposed lifetime. Drop testing efforts and corrosion efforts can be completed in parallel.

Current and Proposed Studies

Both gaseous and aqueous corrosion studies are being conducted to establish the corrosion rate of SAVY-4000 material as a function of chloride loading, source material, temperature and relative humidity. A test matrix involving NaCl, KCl, $MgCl_2$, $CaCl_2$, HCl and NaOH on pristine SAVY-4000 container wall samples will be evaluated. Corrosion will be initiated and measured through aqueous corrosion cells. Molarities of solutions will be tested at 0.1M, 1M, and greater than 6M depending on the saturation level of the chloride containing material in water. Temperature conditions will be tested at 30°C, 40°C and 60-80°C. While the bounding temperature of a SAVY-4000 is taken as 70°C in actual storage, challenges arise from evaporation under testing with liquid solutions. These tests are designed to provide design life information under extreme bounding conditions, and to understand the outer limits of conditions and their relationship to design life. One plausible example is a deliquesced plutonium salt corroding from the inside out, challenging the inner container, the bag-out bag, and the SAVY container wall.

In these experiments, coupon composition will be measured with X-ray Florescence (XRF). For SAVY-4000 coupons, these results can be compared to the production history, which gives the measured material composition associated with the heat number of the sourced material. Coupon thickness will be measured before and after corrosion as well. Wall loss measurement, corrosion currents/rates and pit growth rates will be analyzed. Pit morphology and depth will be measured through optical microscopy methods. The impedance currents measured will be made using titanium standards in a 1mM H_2SO_4 solution over 24 hours at 30°C, 60°C and 80°C to verify functionality of all testing equipment.

Aqueous corrosion testing of SAVY-4000 samples in temperature jacketed flat cells will be performed. The design of experiment is represented in Table 1. To achieve 10M chloride concentrations, ammonium chloride will be used as it is highly soluble in water. In addition to the accelerated corrosion studies, qualitative immersion tests will be conducted to understand the variation possible that is seen under storage conditions. The conditions for the qualitative immersion studies can be found in Table 2.

Table 1. Design of experiment for Sigma's corrosion studies.

DOE - Assessing Corrosion Rate			
Location (along wall)	Top	Middle	Bottom
Time (days)	0	7	14
Cl- Concentration (mol/kg)	0.1	1	10
Temperature (°C)	20	45	70
pH	neutral	acidic	

Table 2. Immersion corrosion study test matrix to qualitatively evaluate variation in temperature and pH dependance of corrosion.

Immersion Testing Experimental Conditions		
T	[Cl⁻]	pH
RT	1 M	2
		7
	10 M	2
		7
70 °C	1 M	2
		7
	10 M	2
		7

Studies have shown that concentration of chloride ions can reach as high as 10 molal (moles per kg solute) under 1gram/m^2 sea salt loading at the deliquescence point [13]. Evidence also suggests that the increasing chloride concentration reduces pitting initiation time, but ammonium ions do not significantly affect pitting times. This study aims to produce and quantify general and pitting corrosion growth rates.

We will also be conducting chloride droplet testing of pristine SAVY-4000 container samples. These droplets will vary between NaCl, KCl, $MgCl_2$ and $CaCl_2$ salts as well as HCl between 1.5μg $[Cl]^-$ cm^{-2} to 1500μg $[Cl]^-$ cm^{-2}. These droplets will be applied using a precision dropper and the area of application will be measured to confirm chloride loading. These samples will be allowed to dry and then placed in a relative humidity chamber at 70°C and 50% relative humidity. These conditions should represent more aggressive conditions found in storage and accelerate the aging process. Samples will be periodically examined under a microscope. General corrosion rate will be measured through mass loss after a nitric acid cleaning procedure. Pitting characteristics will be measured with optical microscopy, laser confocal microscopy and a Wide-Angle Measurement system.

Additional residual stress measurements will be conducted on the full height of the container. The welded region at the collar has been suspected to be the location of the highest stress and was confirmed through the boiling $MgCl_2$ experiments. The bottom radius is also suspected to have higher stress, even after the annealing after each of the drawing steps to create the SAVY-4000 body. Work to determine SCC initiation based on these measured stresses is planned.

In situ measurement of SAVY-4000 headspace gas is also planned. This experiment will utilize the large-scale array established from the MIS Working Group. The headspace of the loaded SAVY-4000 will be connected to a gas chromatograph coupled with mass spectrometry (GC-MS). Headspace samples will also be taken in order to measure them with Fourier transform infrared spectroscopy (FTIR). Due to the reactivity of HCl and Cl_2 gas with metals, the GC-MS tubing will most likely absorb all chloride containing gases, essentially hiding them from analysis. However, FTIR gas samples would not have these problems if the samples were collected in an inert sample collector. Development of this sampling technique is ongoing. It is planned to evaluate 3 different materials with low, medium and high specific wattages. These materials will be packaged into a Conflat container, reducing the variable of material off-gassing. The Conflat containers will be bagged out in the traditional PVC bag-out bag, and the off-gassing and degradation of the bag-out bag will be measured.

Material pitting is not necessarily hemi-spherical, and enhanced material characterization is necessary to ensure that the optical microscopy methods utilized to measure pit depth are accurate. Scanning electron microscopy coupled with focused ion beam etching (SEM/FIB) will also be evaluated. This technique allows for ablation of the base material to “mill” sections of the sample during examination under SEM. This allows for gradual milling of the pit to accurately visualize and measure the propagation characteristics of the pit growth.

Relative humidity has a strong relationship with corrosion rate. Efforts to measure the relative humidity in various locations of PF-4 are underway. One location has been measured for approximately 5 months. Comparison between the room temperature relative humidity with nearby weather stations are being conducted. Additional sensors in other locations have been purchased, calibrated, and will be installed. If a correlation can be made from interior conditions of PF-4 to external weather stations, then historical weather data may provide valuable information on the importance of relative humidity to the corrosion of storage containers.

Annual surveillance will continue to occur providing data for both SAVY-4000 aging and Hagan aging. In FY22, a SAVY-4000 on a floor storage location was identified to have corrosion surrounding the filter as seen in Figure 4. This item was unpacked, and the container passed all surveillance testing. This container has been removed from PF-4 for further study. The material within this container was a low wattage MSE residue. When unpacking, the bag-out bag material did not appear degraded. This suggests that the material itself was off gassing chloride gasses. The corrosion on the container was green, suggesting the formation of iron (II) chloride. This container has since turned rust colored, suggesting further oxidation after unpacking oxidized the container to iron (III) oxide. Additional studies of material off-gassing and the effect of relative humidity on gas generation and corrosion will also be conducted.

Figure 4. A SAVY-4000 discovered in a floor storage location during PF-4 walkdowns in late FY22. The external filter corrosion was identified and the material unpacked. From left to right – the external filter corrosion, the unpacked inner lid corrosion, the mostly pristine bag-out bag, and the inner body corrosion.

<u>**Summary and Conclusion**</u>

The SAVY-4000 nuclear material storage container lifetime has been studied over the course of the last 9 years. After significant research efforts, the lifetime of the series of containers was extended from 5 years to 15 years. Additional efforts building on the initial lifetime extension are underway to evaluate the basis for extending this lifetime. Material pitting and cracking characteristics and growth rates, bag-out bag degradation and off-gassing, and enhanced optical analysis methods are all being evaluated.

After completion of these studies, a lifetime will be assessed, and a justification will be assembled into a technical basis document for evaluation by other container subject matter experts across the DOE complex. Their comments will be resolved, and the final technical basis will be submitted to the LANL field office for DOE review and approval. It is the principle aim of this effort to identify an accurate service life for the SAVY-4000 container is established for more conservative conditions to ensure the continued safety of the worker, public, and environment.

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