Application Note

Seal integrity of Thermo Scientific Nalgene InVitro Biotainer closures

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Key Words
Seal integrity, closure, back-off torque, leak testing, frozen storage

Abstract

Seal integrity of a container is critical to prevent loss of contents or contamination from outside sources. Since plastic is a malleable material, forces borne at the thread and mating surfaces of the closure of a plastic container can deform the material over time or under certain environmental conditions. This can lead to the degradation of torque required to open the cap, essentially loosening the closure. This effect can be exacerbated by the expansion and contraction during freezing and thawing of containers and their contents. Here, we examine the effects of time, temperature, and closure application torque on the integrity of the closure seal, and the propensity of the seal to loosen under certain conditions.

Introduction

In any container used for storage of liquid contents, the seal integrity of the closure system is important to ensure that the contents of the containers remain inside and do not leak out. This seal integrity becomes crucial when the closure also aims at preventing contamination from the outside. Several factors affect the seal integrity of a closure, including the shape of the threads, the shape of the mating surface, the material composition of the container and the closure, and the amount of torque applied to close the container. For example, the best seal is produced when the container and closure materials are of a different composition; when one material is softer than the other, the softer part can conform its shape to the harder corresponding part.

Time-dependent effects can also impact the quality of the closure seal, especially when containers, closures, or both are made of plastic resins. Plastic is a malleable material, and over time the force exerted on the closure threads and mating surface can reshape the material to a small degree. The result is a gradual degradation in the amount of force required to remove the closure, or the back-off torque. The typical usage of the containers can also affect the integrity of the seal. Subjecting the container and its contents to freezing temperatures, as frozen storage is a standard use for them, can affect the seal though expansion and contraction of the materials at the threads and mating surfaces. This movement, in addition to the change in malleability of the resin at cold temperatures, exacerbates the back-off torque degradation.

Thermo Scientific™ Nalgene™ InVitro™ Biotainer™ bottles have a soft material liner within their closure mating surface to ensure a proper seal when the Biotainer bottle is closed. These containers are often used for intermediate storage of process products during pharmaceutical production. As such, it is critical to maintain sterility and purity of the substances contained within them. Since these materials are often kept in frozen storage between production steps, it is also critical for the containers to maintain the integrity of the seal in freezing temperatures and during freeze/thaw cycles. Here, we examine the effect of time, temperature, and initial application torque on the seal integrity of several InVitro Biotainer container types. The data presented are the result of several experiments designed to mimic typical usage and examine the seal integrity through measurement of back-off torque degradation and directly through pressure-gradient leak testing.

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Experimental details

These results represent a compilation of experiments testing the effects of time, temperature, and torque on the seal quality of Biotainer bottles. They are presented here to allow examination of the effects of individual parameters.

Storage time

Polycarbonate (PC) Biotainer bottles of 1L and 5L capacity were tested for the degradation of back-off torque during storage at room temperature. For 1L Biotainer bottles, n=12 bottles per sample time (n=5 for 1 month time point) and applied torque were tested, for a total of 106 bottles. For 5L Biotainer bottles, n=5 bottles per sample time and applied torque were tested, for a total of 50 bottles. The bottles were filled with water and closures were applied to bottles using a manual torque wrench and adapter with torque of 30 in-lbs. or 40-42 in-lbs. Bottles were sampled for back-off torque degradation and leaks at each of the following storage times: 5 min, 1 hour, 1 day, 1 week, and 1 month. Back-off torque was determined using a manual torque wrench to measure the maximum torque during closure removal. Leak testing consisted of inverting the bottle and raising the pressure inside the bottle 2 psi for 2 min. Any water detected in the closure threads constituted a failure.

Temperature and application torque

Two experiments were conducted to analyze the effects of freezing and application torque on the integrity of the seal. In the first, 1L and 5L PC Biotainer bottles were filled with water and closures were applied using a manual torque wrench at 30, 40, 50, and 60 in-lbs. of torque. They were then frozen at -70°C, thawed, and re-frozen for a total of 3 or 6 freeze/thaw cycles. n=5 bottles were tested for each combination of applied torque and number of cycles, for a total of 40 bottles tested. Back-off torque and leak testing was performed as above. In the second experiment, 125mL and 1L Biotainer bottles were filled with a sodium phosphate glutamate solution and closures were applied as in the first experiment. Bottles were frozen at -67°C, for a total of 3 freeze/thaw cycles. n=10 bottles were tested per applied torque and test, for a total of 80 bottles tested (n=40 back-off torque and n=40 leak test). Back-off torque and leak testing was performed as above.

Results and discussion

Storage time

While there was some variation in the amount of torque required to remove closures (back-off torque) over time in storage, all bottles tested passed the pressurized leak test at both minimum and maximum recommended torque applications during the storage test. In the back-off torque analysis, after closures were applied there was a slight initial increase in torque. In both 5L and 1L bottles and at both maximum and minimum recommended torque applications, the back-off torque required increased by a small margin between the initial (5-10 minute) test and the 1 hour test (Figure 1). After the first hour, however, there was a trend downward in back-off torque over the course of the remaining month of testing. This pattern is likely due to initial “setting” of the materials as the closure mating surfaces conform to each other, followed by a gradual loosening as the malleable plastic resin relaxes from the static force experienced at the threads. It is important to note, however, that even at the decreased back-off torque seen after 1 month of storage the seal integrity is maintained, as no leaks were seen in leak testing.

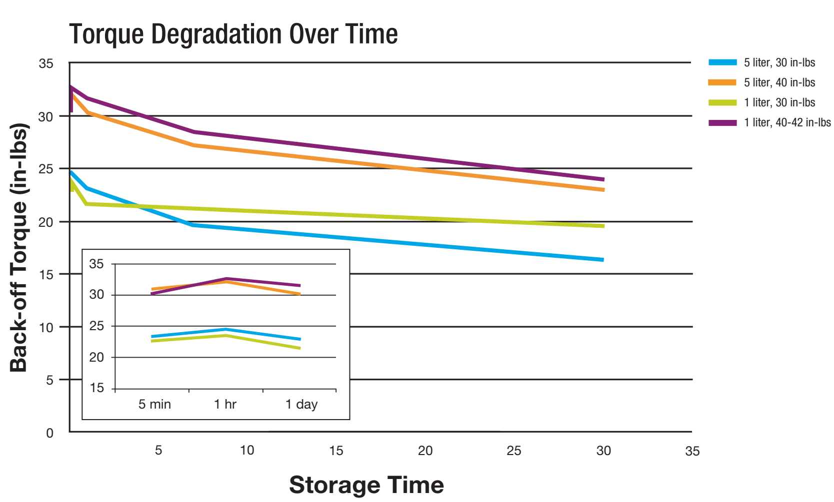

Figure 1. Back-off torque required for closure removal sampled over the course of one month at room temperature. Inset: magnified view of results over the first 24 hours in storage. Note the slight increase in torque required initially until the 1 day time point, then a gradual decrease for the remainder of the month.

Application torque

In order to examine whether a range of application torques had unexpected effects on the seal integrity of the bottles, leak testing and back-off examination was performed on closures with several application torques. Bottles that had undergone 6 complete freeze/thaw cycles were used for comparison to challenge the seal integrity. No leaks were seen during leak testing at any level of application torque, and back-off testing showed a consistent percentage of torque degradation for all application torques (Figure 2).

Temperature cycling

To determine the effect of expansion and contraction through multiple cooling/warming cycles, leak testing and back-off torque assessment was conducted on bottles that had undergone either 3 or 6 complete freeze/thaw cycles. In addition, one group of bottles filled with a sodium phosphate glutamate (SPG) solution was used to determine whether the freezing and other properties of the contents of containers could affect the seal integrity. Freeze thaw cycles had little effect on the back-off torque required to remove closures, as removal torque was similar for both 3 and 6 freeze/thaw cycles to removal torque for bottles left at room temperature (Figure 3). Bottle contents also did not affect back-off torque, as the results for bottle containing SPG were very similar to bottles containing water that underwent 3 freeze/thaw cycles.

During leak testing, no bottles filled with water showed any leaks, highlighting the seal integrity of Biotainer bottles even after several freeze/thaw cycles. Several bottles filled with SPG with closures applied to 30 in-lbs. did leak, however, with a 40% failure rate. This highlights the importance of performing testing of the specific intended application with these containers, as the contents of the container may affect the seal integrity.

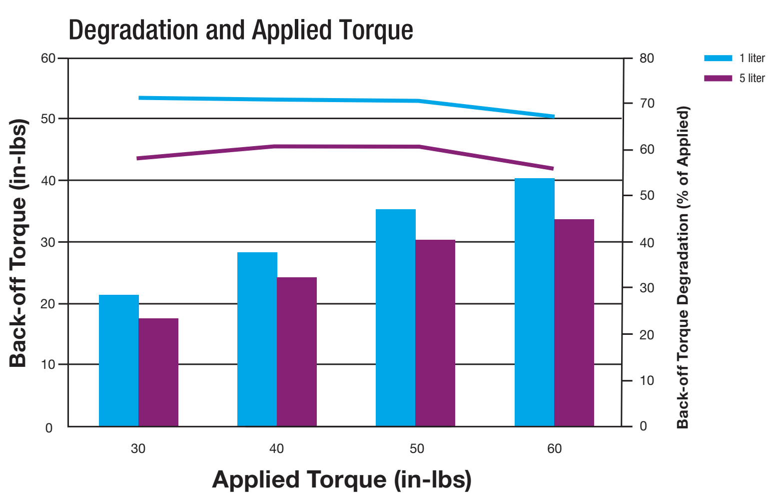

Figure 2. Back-off torque at multiple application torque values is shown in in-lbs (column chart, left axis) and degradation is graphed as percentage of applied torque (line graph, right axis). While higher application torques provide higher back-off torque requirements, the percentage of degradation remains fairly consistent over all torque values.

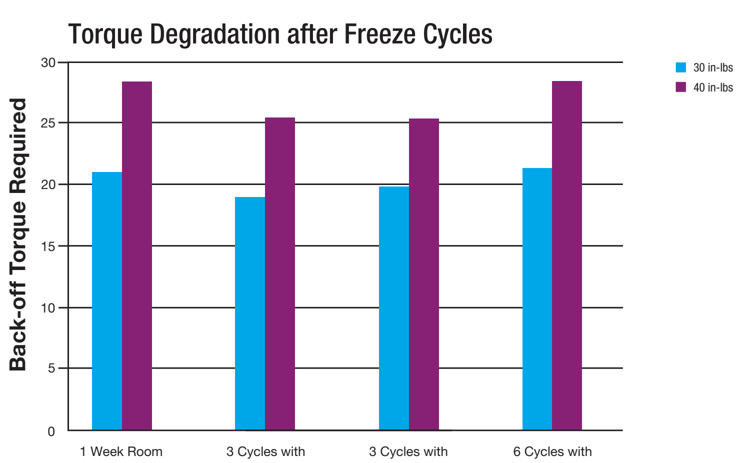

Figure 3. Back-off torque of Biotainer bottles containing water after 3 or 6 freeze/thaw cycles, Biotainer bottles containing SPG after 3 freeze/thaw cycles, or Biotainer bottles left at room temperature for a week with no freezing. All results represent data for 1L Biotainer bottles for comparison.

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Conclusion

- Back-off torque degrades gradually over time, but even after one month of storage bottles closed with the recommended minimum closure torque maintained seal integrity.
- Application torque does not affect the rate of degradation of back-off torque, as the percentage of degradation is maintained across the range of application torques.
- Multiple freeze-thaw cycles do not degrade the back-off torque, however retightening the closure after an initial freeze/thaw cycle is recommended.
- Bottle contents can impact the integrity of the seal. Therefore testing in the specific intended application is highly recommended before use.

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