

Design and innovation

Nalgene PETG bottle performance at -70°C

Keywords

Nalgene, PETG, ULT freezer, cold storage

Abstract

The purpose of this study is to extend the recommended temperature range for Thermo Scientific™ Nalgene™ PETG containers from -40°C to as low as -70°C . Back-off torque studies and pressure leak testing were performed on 1 L and 2 L bottles at -70°C and -40°C . Additionally, impact fracture studies were performed on bottles sterilized with two different sterilization doses, frozen at -70°C and then transferred to -40°C to mimic typical use. Torque degradation was greater at -70°C in comparison to -40°C during back-off torque studies for both 1 L and 2 L bottle sizes. However, pressure leak testing indicated that closure seal integrity was intact at both temperatures. Both bottle sizes survived impact at the equilibrated -40°C after -70°C storage. The use of Thermo Scientific™ Nalgene™ PETG bottles at -70°C is recommended based on the conditions evaluated; testing in actual use conditions is also recommended as results may vary depending upon application.

Background

Polyethylene terephthalate glycol (PETG) is a type of thermoplastic polyester. A thermoplastic is a polymer that becomes soft and pliable when heated without a change in its intrinsic properties. With these physical characteristics, PETG has been a commonly used material because it is a high-performance plastic that is lightweight with good impact strength, and can be used in a wide range of temperatures. PETG is also optically clear and BPA- and animal origin-free. PETG bottles are currently not recommended for use below -40°C . The purpose of this study is to extend the recommended temperature range for existing Nalgene PETG containers from -40°C to as low as -70°C . Here, we discuss the factors to consider when evaluating containers for sample leakage and loss.

Important considerations

Fill volume

Containers should not be filled with a volume greater than the recommended capacity. Additionally, bottles require adequate space during freezing to allow appropriate cooling of the fluid inside the container. Otherwise, the container may show evidence of cracking and/or crazing.

Cracking and crazing

A crack is the separation of an object or material into two or more pieces as a result of applied stress, resulting in leakage of the fluid from the container. Conversely, crazing is a cosmetic observance that does not show evidence of leaking. Crazing occurs at stressed regions and propagates perpendicular to the applied tension.

Sterilization by gamma-irradiation

Experimental samples should be irradiated to at least the highest dose that will be encountered during routine processing. A preferred and more conservative method is to irradiate samples at twice the anticipated maximum dose [1]. It is expected that bottles evaluated with a higher irradiation dose would have a higher failure rate because plastics subjected to irradiation during sterilization results in changes in the polymer structure. Irradiation creates free radicals that recombine to form crosslinks.

Crosslinking impedes the molecular movement of the polymer, resulting in an increase in thermal resistance and the improvement of mechanical strength and chemical resistance. Conversely, with chain scissioning, the polymer chains are broken and molecular mass decreases. Scissioning and crosslinking occur at the same time where one may predominate over the other, depending upon the polymer and the irradiation dose [2].

Torque specifications

Nalgene bottles have minimum and maximum applied torque specifications. The applied torque is the rotational force in which a closure is applied to a container. Applied torque affects the seal integrity of the bottle and the closure. Back-off torque is the rotational force necessary to open, loosen, or remove the closure. Torque degradation is the relationship between applied and back-off torque and establishes the sealing characteristics of the bottle and closure assembly. When evaluating samples, it is recommended that the minimum applied torque specifications are used in order to establish worst-case conditions in testing.

Pressure leak testing

After filling bottles to the nominal capacity and applying the closure to the proper torque specification, the ability to detect leaks is necessary to ensure the seal integrity of bottle and closure assemblies. A pressure apparatus can be inserted into the container in order to pressurize the container with a specified amount of pressure for a defined period of time so that fluid leakage can be visualized. This is a routine quality control test performed on various Nalgene bottles.

Table 1. Materials used in this study.

Description	Cat. No.
Nalgene Square Media Bottles	
PETG with HDPE closures, in shrink-wrapped trays, sterile, 1,000 mL, 38-430 closure, tray pack	342020-1000
PETG with HDPE closures, in shrink-wrapped trays, non-sterile, 1,000 mL, 38-430 closure, tray pack,	322020-1000
PETG with HDPE closures, in shrink-wrapped trays, sterile, 2,000 mL, 53B closure, tray pack	342020-2000
Chemicals	
Fisher Chemical™ Sodium Chloride	S271-1
Fisher Chemical™ Dextrose	D16-1
Environmental chambers	
Transformer (Acme Electric)	T-1-81052
Syle SR Micro Tenn™ II (Tenney Engineering Inc.)	T30R3
Other	
Hydraulic drop testing apparatus (Lansmont Corporation)	
Temperature data logger, USB-2416 (Measurement Computing Corporation)	
MCC DAQ Software Version 6.0 (Measurement Computing Corporation)	
Torque wrench and torque wrench adapters	

Methods

Visual observations

For each experiment, bottles were visually observed for physical deformities, including cracking or crazing.

Time to reach desired temperature

Containers were filled to the recommended capacity with a solution containing 1 g/L dextrose (glucose) and 9 g/L NaCl in water to mimic typical physiological conditions. Thermocouple wires were inserted through holes drilled in each closure and connected to a computer data logger. One thermocouple wire remained at room temperature as a control. Additionally, the frozen drop testing bottles were placed in the freezer in order to ensure that bottles used for testing reached the appropriate temperature (15x 1 L bottles and 15x 2 L bottles). Samples were placed in the -70°C freezer with adequate space and were arranged to prevent any location-based temperature bias. Once the -70°C temperature was obtained, the bottles were transferred to a -40°C freezer. Temperature data were collected every 10 seconds and the average of every 50 temperature readings was calculated. The data were normalized to 0°C , determined by the stabilization of temperature during the freezing phase in each container.

Impact fracture studies

1 L ($n = 30$) and 2 L ($n = 30$) bottles from inventory with the nominal sterilization dose were filled with test solution and were arranged in the -70°C freezer as previously described. After sufficient time for individual samples to reach -70°C and then -40°C , the samples were removed and were drop tested using a hydraulic drop test apparatus set at a height of 36 inches. The bottles were evaluated for breakage. After each impact fracture study, samples were examined for wall failures or deformities. Solutions were allowed to thaw and samples were examined for any solution leakage. 1 L Nalgene PETG bottles were sent to an external vendor for sterilization at two times the normal sterilization dose and were evaluated in the same manner ($n = 30$).

Freeze–thaw back-off torque degradation studies

Bottles from inventory were filled with test solution as previously described. Thirty samples of each bottle size at each temperature were tested. Closures were applied using a manual torque wrench to a specification of 27 in-lb (1 L bottles) or 38 in-lb (2 L bottles). Closure torque specifications were chosen based on the minimum quality control torque specifications for the bottles. After one freeze–thaw cycle, the same torque wrench was used to measure the maximum torque applied to remove the closure. The results for each condition were averaged and were reported as the decrease in torque as a percent of applied torque.

Pressure leak testing

Briefly, containers were filled and closures were applied using a manual torque wrench as described previously. 30 samples of each bottle size (1 L and 2 L) at each temperature (-70°C and -40°C) were evaluated after one freeze–thaw cycle. A pressure apparatus was inserted into the container and the 2 L and 1 L containers were pressurized for 2 minutes at 2 or 10 psi, respectively. A container was considered a failure if water escaped the closure.



Results

Visual observances

For each experiment, no bottles showed any evidence of deformity, cracking, or crazing.

Time to reach desired temperature

During -70°C storage, 1 L bottles were able to reach 0°C and cooled to -70°C slightly faster in comparison to the 2 L bottles, as expected. All bottles evaluated reached a final temperature of -70°C . One of the 2 L bottles required approximately 10 hours and 30 minutes to reach the desired temperature, which was the longest time observed (Figure 1). The bottles were transferred to a -40°C freezer and were continuously monitored with thermocouples. All bottles evaluated required approximately 2 hr 30 min to reach a temperature of -40°C (Figure 2). The times required to freeze were used as guidelines for future studies.

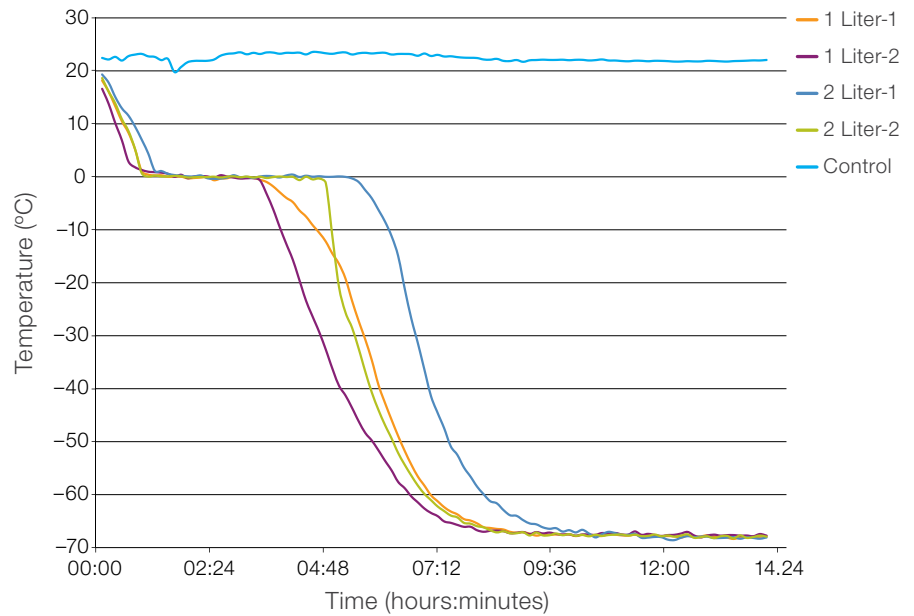


Figure 1. Averages of every 50 temperature readings over time for 1 L and 2 L Nalgene PETG bottles in a -70°C freezer.

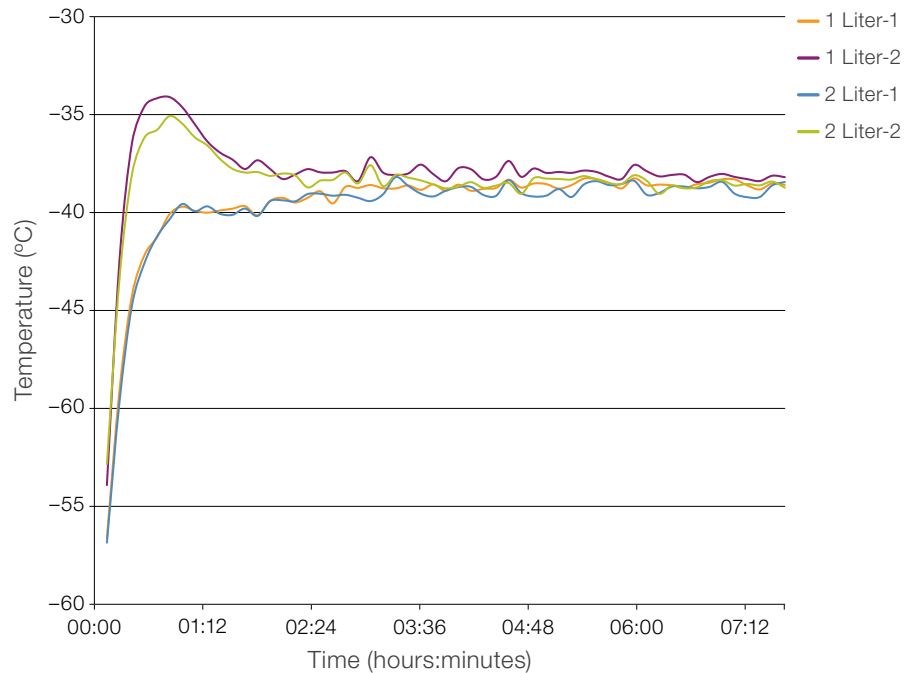


Figure 2. Averages of every 50 temperature readings over time for 1 L and 2 L Nalgene PETG bottles transferred from a -70°C freezer to -40°C freezer.

Impact fracture studies

None of the containers showed evidence of breakage or physical deformities at the nominal sterilization dose.

Two 1 L bottles containing twice the normal radiation dose (actual: 46–50 kGy) failed this test resulting in a 6.67% failure rate (Table 1). It is expected that bottles evaluated with a higher irradiation dose would have a higher failure rate, because plastics subjected to irradiation during sterilization acquire changes in polymer structure.

Freeze–thaw back-off torque degradation studies

1 L bottles showed 4.69% increased torque degradation and 2 L bottles showed 7.79% increased torque degradation after one freeze–thaw cycle at –70°C in comparison to one freeze–thaw cycle at –40°C (Table 2).

Pressure leak testing

After one freeze–thaw cycle at –40°C and –70°C, all 1 L bottles passed pressure leak tests at 10 psi for 2 minutes and all 2 L bottles passed pressure leak tests at 2 psi for 2 minutes (Table 3).

Discussion

The purpose of this study was to extend the recommended temperature range for existing Nalgene PETG containers from –40°C to as low as –70°C. In general, the performance of the two container sizes at the temperatures evaluated are comparable in the tests conducted.

Time to reach desired temperature

During initial testing, bottles were monitored for time to reach the appropriate temperature. These results were used as guidelines for testing that followed to ensure that the proper temperature was obtained.

Impact fracture studies

No failure was observed for either bottle size in frozen drop testing studies with nominal sterilization conditions when equilibrated to –40°C. 1 L bottles were drop tested with twice the normal sterilization dose, which is recommended for worst-case conditions, resulting in two failures (6.67% failure rate) when equilibrated to –40°C. It is expected that bottles evaluated with a higher irradiation dose would have a higher failure rate because plastics subjected to irradiation during sterilization will acquire changes in polymer structure. The risk here is minimal since customers typically will not encounter a need for such high sterilization dose.

Freeze–thaw back-off torque degradation and pressure leak testing

While 1 L and 2 L Nalgene PETG bottles showed better retention of closure torque after one freeze–thaw cycle at –40°C in comparison to –70°C, all bottles passed the pressure leak testing, indicating that closure seal integrity was maintained.

Table 1. Impact fracture study results of Nalgene PETG bottles stored at –70°C and then equilibrated to –40°C.*

Test condition	Sterilization dose	Sample size (n)	Number of failures	Failure rate (%)
1 L PETG bottle	Nominal	30	0	0
	Double	30	2	6.67
2 L PETG bottle	Nominal	30	0	0

* Upon immediate removal from –70°C storage, bottles have a high likelihood of fracture when dropped.

Table 2. Back-off torque results, shown as average actual torque required and as a percentage of the applied closure torque.

Test condition	Sample size (n)	Applied torque (in·lb)	Average back-off torque (in·lb)	Standard deviation	Torque degradation (% of applied torque)
–40°C	1 L PETG bottle	30	27	3.27	62.10
	2 L PETG bottle	30	38	3.13	57.19
–70°C	1 L PETG bottle	30	27	3.61	66.79
	2 L PETG bottle	29**	38	3.61	64.97

** For one sample, back-off torque was greater than the applied torque. This sample was removed from analysis.

Table 3. Pressure leak testing results.

Test condition	Pressure condition	Sample size (n)	Number of failures	Failure rate (%)
–40°C	1 L PETG bottle	2 min, 10 psi	0	0
	2 L PETG bottle	2 min, 2 psi	0	0
–70°C	1 L PETG bottle	2 min, 10 psi	0	0
	2 L PETG bottle	2 min, 2 psi	0	0



Conclusions

- Both 1 L and 2 L Nalgene PETG bottles performed comparably under simulated customer conditions (impact fracture studies).
- Both 1 L and 2 L Nalgene PETG bottles performed slightly better at -40°C in comparison to -70°C during back-off torque studies, and no bottles leaked during pressure leak testing, indicating that closure seal integrity was maintained.
- The use of Nalgene PETG bottles at -70°C is recommended based on the conditions evaluated; testing in actual use conditions is also recommended as results may vary depending upon application.

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Note: While the TSX Universal Series ULT Freezers were not utilized in this study of Nalgene PETG bottle performance at -70°C , they are part of the product offerings provided by Thermo Fisher Scientific.

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