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EXTENDING PERIODIC LEAKAGE RATE TESTING INTERVAL FOR TYPE B RADIOACTIVE MATERIAL PACKAGING

Z.H. Han
Argonne National Laboratory, 9700 South Cass Avenue, Lemont, IL 60439

Y.Y. Liu
Argonne National Laboratory, 9700 South Cass Avenue, Lemont, IL 60439

J.M. Shuler
U.S. Department of Energy, 1000 Independence Avenue SW, Washington, D.C. 20585

ABSTRACT

According to American National Standards Institute N14.5, American National Standard for Radioactive Materials—Leakage Tests on Packages for Shipment, 2014, the periodic leakage rate testing of a Type B radioactive materials (RAM) transportation packaging shall be performed within 12 months prior to each shipment, unless an alternative frequency is justified and approved by the regulatory authority. The purpose of periodic leakage rate testing is to confirm that a packaging built to an approved design standard can still perform the containment function after a period of service. Periodic leakage rate testing must be performed on the packaging containment boundary components that are subject to wear or degradation, such as O-ring seals, closures, valves, and rupture disks. For Type B transportation packaging, a common practice is to replace the O-ring seals of the containment boundary every 12 months and then perform leakage rate testing to the design standard. Meanwhile, Type B transportation packages, after loading of the radioactive contents, may be placed in interim storage for a period of more than 12 months before there is a plan for shipment. Therefore, it is highly desirable to extend the periodic leakage rate testing interval beyond 12 months, for example, up to five years, which is the regulatory limit for timely renewal of the Certificate of Compliance for Type B transportation packages. The extension of the periodic leakage rate testing interval greatly reduces the exposure of personnel to radiation and associated operating costs.

Two methodologies have been developed and used to support the extension of the periodic leakage rate testing intervals for DOE-certified Type B transportation packages. Selected examples of demonstration and application of the methodologies are presented using Model 9977 and Model ES-3100 packages in interim storage. Using the long-term test data obtained by using the 9975 mockups with Viton® GLT/GLT-S O-rings, the technical basis has been established for using the ARG-US RFID remote monitoring system to extend the periodic leakage-rate testing interval to five years for the Model 9977 package. A combination of testing and analysis by comparison to similar ethylene propylene diene monomer (EPDM) seal applications was used as the basis to extend the periodic leakage-rate testing interval to two years for the Model ES-3100 package. Aging degradation of elastomeric O-rings for RAM packaging and spent fuel dry storage casks under service conditions remains a concern and the subject of continuing studies.

INTRODUCTION

Title 10 Code of Federal Regulations Part 71 (10 CFR 71) requires in §71.43 that Type B packages of radioactive materials (RAM) be designed, constructed, and prepared for shipment so that there shall be no loss or dispersal of radioactive contents under normal conditions of transport (NCT). The U.S. Nuclear Regulatory Commission (NRC), in NUREG-2216 [1], accepts the use of American National Standards Institute (ANSI) N14.5-2014 [2] for demonstrating that packages of RAM comply with the leak test requirements of 10 CFR 71. According to Section 7.5 of ANSI N14.5-2014, periodic leakage rate testing of Type B packages is to be performed within 12 months prior to each shipment, unless an alternate frequency is justified and approved by the applicable regulatory authority, to confirm that the containment capability of the package has not deteriorated during that period. Certified transportation packages Models 9975, 9977 and ES-3100, for Type B radioactive and fissile materials, have been used for interim storage for a period greater than 12 months, and it is highly desirable to extend the periodic leakage rate testing interval up to five years, which is the regulatory limit for timely renewal of the Certificate of Compliance (CoC) for Type B transportation packages, to reduce personnel radiation exposure and operational cost. A CoC amendment for Model 9977 Type B(M)F packages was issued in 2010 that extended the periodic leakage rate testing interval to 2 years with continuous temperature monitoring using the ARG-US RFID system [3, 4]. Similarly, an amendment of the CoC for Model ES-3100 Type B(U)F package was issued in 2019, which also extended the periodic leakage rate testing interval for the package to 2 years. The following sections address the issues regarding the extension of the periodic leakage rate testing interval: (1) Component and system reliabilities of containment using O-rings; (2) Long-term sealing capabilities of Viton® GLT and GLT-S O-rings; (3) O-ring service temperature and temperature monitoring of the 9977 package; (4) Extending the periodic leakage rate testing interval for ES-3100 packaging that uses EPDM O-rings for its containment boundary; (5) Discussion; and (6) Summary and Conclusions.

COMPONENT AND SYSTEM RELIABILITIES OF CONTAINMENT USING O-RINGS

The basis of the 12-month periodic leakage rate testing interval before each shipment can be traced to two papers by Lake [5, 6] on containment closure designs using elastomeric O-ring seals. Lake addressed both component and system reliabilities. The component reliability may be defined as the probability of that component performing, as required, under specified environmental conditions over a specified period. The system reliability may be defined similarly, but the definition is expanded to encompass an interacting system of components. An example of the latter is a closure system with redundant seals and with a test port/plug. The reliability of the closure seal system has been found to be design dependent.

The failure rate of a component λ(t) is defined as the conditional instantaneous probability of failure at time t, given that failure has not yet occurred. A mortality curve that plots the component failure rate against time (or age) has the familiar “bathtub” shape, where λ(t) decreases initially as faulty components are eliminated by premature failure. The useful life is characterized by a constant λ(t), and the wear-out period is characterized by an increasing λ(t). The practice that is suggested by the mortality curve is to use components that fall within the useful-life period. This processing involves screening components to eliminate potential early failures and replacing components in service before the wear-out period. The component reliability R(t) is commonly described by the Weibull distribution:

$$R(t) = exp\left[-(t/\alpha)^{\beta}\right], \qquad (1)$$

where α and β are the scale and shape parameters, respectively, of the Weibull distribution. The failure rate, or the hazard function h(t), associated with the Weibull distribution is given by

$$h(t) = \beta\alpha^{-\beta}t^{\beta-1}, \qquad (2)$$

where h(t) is a measure of the “proneness to failure” of a component after time t has elapsed.

A special case of the Weibull distribution is the exponential distribution that occurs when β =1 (i.e., for the flat portion of the "bathtub," where the components fail by chance alone); then, equations (1) and (2) become

$$R(t) = exp(-t/\alpha) \quad (3)$$

$$h(t) = \alpha^{-1} , \quad (4)$$

where h(t) is a constant failure rate λ (=α^{-1}) that reflects no aging effect on the component in service, and

$$R(t) = exp(-\lambda t) \quad (5)$$

Using data found in the literature for static seals having a life expectancy of 3.5×10^{4} h (4 years) and a failure rate of $\lambda = 3\times10^{-6}\ h^{-1}$, Lake estimated component reliability values of 0.9999, 0.9995 and 0.9950 for periods of 1 day, 1 week and 10 weeks, respectively. Using the same failure rate of $\lambda = 3\times10^{-6}\ h^{-1}$, the component reliability would be 0.9741 and 0.8769 for a period of 1 and 5 years, respectively.

Mathematical models were then developed by Lake for estimating the reliability of three different closure designs: (i) single-seal design, (ii) redundant-seal design without a test port, and (iii) redundant-seal design with a test port. The elastomeric O-rings were assumed to have the same component and assembly reliabilities, and the seal plug for the test port was assumed to have its own reliability (a product of its component and assembly reliabilities). A comparative reliability analysis of the three closure designs showed that the redundant-seal design with a test port has the highest closure reliability, followed by the redundant-seal design without a test port and then the single-seal design. Reliability testing also showed that a closure verification test always improves the reliability of a system. For a single-seal design, a verification test results in an assembly reliability of unity, and the closure reliability is equal to the component reliability.

Several transportation package designs have been approved by NRC on the condition that closure seals are replaced annually. Based on life expectancy data of 4 years for seals and the possible uncertainty in the data, it was concluded that the annual replacement of elastomeric seals for the more common applications in closure designs for RAM transportation packages is a reasonable approach. It was also concluded that the estimates of reliability are strongly dependent on life test data, and confidence increases significantly if data for specific components in realistic transportation environments are used. Lake's analyses from 45 years ago [5, 6] of component and system reliabilities of containment using O-rings remain as the technical basis for Type B transportation packaging containment design to date.

LONG-TERM SEALING CAPABILITIES OF VITON® GLT AND GLT-S O-RINGS

Compressive Stress Relaxation

The service life of O-rings depends primarily on the degradation of the compressive sealing force between the seal and the mating surfaces; compressive stress relaxation (CSR) is a commonly used parameter for O-ring service life prediction. Long-term CSR data for GLT O-rings have been obtained at temperatures ranging from 175 to 350°F (79.4 to 176.7°C) [7, 8]. The CSR data are plotted as a function of inverse temperature (1000/T) in Figure 1, assuming (somewhat arbitrarily) that service life is represented by 90% or 100% loss of this sealing force. The 90% loss of sealing force has been used in other studies, but it is not an absolute failure parameter. For the 9975 and 9977 primary containment vessel (PCV) designs, the relationship between sealing force loss and leakage rate has not been established. The activation energy calculated for the GLT O-rings is 56 kJ/mol, using the CSR data. Using 90% loss of sealing force, the predicted service life of the GLT O-rings at 200°F (93.3°C) is 3,655 days (10 years), which increases to 16,257 days (44.5 years) at

150°F (65.6°C). Similar predicted service lives for the GLT-S O-rings based on the CSR data are expected, owing to the material similarity between GLT-S and GLT. Baseline characterization of GLT-S O-rings indicates that the GLT-S compound is comparable to GLT, with some minor variations. The baseline tensile properties are slightly different, although they all meet the Aerospace Material Specifications. The short-term CSR behavior of the GLT-S O-ring at elevated temperature is improved over that of the GLT O-ring. Composition analysis indicates that the polymer structures of GLT and GLT-S are very similar, with some variations in additives. The glass transition temperatures (T_g) for GLT-S, as determined by dynamic mechanical analysis, are lower than those for GLT, indicating better low-temperature performance for GLT-S.

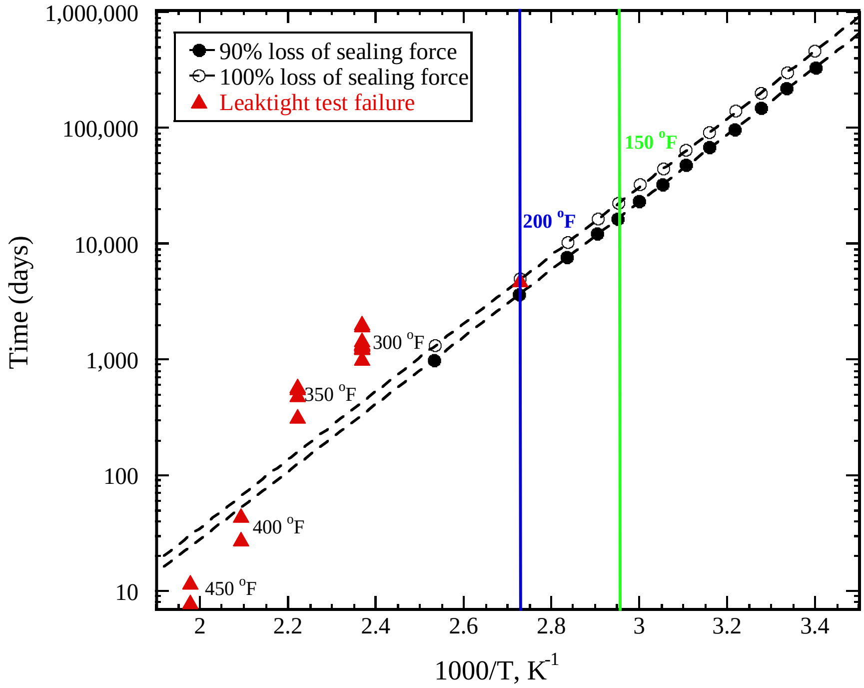

Figure 1. CSR data for GLT O-rings based on 90% and 100% loss of sealing force

<u>Service Life Evaluation Using Weibull Statistics</u>

Leak-testing experiments to monitor the aging performance of GLT and GLT-S fluoroelastomeric O-rings (Parker Seals V0835-75 and VM835-75) used in the Model 9975 and 9977 shipping packages have been ongoing since 2004 at Savannah River National Laboratory. Seventy tests using mock-up 9975 PCVs with GLT O-rings were assembled and heated to temperatures ranging from 200 to 450°F (93.3 to 232.2°C). Owing to material substitution and the resulting availability issue of GLT O-rings, 14 tests with GLT-S O-rings were initiated in 2008, with heating to temperatures ranging from 200 to 400°F (93.3 to 204.4°C). Data from the O-ring fixtures were generally consistent with results from CSR testing and provided confidence in the predictive models based on those results. However, uncertainty exists in extrapolating these elevated-temperature results to the lower temperatures of interest for normal storage in the K-Area Material Storage (KAMS) facility at the Savannah River Site. Oxygen consumption tests were conducted to provide corroborative data for the extrapolations. The collective data from these testing efforts suggested that the minimum O-ring service life under the KAMS normal storage conditions should be at least 34 years for GLT and GLT-S O-rings at a temperature lower than 200°F (93.3°C). As of August 2022, all GLT O-ring test fixtures aged at 300, 350 and 400°F (148.9, 176.7 and 204.4°C) have failed; only one of the 21 GLT O-ring test fixtures aged at 200°F (93.3°C) failed the leakage rate test, with an elapsed test time up to 13.4 years, and two test fixtures at 270°F (132.2°C) have shown no failure for up to 10.4 and 10.7 years, respectively. All GLT-S O-ring test fixtures aged at 300°F (148.9°C) and above have failed, and one of the two GLT-S O-ring test fixtures aged at 200°F (93.3°C) failed the leakage rate test, with an elapsed test time of 12.4 years. Two GLT-S O-ring test fixtures aged at 250°F (121.1°C) have shown no failure for up to 12.5 years [9].

One of the 42 GLT O-rings in the 21 GLT test fixtures and one GLT-S O-ring in the 4 GLT-S test fixtures failed the leakage rate test at 200°F (93.3°C). Using Weibull statistics on the above sample size for GLT and GLT-S O-rings, the service lives t can be calculated for a given reliability R(t), using Equation (1), which gave a service life of 12.5 years based on 42 GLT O-ring samples, and 10.9 years based on 42 GLT plus 4 GLT-S O-ring samples, for R(t) = 99.9% [10].

O-RING SERVICE TEMPERATURE AND TEMPERATURE MONITORING OF THE 9977 PACKAGE

The service temperature of GLT or GLT-S O-rings in the 9977 packages in a storage area without insolation was evaluated using ABAQUS, with the thermal model shown in Figure 2. For a given contents heat load in a 9977 package, the service temperature of the O-rings can be calculated for a given ambient temperature. Conversely, the maximum allowable ambient temperature can be calculated for an O-ring temperature limit of 200°F (93.3°C), which is conservative for GLT or GLT-S O-rings on 9977 when stored in KAMS [9]. Table 1 shows that for a contents heat load of 19 W, the maximum ambient temperature would need to reach 100°F (37.8°C), for a calculated O-ring temperature of 198°F (92.2°C). The CoCs previously issued for the 9977 package with continuous ambient temperature monitoring by the ARG-US RFID system have a heat load limit of 15 W. This limit allows an ambient-temperature upper limit of 125°F (51.7°C) to be used as an alarm threshold for the RFID temperature monitoring system. Excessive temperature, if it occurred, would be recorded as a violation with a non-conformance report that would require disposition. For the 9977 packages with ARG-US RFID tags installed and whose periodic leakage test intervals were extended, the upper limits on ambient temperatures for alarm thresholds are listed in Table 1 for contents heat loads ≤ 15 W. If the ambient temperature in the storage area is 75°F (23.9°C), with 15-W contents heat load, the O-ring temperature will be approximately 150°F (65.6°C).

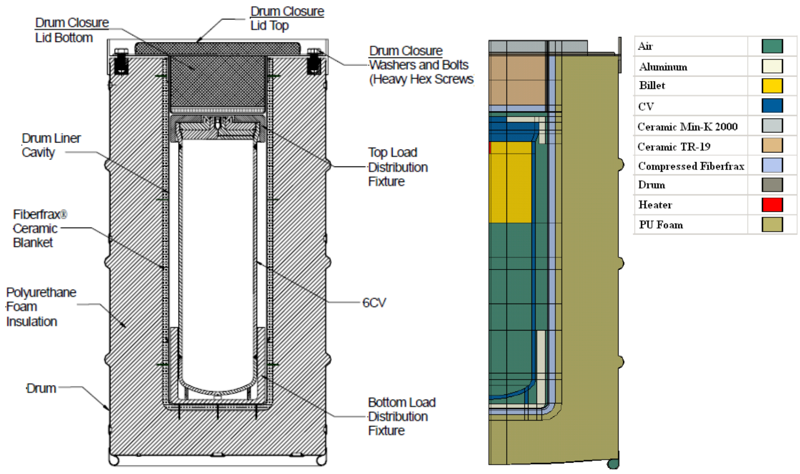

Figure 2. Thermal model for 9977 package O-ring temperature evaluation [10]

Table 1 O-ring temperatures with varying contents heat loads and ambient temperatures*

Heat Load (W)	Maximum Ambient Temperature [°F(C)]	Calculated O-Ring Temperature [°F(C)]	Upper Limit on Ambient Temperature [°F(C)]
19	100 (37.8)	198 (92.2)	100 (37.8)
17	110 (43.3)	197 (91.7)	110 (43.3)
15	125 (51.7)	200 (93.3)	125 (51.7)
13	135 (57.2)	200 (93.3)	125 (51.7)
10	150 (65.6)	199 (92.8)	150 (65.6)
7	160 (71.1)	195 (90.6)	150 (65.6)
5	175 (79.4)	200 (93.3)	150 (65.6)
2	185 (85.0)	195 (90.6)	150 (65.6)
0	200 (93.3)	200 (93.3)	150 (65.6)

* For conservatism, contents were located near the top of the CV and close to the O-ring (see Fig. 2).

The highest RFID-recorded ambient temperature in the KAMS facility during the summer months of June to September in 2010 was ~35°C (95°F) [11], which was well below the upper limit of ambient temperature of 51.7°C (125°F) for 15 W, and the margin increases as the contents heat load decreases below 15 W with time. The periodic leakage rate testing interval was further extended to 5 years in 2012 based on the test results.

EXTENDING PERIODIC LEAKAGE RATE TESTING INTERVAL FOR ES-3100 PACKAGE

The Model ES-3100 Type B packaging, shown in Figure 3, uses EPDM O-rings in the containment vessel (CV) closure assembly. A CoC amendment was requested in 2016 to extend the leakage rate testing interval from 1 year to 2 years. The methodology used to support the change request was a combination of testing and analysis by comparison to similar seal applications and their performance environment. The test data from ES-3100 EPDM O-rings are contained in a document, ES-3100 Containment Vessel (CV) O-rings Life Extension Testing [12], and its references, which provided the technical justification.

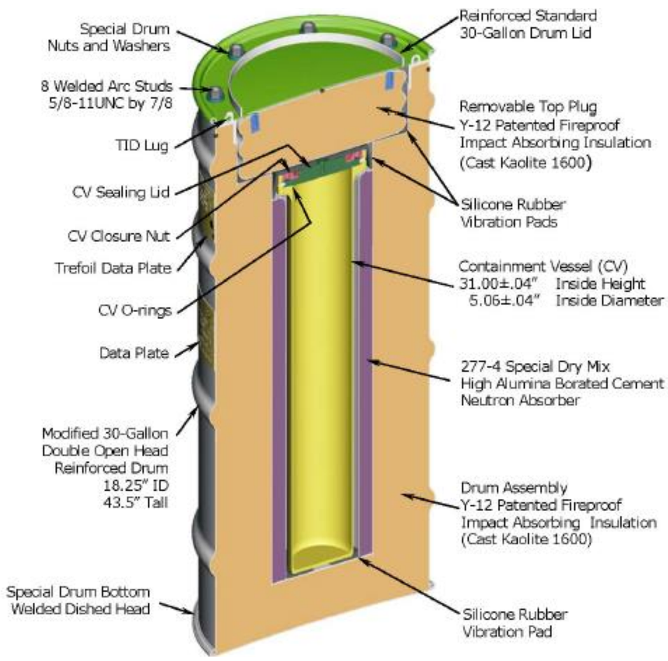

Figure 3. Model ES-3100 Type B packaging [12]

Reference [12] contains data from air (the fabrication verification of the O-ring seal at initial fabrication on the fully assembled CV with both inner and outer O-rings installed to demonstrate the functionality of the leak-test port and sealing capability of the outer O-ring) and helium (the periodic leakage rate testing of the ES-3100 containment boundary) performed on 20 Model ES-3100 packaging removed from storage; 16 of them are new and unused, and 4 are used. The 16 new packaging's were in storage for at least seven years and the 4 used packaging were in storage for four years prior to removal for testing. All leakage rate tests were conducted under ambient conditions. Test results showed that all 4 used packaging's passed leakage rate tests for both air (leakage rate $<1.0 \times 10^{-4}$ atm-cc/s) and helium (leak rate $< 1.9 \times 10^{-7}$ cc/s). Three of the 16 new packaging initially failed both air and helium leak rate tests. After the helium leakage test failure, the test flange was removed and the inner CV O-ring and O-ring grooves were carefully examined. No debris, surface damage, or other physical damage could be seen on the O-ring; no debris, moisture, or damage (scratches, nicks) was found in the CV grooves; and no corrosion was found. The O-rings and O-ring grooves were wiped clean with alcohol and a lint-free cloth. A thin coat of Super-O-Lube was rubbed on the inner O-ring, and it was re-installed in the CV groove. The helium leak test was performed again, and the inner O-ring passed the helium test. The CV was then re-assembled and subjected to air leak testing, which it also passed. Although the test report did not identify the root cause of the initial failure, retesting the CV is allowed and consistent with the operating procedures in the Safety Analysis Report for Packaging (SARP). However, the ES-3100 package is designed to ship RAM content with a decay heat limit of 5 W. The normal service temperature range for the EPDM O-ring used in the containment boundary is -40 to 302°F (-40 to 150°C). According to thermal evaluation in the SARP, the maximum predicted temperatures of the containment boundary O-ring under NCT are 109.3°F (42.9°C) and 197.36°F (91.9°C), respectively, in shade and with solar insolation in still air at 100°F (37.8°C) ambient. The service temperatures of the O-rings in ES-3100 packages with RAM content are expected to be higher than that for empty ES-3100 packages in storage.

Two aging studies of EPDM O-rings, referenced in the test report [12], were also used to perform an analysis-by-comparison to predict the performance of the EPDM O-ring in ES-3100 for up to 2 years. The first aging study examined the Model H1616, a drum-type package design with a containment boundary seal of EPDM O-rings [13]. The O-rings were aged in the H1616 CVs at temperatures ranging from 160°F (71.1°C) to 300°F (148.9°C). The H1616 CVs were helium leak tested and re-tested periodically, depending on the aging temperature, to determine whether they continued to meet the ANSI N14.5 leaktight criterion of a leakage rate less than or equal to 1×10^{-7} ref·cm^3/s. The data showed that at the O-ring aging temperature of 160°F (91.9°C) for up to 3 years, both inner and outer EPDM O-rings of the H1616 CV are leak-tight in multiple helium leakage rate tests. The data also showed that for an O-ring aging temperature of 235°F (112.8°C) for up to two years, both inner and outer EPDM O-rings of the H1616 CV were leak-tight in multiple helium leakage rate tests. The H1616 CV test results bounded the ES-3100 CV NCT temperatures calculated for the containment boundary O-ring at 109.4°F (43°C) in shade and 197.4°F (91.9°C) with solar insolation.

The second aging study evaluated the influence of aging on the sealability of EPDM O-rings aged at four different test temperatures: 167, 212, 257, and 302°F (75, 100, 125, and 150°C) for up to 1.5 years [14]. The data showed considerable degradation effects as demonstrated by hardness, CSR and compression set (CS, which represents the recovery behavior of a seal after release from compression). The leakage rate stayed relatively constant, or even decreased, until shrinkage combined with the loss of resilience of the aged seal led to leakage. Extrapolating the testing data to 140°F (60°C) indicates that the CS would reach 50% in approximately 17 years. This result (17 years) is much longer than the requested 2-year extension of the periodic leakage rate testing interval for the EPDM O-rings of ES-3100.

Based on the technical review of the amendment request and confirmatory evaluation, it was concluded that reasonable assurance has been provided that the proposed alternate frequency is adequate, and the amendment application of CoC was approved for Model ES-3100 Type B packaging in 2019, extending the periodic leakage rate testing interval for the package to 2 years.

It should be noted that the effect of radiation on aging and long-term sealing capabilities of Viton® and EPDM O-rings is not considered in this paper. Experience and previous investigations indicate that Viton® O-rings of all types, including GLT and GLT-S, have sufficient resistance to the radiation doses expected in most RAM packaging [15]. For the EPDM O-rings in the ES-3100s, according to the Parker O-Ring Handbook, practically all elastomers exhibit no change in their physical properties at a cumulative dose up to 1×10^6 rad. Based on the calculated dose rate of 0.5 rad/hour in the ES-3100 Safety Analysis Report for Packaging, the maximum cumulative dose for the containment boundary O-ring location would be $<1 \times 10^4$ rad for an exposure period of 2 years.

DISCUSSION

It is important to understand the basis for the ability of an elastomeric O-ring to perform its sealing function. When two “hard” materials such as stainless steel are in contact, the roughness of the mating surfaces creates microscopic open gaps in the contact region. Any long-range connectivity of these open gap regions would provide potential leak paths from one contact boundary to another [16]. If this long-range connectivity is disrupted and the gaps become isolated regions, then the two contacting mating surfaces provide a leak-tight barrier against leakage of liquid or gas. This disruption of the long-range connectivity among the gaps between the mating surfaces can be accomplished by the placement of an elastomeric O-ring between the two mating surfaces. The viscoelastic property of the O-ring materials allows the O-ring to deform under the external applied stress to fill up many of the open gap regions, thereby changing them into closed gap regions and leading to leak-tightness. Relaxation of the stress state of the elastomeric materials over time reduces the capability of the O-ring to deform and fill up the open gap regions. Previously closed gaps may become open, so that leak tightness can no longer be maintained. This change signals the beginning of O-ring failures. This decline in the O-ring sealing capability over time can occur even if external environmental factors such as applied stress and temperature remain nominally the same. The "intrinsic' lifetime of an O-ring under a nominally unchanging external environment is a critical factor in the maintenance of containment integrity for the transportation and storage of RAM. A conservative bound on the effect of O-ring size (for O-rings of the same material and manufacturer's category) on reliability has been incorporated into estimates of the total reliability of the systems of O-ring applications [17]. Percolation, triggered by external events such as vibration or sudden impact, can establish long-range connectivity among the gaps between the mating surfaces, causing temporary loss of the sealing capability of elastomeric O-rings.

It should also be noted that elastomers are widely used as sealing materials in packages and containers for low- and intermediate-level radioactive wastes, and as an additional component of metallic seals in bolted casks for spent fuel and high-level waste. Such "auxiliary" seals enable proper leakage rate testing of the metal barrier seals. Maintaining the confinement/containment boundary for bolted dry cask storage systems during long-term storage and transportation requires understanding of the aging mechanisms that affect the long-term performance of elastomeric O-ring seals with respect to their function as auxiliary seals. The work conducted by researchers at the Federal Institute for Materials Research and Testing on elastomeric seals in recent years can be found in Refs. [18-22]. Accelerated aging tests of elastomeric seals under operation-relevant conditions, coupled with physics-based modeling simulation, are combined in the development of prediction methods for long-term performance of elastomeric O-ring seals used for storage and transportation casks.

SUMMARY AND CONCLUSIONS

Type B transportation packages may be placed in interim storage for a period greater than 12 months before shipment; therefore, it is highly desirable to extend the periodic leakage rate testing interval to reduce the exposure of personnel to radiation and associated operating costs. The NRC has endorsed ANSI N14.5, which allows for an alternative frequency for the periodic leakage rate testing of Type B RAM transportation packaging if it is justified and approved by the regulatory authority. Two different methodologies have been developed and used to support requests for extending the periodic leakage rate testing intervals for DOE-certified 9977 and ES-3100 Type B transportation packages that are loaded but not immediately shipped. The methodology for 9977 was based upon acceptable results of long-term O-ring seal performance testing data on GLT and GLT-S O-rings and continuous monitoring of environmental conditions of the packages using the ARG-US RFID system. A combination of testing and analysis by comparison to similar EPDM seal applications was used as the basis for extending the periodic leakage-rate testing interval for the Model ES-3100 package. Lifetime evaluation using CSR data and the Weibull distribution showed that for the 9977 packages with GLT and GLT-S O-rings, the periodic leakage rate testing interval can be extended to 60 months (5 years), and to 2 years for the ES-3100 package with EPDM O-rings, based on long-term package performance data and comparison to similar seal applications. Aging degradation of elastomeric O-rings for RAM packaging and spent fuel dry storage casks under service conditions remains a concern, with continuing studies.

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