Preventing leakage, contamination, and loss in critical applications

Nalgene PET and PETG Media Bottles

Abstract

To maintain product integrity, a container-closure system should be compatible with stored solutions and prevent content leakage, contamination, and the ultimate loss of precious resources. In addition, closure seal quality, integrity, and the ability to withstand accidental drops from benchtop heights are important considerations when choosing a media bottle for use in critical applications, especially when involving below-freezing temperatures. In this study, polyethylene terephthalate (PET) and PET glycol-modified (PETG) Thermo Scientific™ Nalgene™ Media Bottles of different sizes were evaluated in four key areas of bottle functionality to evaluate performance. Results were compared with findings for seven other commercially available media bottles to determine which features the best overall performance to aid in product selection. While many of the competitor brands examined in this study performed well in one or some of the practical challenges, the only bottles that excelled in all categories examined were Nalgene Media Bottles, making them the superior media bottle choice.

Introduction

Material compatibility and closure seal integrity are important aspects to consider when storing valuable contents. In order to maintain product integrity, it is important to carefully choose an appropriate container-closure system that will protect precious resources from sample loss due to leakage or contamination. PET and PETG resins have superior physical and chemical properties that render them well-suited for use in biological applications. Nalgene PET and PETG Bottles with HDPE closures help keep contained solutions safe by meeting the latest biological testing standards including USP Class VI, USP <661>, and ISO 10993-3, and are made with high-quality virgin resins that meet pharmaceutical, laboratory and food grade standards. Resins are carefully selected to minimize additives and reduce potential leachables. No plasticizers or fillers are added, and Nalgene Media Bottle plastics have lower total ash content, a measure of inorganic impurities, than the plastics used in competitive container offerings. In addition, the HDPE closures do not include a liner that can wear, corrode, crease, leak, or introduce contamination. Nalgene Bottles and Closures are engineered to work together with a proprietary valve seal and a strong, semi-buttress thread design. Both components are manufactured and inspected together to deliver a leakproof* guarantee.

To ensure adequate closure seal integrity, the application and removal torque should be measured to ensure the proper engagement of the threaded closure to the bottle, which is necessary to maintain a proper seal. The application torque is the rotational force by which a closure is applied to a container and affects the seal integrity of the bottle and the closure. Application torque degrades over time due to stress relaxation and the closure may start to "back off" the threads of the bottle neck. The removal, or back-off torque, is the rotational force necessary to open, loosen, or remove the closure. Back-off torque is influenced by time and other environmental variables such as transportation, vibration, temperature cycling, or compression. The relationship between applied and back-off torque is torque degradation. Torque degradation establishes the sealing characteristics of the bottle and closure assembly. A properly designed and applied closure will retain sufficient sealing force until the bottle is opened at the time of use.

Due to the sensitivity and potential for degradation of biologics at room temperature, many high-value substances are stored at very low temperatures. The ability to freeze solutions allows for many benefits during manufacturing, such as batch processing, increased shelf life, reduced microbial growth, and stable transport and storage options for the substance within, and may reduce costs associated with many aspects of product quality. However, water expansion during freezing strains plastic, and may result in crazes and cracks, and subsequently, leaks when the stored solution is thawed. In addition, the bottle-closure interface may experience expansion and contraction during freezing and thawing, which may potentially inhibit the effectiveness of the threaded seal. Of additional importance, particularly for pH sensitive contents including biological materials, is the ability to maintain the appropriate pH of the contained solution. Changes in atmospheric carbon dioxide (CO_2) during cold chain shipment can alter the solution pH if CO_2 gas enters the bottle.

Among the most important features of a container-closure system are closure seal integrity and sample protection at practical temperatures encountered in various applications. In this study, these aspects of media bottle functionality are examined in Nalgene PET and PETG Bottles, as well as in seven other bottle brands (Table 1). Nalgene PET and PETG Bottles are often implicated in freezing applications, due to the relatively low brittleness temperatures of each type of polymer. Here, bottles undergo impact fracture, pressure leak, back-off torque, and gas permeability testing after freeze-thaw cycles, to investigate these stressors on bottle performance.

Materials and methods

Bottle selection

Table 1 shows the bottle and closure parameters used in this study. Nalgene 30 mL and 1 L PETG Bottles were analyzed, in addition to 125 mL and 1 L PET Bottles manufactured in both our Rochester, NY and Newport, UK locations. Nalgene PET Bottles manufactured in both locations were evaluated to demonstrate that Nalgene Bottles deliver a robust and reproducible product across manufacturing locations. Nalgene Bottles were further challenged with a relatively high maximum gamma sterilization dose of 45 kGy. In addition, three lots of each Nalgene product were tested, to ensure repeatable results. One lot each of sterile competitor brand bottles was analyzed.

We provide recommended minimum and maximum applied torque specifications for our Nalgene Bottles (Table 1). Where competitor torque recommendations were not available, application torque values were devised based on those for similar Nalgene closure sizes, and the general principle that an appropriate application torque is approximately half the diameter of the closure.

[*] Our guarantee for a leakproof seal is subject to our standard product warranty, as set forth in the Thermo Fisher Scientific Terms and Conditions of Sale. Our products are leakproof at ambient temperature and pressure when used with their corresponding closures. However, customers are advised to test our containers and closures under conditions of their planned applications. Please contact **technicalsupport@thermofisher.com** if you need additional information about our products.

Table 1. Media bottle and closure parameters used in this study

Media bottle	Closure	Closure size (mm)	Application torque (in-lb)
PETG nominal volume 1,000 mL			
Nalgene media bottles: Cat. No. 342020-1000 (Lot No. 1183665, 1184397, 1178854)	Standard	38-430	27
Competitor A	Tamper-evident/ Standard*	38-430	26-27 (tamper-evident)/ 20-21 (standard)
PETG nominal volume 30 mL			
Nalgene media bottles: Cat. No. 342020-0030 (Lot No. 1184149, 1183661, 1173345)	Standard	20-415	10
Competitor A	Tamper-evident	20-415	10
PET nominal volume 1,000 mL			
Nalgene media bottles US: Cat. No. 342040-1000 (Lot No. 182760, 1183244, 1184855)	Standard	38-430	27
Nalgene media bottles UK: Cat. No. 842040-1000 (Lot No. M198580, M238850, 000099080)	Standard	38-430	27
Competitor B**	Tamper-evident	31.7	20
Competitor C**	Standard	45	30
Competitor D**	Tamper-evident	31.7	20
Competitor E	Standard	42	28
Competitor F	Standard	38-430	19
Competitor G**	Standard	42	28
PET nominal volume 125 mL			
Nalgene media bottles US: Cat. No. 342040-0125 (Lot No. 1182226, 1172046, 1177901)	Standard	38-430	27
Nalgene media bottles UK: Cat. No. 842040-0125 (Lot No. M194640, M211840, M221800)	Standard	38-430	27
Competitor B**	Tamper-evident	31.7	20
Competitor C**	Standard	45	30
Competitor D**	Tamper-evident	31.7	20
Competitor E	Standard	42	28
Competitor F	Standard	33-430	17
Competitor G**+	Standard	42	28

* Tamper-evident closures were applied for pressure leak, CO_2 ingress, and back-off torque tests, while standard closures were used during impact fracture testing.
** These manufacturers do not provide torque recommendations for their products.
\+ 100 mL volume was tested in this study.

Summary of testing procedures

All bottles were filled to nominal volume and torqued according to the minimum recommended manufacturer application torque (if provided) using a calibrated electronic torque meter, to ensure that worst-case conditions were represented for each test. Freezing temperatures used in this study (–40°C, –70°C, and dry ice) were verified using a calibrated environmental chamber, as well as a datalogger and type T thermocouples, during each freeze and thaw. Results for same-sized Nalgene PET Bottles manufactured in the United States and in the United Kingdom were averaged for each test. Equipment and consumables used in this study are noted in Table 2. Table 3 shows a detailed summary of each test.

Table 2. Equipment and supplies used for testing

Equipment/consumables	Manufacturer
Hydraulic Drop Test Apparatus	Lansmont Corporation
Tenney Environmental Chamber, Model T20C-4	Tenney
Portable Datalogger OM-SQ2040	Omega
Electronic torque tester	Kaps-All Packaging Systems
Thermo Scientific™ OrionStar™ Star A Series A111 pH Benchtop Meter	Thermo Fisher Scientific
Pressure leak vacuum apparatus	Manufactured in-house
Gibco™ PBS Powder	Thermo Fisher Scientific
α-D(+)-Glucose, ≥99%, anhydrous	ACROS Organics
Invitrogen™ UltraPure™ 5M NaCl	Thermo Fisher Scientific

Table 3. Testing parameters used in this study

Test	Bottle contents	Test samples	Control	Test conditions	Passing criteria
Impact fracture	1 g/L glucose, 9 g/L NaCl, deionized water	Freeze to –40°C	Room temperature	Drop from 36 in	<10% leakage
Pressure leak	Water, red dye	Freeze to –40°C, thaw x2 Freeze to –70°C, thaw x2	Room temperature	Pressurize at 2, 5, 7.5, and 10 psig for 2 min	No leakage at ≤10 psig
Back-off torque	Water	Freeze to –40°C, thaw Freeze to –70°C, thaw	Room temperature	Measure removal torque	Mean back-off torque within 40–60% of application torque
CO_2 ingress	PBS, pH 7.4±0.1	Freeze to –70°C, place in dry ice for ≥60 hr, thaw	Freeze to –70°C	Measure pH	End pH within starting pH range

Impact fracture studies

After freezing to –40°C, the test bottles (n=15) were placed onto a hydraulic drop test apparatus, and dropped from a height of 36 inches onto a steel base to mimic dropping from bench height. Control bottles of each brand (n = 15) were also dropped, after storing undisturbed at room temperature for the same amount of time as the frozen bottles. The test bottles were thawed, and upon reaching room temperature, all bottles were inspected for leaks.

Pressure leak testing

Two freeze-thaw cycles, freezing to either –40°C or –70°C in a calibrated freezer, were performed on the test bottles (n=30, except 1 L Nalgene PETG Bottles, n=15), while control bottles (n=15, except 1 L Nalgene PETG Bottles and Nalgene Bottles 1 L and 125 mL PET, n=7) were left undisturbed at room temperature for the same amount of time. A small hole was then drilled into the side of each bottle to accommodate tubing that connected an air pressure apparatus to the bottle. The bottles were pressurized at 2, 5, 7.5, and 10 psig for 2 minutes at each fixed pressure, or until the container-closure system failed. A container-closure system was considered a failure if water escaped the closure.

Freeze-thaw back-off torque degradation studies

Back-off torque testing was performed using a calibrated electronic torque meter on test bottles (n=d30, except 1 L Nalgene PETG Bottles, n=15) after one freeze-thaw cycle, freezing to either –40°C or –70°C in a calibrated freezer.

Control bottles (n=15, except 1 L PETG and Nalgene Bottles 1 L and 125 mL PET, n=7) were not frozen, but left undisturbed at room temperature for the same amount of time as the test bottle freeze-thaw cycle.

CO_2 ingress testing

The pH of a prepared batch of phosphate buffered saline (PBS) was recorded (pH 7.4±0.1). The media bottles were filled with the PBS and frozen to –70°C in a calibrated freezer. Test bottles (n=30, except 1 L Nalgene PETG Bottle and Nalgene Bottles 1 L and 125 mL PET Bottles, n=7) were then transferred to a dry ice bunker, where they remained undisturbed for at least 60 hours, to simulate the storage process during cold shipment. Control bottles (n=15, except 1 L PETG bottle brand A, n=6, all 1 L and 125 mL Nalgene Bottles, n=7) remained in the –70°C freezer until test bottles were removed from the dry ice environment. The bottles were allowed to thaw to room temperature, and the pH of saline in each bottle was recorded using a calibrated pH meter.

Results and discussion impact fracture studies

Upon immediate removal from –40°C storage, bottles have a high likelihood of fracture when dropped and extra caution should be taken while handling frozen bottles. Overall failure rates of competitor brands were higher than Nalgene Bottles in all cases, except competitor F, which who performed poorly in several of the other tests (Figure 1).

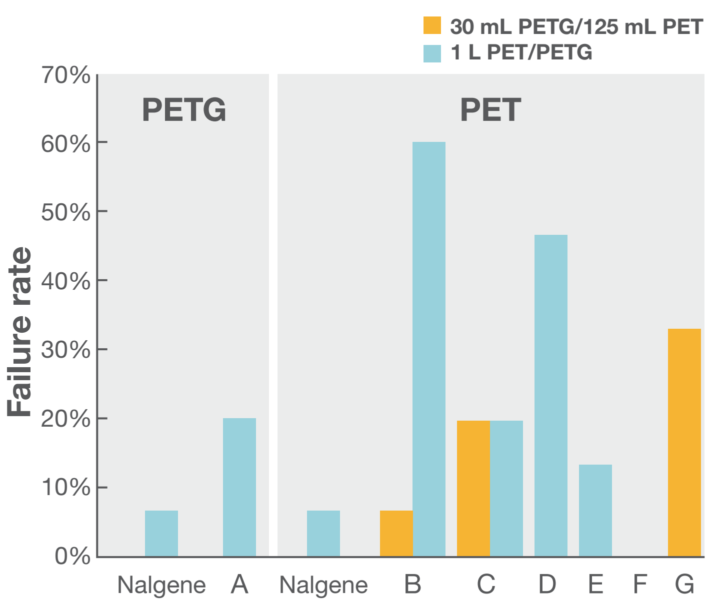

Figure 1. Impact fracture studies results

Pressure leak testing

Pressure leak testing is performed on all Nalgene Bottles as part of our routine quality control procedures. As expected, all Nalgene PET and PETG Media Bottles surpassed the cumulative pressurization to 10 psig without leaking after room temperature storage and after two freeze-thaw cycles at either –40°C or –70°C. Failure rates for most competitor bottles were higher than Nalgene Bottles. Results can be found in Table 4. The pressure (psig) at which the first failure occurred is shown for each bottle type. If test or control bottles did not leak after 2 minutes at 10 psig, "No failures" was reported. The "Failure rate (%)" represents the cumulative percent of bottles that failed after 2 min at testing up to 10 psig at all temperatures studied.

Table 4. Pressure leak testing results

PETG	30 mL	Failure rate (%)	1 L	Failure rate (%)
Nalgene	No failures	0	No failures	0
A	0 psig	85	0 psig	25
PET	**125 mL**	**Failure rate (%)**	**1 L**	**Failure rate (%)**
Nalgene	No failures	0	No failures	0
B	No failures	0	5 psig	3
C	5 psig	92	5 psig	100
D	2 psig	97	No failures	0
E	No failures	0	No failures	0
F	10 psig	1	10 psig	5
G*	No failures	0	2 psig	1

* 100 mL

Freeze-thaw back-off torque

There is a common misconception that the tighter the closure is applied, the better the seal; however, overtightening can cause some points on the closure to become more pressurized than others, and can cause the closure to miss threads, strip threads, or break, creating an improper seal. The standard acceptable removal torque in the plastics industry is between approximately 40% and 60% of the recommended application torque at least 24 hours after the closure is applied. As illustrated by four bottle brands in Figure 2, leaks during the pressure leak test (denoted with *) may be attributable to the high (>60% of applied) torque required to remove the closures. Three brands exhibited back-off torque within the 40–60% recommended range, but also leaked during the pressure leak test. An easy-to-remove closure assembly (a lower average back-off torque as a percent of the applied torque) may be desirable, as it lessens operator strain, but only with a guarantee that the bottle will not leak, as is provided by Nalgene Media Bottles.

Back-off torque measurements for tamper-evident closures are typically higher than those for standard closures, due to the presence of the tamper-evident seal ring (as evidenced by results for Competitor A). Slip torque identifies the torque required to initiate rotation of the cap around the thread of the bottle. Bridge torque identifies the torque required to initiate rupturing of the bridge, or webbed portion, of the closure. Removal torque values for tamper-evident closures represent measurements for the slip torque, not the bridge torque.

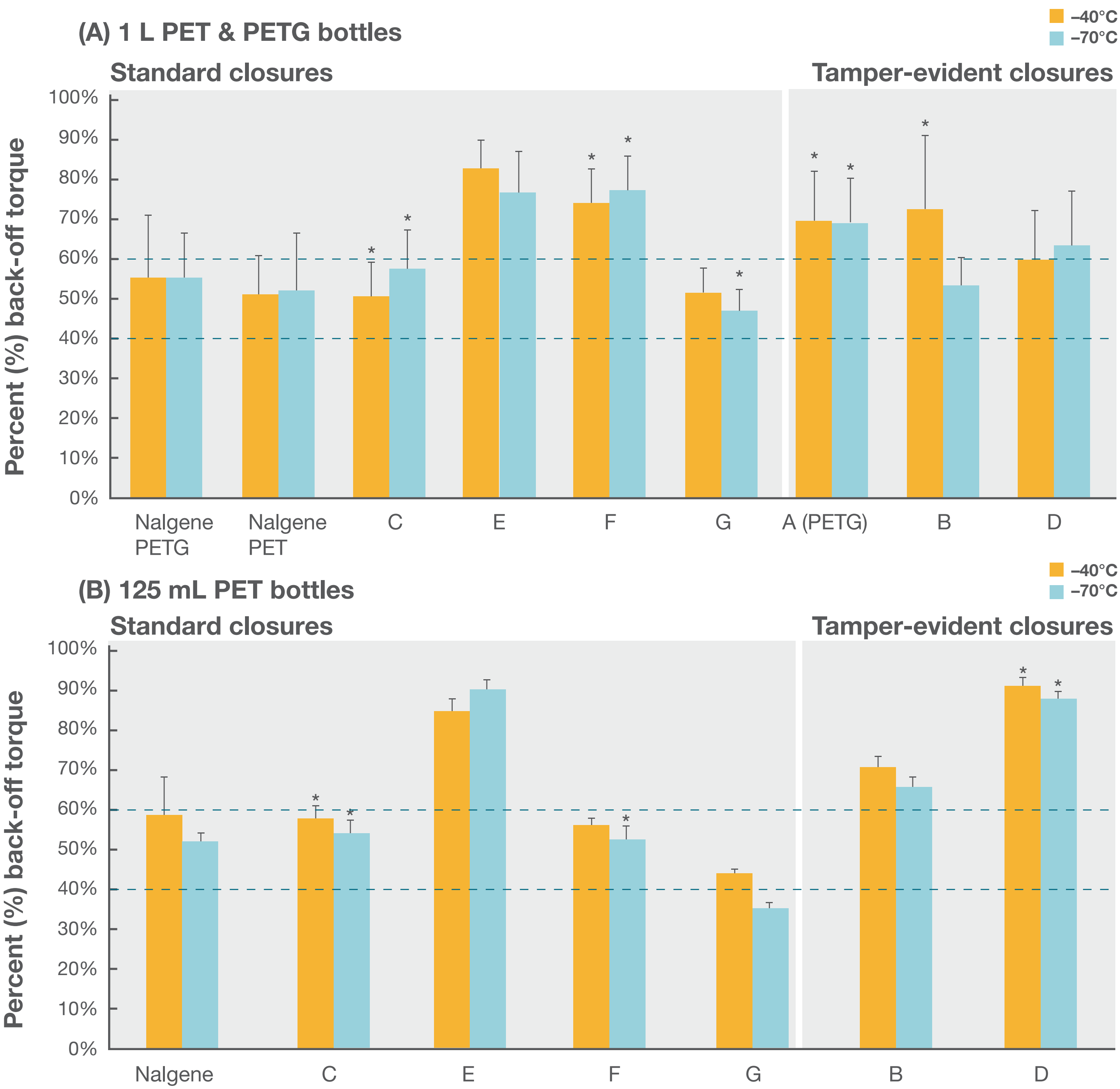

Figure 2(a-b). Percent back-off torque (± 1 SD) applied to (a) 1 L bottles and (b) 125 mL bottles after freezing to either –40°C or –70°C. The horizontal dashed line indicates the accepted range for back-off torque (40–60% of applied torque). Note that the volume of brand G bottles is 100 mL. * Bottles from these groups failed during pressure leak testing.

CO_2 ingress

Saline pH measurements show that many tested bottle brands were able to resist CO_2 ingress into 1 L PET and PETG bottles during prolonged periods of dry ice exposure. The contents of Nalgene 30 mL and 1 L PETG and 1 L PET Bottles experienced insignificant shifts in pH after dry ice exposure when compared with control samples stored at –70°C. Results for 30 mL and 1 L Nalgene Bottles and competitor bottles are shown in Figure 3.

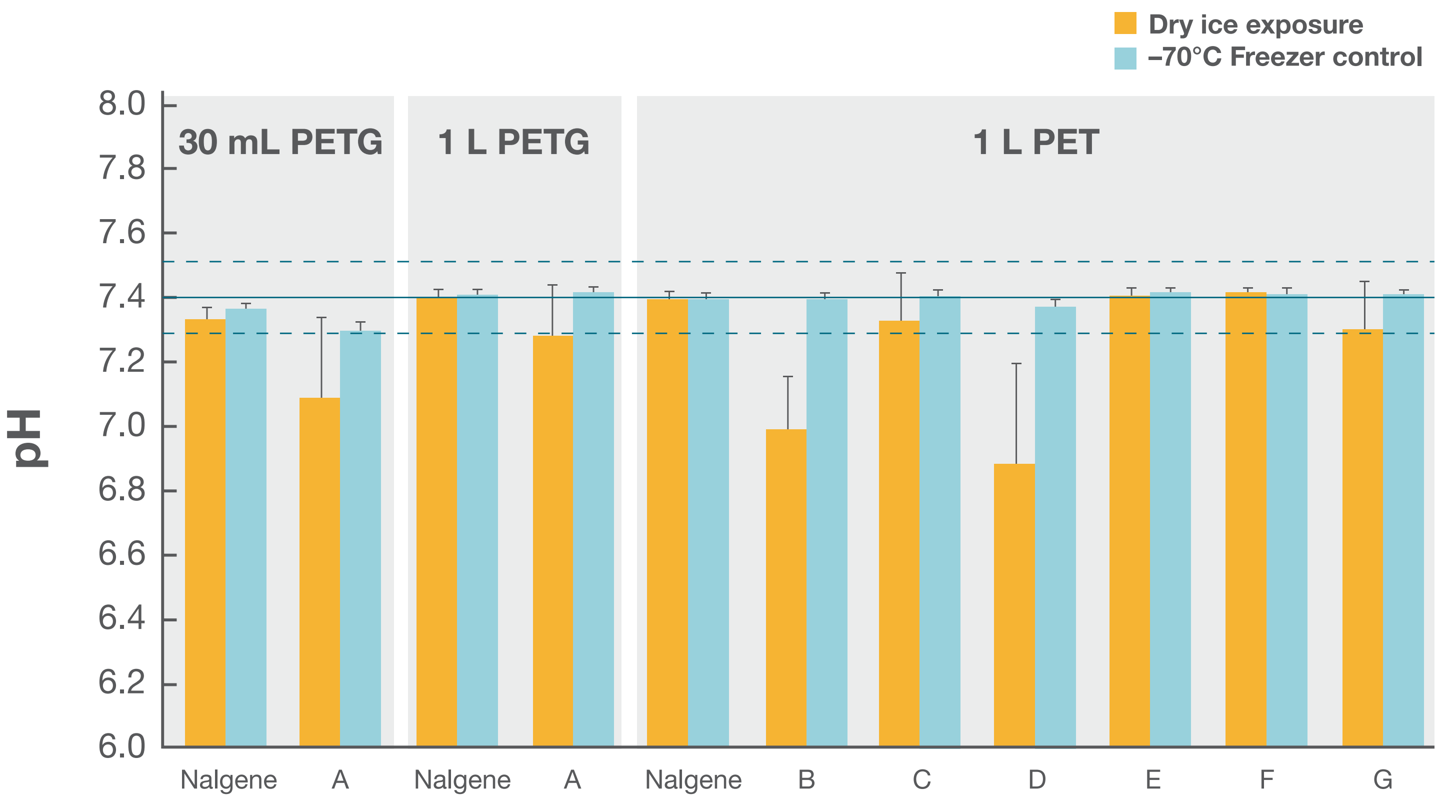

Figure 3. CO_2 ingress (pH) measurements (± 1 SD) for 30 mL PETG and 1 L PET Nalgene and competitor bottles after dry ice exposure compared with control bottles stored at –70°C. The horizontal dashed line indicates the starting pH range (7.4±0.1) of the phosphate buffered saline used to fill bottles, and the solid line represents a pH of 7.4.

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Conclusion

In this study, Nalgene Media Bottles were challenged at low temperatures and were evaluated for impact fracture, pressure leak, back-off torque degradation, and CO_2 ingress performance. Results were compared with seven competitor brands of bottles. No competitor was able to meet the strong functional performance exhibited by Nalgene Bottles (Table 5), making Nalgene PET and PETG Media Bottles the superior media bottle choice to keep your precious contents safe and secure.

Key: - denotes a failure

Table 5. Summary of results

	Impact fracture	Pressure leak	Torque degradation	CO_2 ingress
Nalgene PETG	Pass	Pass	Pass	Pass
Competitor A	-	-	-	-
Nalgene PET	Pass	Pass	Pass	Pass
Competitor B	-	-	-	-
Competitor C	-	-	Pass	Pass
Competitor D	-	-	-	-
Competitor E	-	Pass	-	Pass
Competitor F	Pass	-	-	Pass
Competitor G	-	-	-	Pass
Passing criteria	<10% leakage	No leakage at ≤10 psig	Mean back-off torque within 40-60% of application torque	Ending pH 7.4±0.1

Find out more at **thermofisher.com/mediabottles**

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