NRC·CNRC

Test Report

Intermediate Bulk Container Life Extension Research

Prepared for: Transport Canada
330 Sparks St.
Ottawa, ON K1A 0N5

Prepared by: Todd Garrett
Don Leblanc
Patrick Kehoe
Damien Maillard

National Research Council of Canada
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Prepared by:

for, **Todd Garrett, BASc., P.Eng.**
Test Engineer, Testing and Evaluation

Don LeBlanc, BASc (Hons.), P.Eng.
Manager, Climatic Testing Research Facility

Patrick Kehoe, P.Eng.
Vibration Engineer

Damien Maillard, Ph.D.
Research Officer

Reviewed by:

Bruce Gaudet, P.Eng.
Test Engineer, Testing and Evaluation

Jon Preston-Thomas, P.Eng.
Principal Engineer, Transportation Engineering Centre

Approved by:

Philip Marsh, P.Eng.
Director R&D, Transportation Engineering Centre

Executive Summary

The NRC Automotive and Surface Transportation (NRC-AST) Research Centre was tasked by Transport Canada (TC), in the context of a lifespan testing program, to evaluate the performance of a selection of rigid plastic and composite intermediate bulk container (IBC) designs when subjected to a range of performance tests as prescribed in CAN/CGSB-43.146-2016 [1]. In addition, a range of materials tests was performed to characterize the polymers used in the inner bottles, assess the condition of the materials and investigate any indications of damage.

UN standardized plastic and composite IBC prototypes must first pass a set of performance tests that includes bottom lift, top lift, stacking, leakproofness (air), hydraulic pressure (water), cold drop and vibration, before the design can be registered and manufactured. Rigid plastic IBCs and inner bottles of composite IBCs are only permitted to be used for dangerous goods transport for a period totalling 60 months from their date of manufacture, after which time they must be removed from service, or in the case of composite IBCs, the inner bottle must be replaced. The goal of this study was to determine if IBCs that had reached or exceeded the 60-month period could still successfully pass the performance tests of the CAN/CGSB-43.146 standard. A total of at least three samples of each IBC design type was tested, with the exception of composite IBCs, where the number of metal frames available was limited. All IBCs and inner bottles sourced for this study met the criteria of being beyond their prescribed period of use of 60 months by approximately one to two years.

Three IBC designs provided by users to TC/NRC were subjected to testing. The designs were given a code letter, "A" and "B" for the two composite designs and "C" for the rigid plastic design. Results were as follows, with a pass result denoted by a "✓", and a fail result denoted by an "X":

Test	**Manufacturer and IBC identifier**																								
	A							**B**							**C**										
	1	2	3	4	5	6	7	1	2	3	4	5	6	7	1	2	3	4	6	7	8	9	11	13	16
Bottom lift	✓	✓	✓					✓	✓	✓					✓	✓	✓								
Top lift			X							X					✓	✓	✓								
Stacking	✓	✓						✓	✓						✓	✓	✓								
Leakproofness		✓		X		X				✓	X		X		✓			X	X						
Hydraulic pressure		X		✓		X				X	X		✓		X			X	X						
Flat drop (ambient)				✓														X							
Flat drop (cold)												✓										X			
Corner drop (ambient)														X						X					
Corner drop (cold)					X																X		X	X	
Vibration							X		✓																✓

Note: IBC B5 passed the flat drop test (cold) from a height of 0.8 m vs 1.9 m

The following describes the findings displayed in the figure above:

Bottom lift: All nine IBCs passed.

Top lift: Plastic IBCs passed but two failures were observed on metal frames of the composite IBCs. The issue was mechanical failure of a lifting eye and further testing was suspended to preserve the sample frames for later tests, noting also that metal components were not the focus of this study.

Stacking: All nine IBCs passed.

Leakproofness (air): Only three of nine IBCs initially passed the 20 kPa leakproofness test, leaking past their closures. It was also noted that air leakage occurred past the pressure relief devices that were rated above the test pressure. Several 2-in. closures on the plastic type IBCs were found to have broken, with a likely cause determined to be over torquing of the closure. The closures appeared to have chemical residues on their exterior which may be an indication that leakage occurred in service. Six IBCs were retested with new gaskets and all but one passed at the rated torque application.

Hydraulic pressure (water): Only two of the nine IBCs ultimately passed the test, at a pressure of 100 kPa. As expected, failures occurred at higher pressures than those where failures were observed in the leakproofness test. Deformation on one composite IBC was such that the bottom discharge valve was inoperable during testing and the locking mechanism was damaged. Of the six IBCs tested with new gaskets, only three passed the test.

Drop tests: Only one of six IBCs passed the cold drop test, at a reduced drop height of 0.8 m. Only one of four IBCs passed a drop test at ambient temperature.

Vibration: Only one of each type of IBC was vibration tested, with one clear failure, one clear pass and one with a developing fault.

Materials: Materials testing of the inner bottle walls generally showed some degradation/oxidation of the outer and inner surface layers, but the bulk materials were intact. Gasket materials did show significant material degradation. Examination of 2-in. bung material indicated that over torquing of the closure was a likely cause of observed failures. As well the 2-in. bung closure gaskets showed evidence of significant hardening and degradation, which may explain why more force was used to seal it while in use.

Based on the results observed, the five year old IBCs do not continue to meet the performance requirements of the standard.

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1 Introduction

The NRC Automotive and Surface Transportation (NRC-AST) Research Centre was tasked by Transport Canada (TC), in the context of a lifespan testing program, to evaluate the performance of a selection of rigid plastic and composite intermediate bulk containers (IBCs) beyond their prescribed period of use when subjected to a range of performance tests as prescribed in CAN/CGSB-43.146-2016 [1]. In addition, a range of materials tests were performed to characterize the polymers used in the inner bottles, assess the condition of the materials and investigate any indications of damage.

1.1 Purpose

The purpose of this study was to evaluate the potential for extending the prescribed period of use of certain plastic IBCs beyond the five year (60 months) limit. This was achieved by conducting performance testing on plastic IBCs which were beyond their prescribed period of use to determine if they could meet the same performance-based standards as newly manufactured ones.

1.2 Background

The United Nations Model Regulations [2] restricts the use of plastic IBCs for the transportation of Dangerous Goods to a maximum prescribed period of use of five years (60 months) from their date of manufacture (except where a shorter period of use is prescribed based on the type of dangerous goods to be transported).

The Transportation of Dangerous Goods (TDG) directorate at TC has adopted this prescribed period of use within its container safety standards. The data from this test program will assist the TDG directorate in better understanding the durability and performance of these IBCs once they are beyond their prescribed period of use, and may assist in future decision-making related to these IBCs.

1.3 Scope

A number of sample IBCs were provided to enable this study to take place. Because it is expected that the condition of each of the IBCs will have potentially deteriorated to slightly different degrees, based on variabilities within the distribution and use cycles, it is not a matter of simply testing a small sample set of IBCs as is typical for the initial design qualification tests. New IBCs should be of a much more consistent quality and performance level. Accordingly, it was decided that a greater number of IBCs should be subjected to the series of performance tests, and therefore a variety of IBC designs from several different suppliers were obtained for this study. Three types of UN standardized IBCs were obtained for study, all of which were beyond the prescribed period of use of 60 months. This included one IBC design type certified and marked as a rigid plastic design (UN31H1), and two composite IBC designs with plastic bottles in metal frames (UN31HA1).

The IBCs were subjected to bottom lift, top lift, stacking, leakproofness (air leakage), hydraulic pressure (water), ambient drop, cold drop, and vibration tests as prescribed by the CAN/CGSB-43.146 standard.

Originally, a minimum of three IBCs of each design type were to be tested. Unfortunately, only a limited number of metal frames could be obtained for the study and thus frames were reused in some cases to preserve those in good condition to be used in later tests. It was determined that this would not impact the overall results in any significant way.

Materials testing was directed on the basis of results observed during the physical tests. The goal was to choose materials tests which would help distinguish IBCs which failed design tests from those which passed, which might identify factors causing failures.

1.4 Limitations

The limited sample size subjected to each of the performance tests, in relation to the IBC population of these exact types in service, presents difficulties from a statistical standpoint. It was difficult to obtain a wide variety of IBC types in use for this study. Three types were ultimately obtained from users.

As is often the case with products that are relatively valuable, it is difficult to justify testing large, statistically significant sample sizes. Failures in this sample size will be considered in the context of the intent of this Study. To better understand the failures observed, testing of a larger sample would be required. Though the results apply only to the parts tested, conclusions can be drawn about the general population from them, albeit at low confidence levels. No known history of use over the lifespan of the IBCs while in service (e.g., type of dangerous goods transported, storage conditions) was able to be obtained for the sample IBCs obtained for this study.

2 Apparatus and test equipment

2.1 Intermediate bulk containers under test

Two different design types of IBCs, as described in Table 1, were sourced by the NRC to be subjected to the performance and materials tests. Of the two design types, sample IBCs produced by three manufacturers were selected from the available samples. Two of the manufacturers produced the 31HA1 composite type (designated Manufacturer A and Manufacturer B), and one of the manufacturers produced the 31H1 type (designated Manufacturer C). The inner bottles of the composite IBC design type and the rigid plastic IBC design type were all beyond the prescribed period of use of 60 months. The IBCs were all manufactured in 2014, thus they were 1 to 2 years past their expiry date. The IBCs were stored indoors during the test period to protect them from environmental exposure.

IBC code	Manufacturer	Material of construction		Description
		Frame	Inner bottle	
31HA1	A, B	Steel	Plastic	Rigid, designed for transporting liquids, loaded or unloaded by gravity, with rigid plastic inner bottle
31H1	C	Plastic	Plastic	Rigid, designed for transporting liquids, loaded or unloaded by gravity, fitted with structural equipment

Table 1: Design type and description of IBC test samples

The 31HA1 design type IBC includes a rotationally molded bottle within an outer welded steel frame including “kick plates” and an openable access to the 2-in. bottom discharge valve. Each had a 6-in. top mounted closure which incorporated a pressure relief fitting rated for 34.5 kPa (5 psi) according to markings present. A set of black pillow pads was supplied with each of the 31HA1 IBCs.

The 31H1 design type includes a blue rotationally molded polyethylene outer frame comprising two main pieces. The inner bottle is also rotationally molded polyethylene and is fitted with a 6-in. closure on the top surface, two 2-in. drum type closures, and top lifting eyes made of steel and configured to hinge out of the way when not in use. A 2-in. bottom discharge valve is installed with no pressure relief devices fitted.

Several IBCs of each design type were obtained directly from end users with specific IBCs selected by the user. Only the inner bottle, closure and pillow pads were provided for the composite style IBCs, with no steel frames provided. The NRC had three frames in good condition from the same supplier of the inner bottles designated as Manufacturer A which had been used for previous work with TC. In addition, three new frames were obtained from Manufacturer A. Inner bottles from Manufacturer A and Manufacturer B appear to be of virtually identical design and given the limited availability of frames, for the purposes of this study it was deemed an acceptable option to cross-bottle.

2.2 Instrumentation

A range of instrumentation was used during the IBC performance tests to measure and record pressure applied during the leakproofness and hydraulic pressure tests; IBC weight prior to the bottom lift, top lift, stacking and drop tests; displacement during the stacking test; accelerations during the vibration test; and temperatures where applicable.

As shown in Table 2, four IMC Cronos data acquisition systems (DAS) were used to record the instrumentation signals for these performance tests. Each DAS is equipped with 24-bit sigma-delta converters that provide a 16-bit final resolution and simultaneous sampling.

The DAS control software, Studio V5.0 by IMC, was used to configure the DAS and record data during all the tests. Pressure, temperature and acceleration data were measured and saved with two steps. In the first step, the pressure and temperature data was sampled at 10 Hz and acceleration at 2 kHz using an 8th order Cauer antialiasing filter. For the pressure and temperature data the pass band corner was set at 4 Hz with a pass band gain uncertainty of -0.1 dB, and the stop band corner set at 6 Hz with a stop band gain uncertainty of -80 dB. For the acceleration data the pass band corner was set at 800 Hz with a pass band gain uncertainty of -0.1 dB, and the stop band corner set at 1,200 Hz with a stop band gain uncertainty of -80 dB. In the second step, the preprocessing feature was used with an arithmetic mean factor of 10 selected, so the pressure and temperature data gets recorded at 1 Hz, the acceleration data at 200 Hz, and each recorded data point is the mean of ten sampled and filtered values.

Type	Model	Serial No.	Calibration expiry	Signal description
IMC Cronos	CRC-400-17	142225	2021-08-19	Temperature
IMC Cronos	CRC-400-17	142226		Liquid/air pressure
IMC Cronos	SL-4	142735	2022-08-17	Weight
IMC Cronos	CRC-400-11	141500		Temperature

Table 2: Data acquisition systems used to record transducer measurements in the IBC performance tests

As shown in Table 3, five different types of transducers were used to measure signals for these performance tests. A total of 20 thermocouples permanently installed in a grid in the climatic facility were used to provide feedback to the control system and maintain a temperature set point on average within the facility. Each of the IBCs had one of the three pressure sensors installed in either the closure or inner bottle body for the leakproofness and hydraulic pressure tests. Weight measurements for the bottom lift, top lift, stacking, drop and cold drop tests were taken at the four corner locations using one of the weigh scales and summed while linear potentiometers were attached at all four corners to monitor any displacement resulting from frame deformation.

Signal description	Sensor manufacturer, model no.	Serial No.	Range	Accuracy	Recording rate, Hz	Calibration expiry
Pressure	Setra, C206	1279515	0 to 690 kPag (0 to 100 psig)	±0.13% F.S.	1	2021-08-03
		1252695				2021-08-03
		1279519				2021-08-03
Weight	Massload, ML-SLIM-MN4K	71091	0 to 907 kg (0 to 4,000 lb)	±0.5% F.S.	1	2020-10-05
		71085				
		71090				
		71083				
	Massload, ML-700	71195	454 to 11,340 kg (1,000-25,000 lb)	±0.1% F.S.	1	2020-10-05
Temperature	Type-T	Various	-270 ºC to +370 ºC	±0.5 ºC	1	2021-05-01
Acceleration	3741B1230G	12017	±30 g	±5%	2,000	2022-03-09
Displacement	Celesco, PT101-0015-111-1140	H2621724C	38.1 cm (15 in.)	0.15% F.S.	1	2022-08-25
		H2621726C				
		I2861684				
		H2621722C				

Table 3: Transducers used to measure signals in the IBC performance tests

2.3 Test facility

The NRC climatic testing facility measures approximately 30.5 m (100 ft) long by 6.1 m (20 ft) wide and 6.7 m (22 ft) high. Refrigeration systems provide cooling to below -40°C, while electrical heaters provide the capability to heat to +55°C. Variable speed portable fans can generate spot winds or generalized air circulation in the climatic facility. Refrigerated make-up air can be supplied to the climatic facility, and exhaust products can be extracted from it. Portable steam humidifiers can raise climatic facility humidity to controlled levels. A control room along one side of the climatic facility contains climatic facility control equipment. There is direct personnel access to the climatic facility through three doors and tests inside the climatic facility can be observed through three viewing ports. The facility has a 61 cm thick concrete floor heavily reinforced with steel rebar.

The NRC dynamics bay facility is equipped with multiple hydraulic actuators designed for reproducing dynamic motion encountered in the transportation industry. At approximately 70 m long and 8 m wide, full rail or road vehicle simulations can be performed under precisely controlled conditions. As well the facility incorporates a wheel bearing and brake rig used to simulate rail wheel interface forces.

A single 178 kN hydraulic actuator was set up to conduct the testing with uniaxial motion in the vertical direction. A steel support plate approximately 1.5 m x 1.5 m was bolted to the actuator to support the individual IBCs. The mass of the support plate was 1,134 kg. The minimum plate resonant frequency was determined to be approximately 200 Hz and the oil column frequency of the actuator was identified at 50 Hz, both of which would not affect the test frequency of approximately 4.5 Hz. Corner lift rings were used to hoist the plate into position on the actuator, and it was secured with eight M20 x 90 mm long socket head cap screws. The IBCs were hoisted into position on the steel plate and centred within ±20 mm. Exact positioning was deemed unnecessary due to the amount of motion experienced during the test run. Four aluminum angle retaining walls (75 mm x 75 mm x 6.4 mm thick), each with three neodymium magnets (rated to 59 kg per angle), were used to secure the IBC to the steel plate to prevent it from “walking-off” while vibrating. During the initial trials, the specimen was still experiencing “walk-off” and several C-clamps were added to help secure the retaining walls. IBC C16 required a higher side wall, so 2x4 wood was added to the retaining wall. Four rubber bungee cords were used to prevent the support plate from rotating while under test, two securing the southeast corner of the plate to the wall and two securing the southwest corner to the wall. Safety railing was added along the perimeter of the upper floor to prevent observers from falling into the test area at the lower floor.

3 Test procedure

3.1 Overview

Test procedures described within Section 3.2 to Section 3.8 herein follow the National Standard of Canada CAN/CGSB-43.146-2016 standard, except where otherwise noted, with the relevant test section from the standard and the date of test performance shown in Table 4.

Test	CAN/CGSB-43.146-2016 section	Test performance date
Bottom lift	7.3	2020-11-20
Top lift	7.4	2020-12-03
Stacking	7.5	2020-12-09
Leakproofness	7.6	2021-02-19
Hydraulic pressure	7.7	2021-02-19
Flat drop	7.8	2021-03-12
Corner drop	7.8	2021-03-12
Flat drop (cold)	7.8	2021-06-25 to 2021-06-29
Corner drop (cold)	7.8	2021-06-25 to 2021-06-29
Vibration	7.13	2021-09-29 to 2021-10-01

Table 4: Type, standard reference and dates of tests conducted on IBC samples

3.2 Bottom lift test

The following sequence of steps was followed in performing a bottom lift test of three IBCs from each of Manufacturer A, Manufacturer B and Manufacturer C.

1. The IBC was filled with water and sand to achieve the maximum permissible gross mass.
2. A further 25% of the maximum permissible gross mass was added by applying lead weights and rubber mats evenly distributed on top of a sheet of plywood, to the top of the IBC.
3. The IBC was raised and lowered twice from each of the four directions of entry. IBCs were lifted such that they cleared the ground surface and were held in position for a minimum of 10 s.
4. A visual inspection of the IBC was performed and any permanent deformation of the structural equipment or release of the IBC contents was noted.

3.3 Top lift test

The following sequence of steps was followed in performing a top lift test of three IBCs from Manufacturer C and one IBC from both Manufacturer A and Manufacturer B.

1. The IBC was filled with water and sand to achieve the maximum permissible gross mass.
2. A further load equivalent to 100% of the maximum permissible gross mass was assembled and hung with chains off the top of the IBC using two I-beams, chains and a plywood base. The I-beams were configured so as to spread the load evenly while not interfering with the lifting chains in either the straight lift or 45° lift.

3. The IBC was raised twice in total, once using each pair of diagonally opposite lifting devices configured so that the hoisting forces were applied vertically, and held in a raised position for 5 min.
4. A visual inspection of the IBC was performed and any permanent deformation of the structural equipment or release of the IBC contents was noted.
5. The IBC was raised twice in total, once using each pair of diagonally opposite lifting devices configured so that the hoisting forces were applied toward the center at 45° to the vertical, and held in a raised position for 5 min.
6. A visual inspection of the IBC was performed and any permanent deformation of the structural equipment or release of the IBC contents was noted.

3.4 Stacking test

The following sequence of steps was followed in performing a stacking test of three rigid plastic IBCs from Manufacturer C:

1. The IBC was filled with water and sand to achieve the maximum permissible gross mass, and soaked overnight in a climatic chamber at 40°C prior to loading.
2. A reproduction of the base of the IBC was manufactured by partially disassembling a separate identical frame. A concrete mixture was poured into the concave bottom section, lined with a section of tarp. Steel boxes containing lead as well as rubber mats were added to this resulting flat base to achieve a target mass of 1.8 times the mass that will be supported by the IBC during transport.
3. String potentiometers were attached at all four corners to monitor any displacement resulting from frame deformation.
4. The IBC was subjected to the test load for a duration of 28 days at 40°C.
5. A visual inspection of the IBC was performed and any permanent deformation of the structural equipment or release of the IBC contents was noted.

The following sequence of steps was followed in performing a stacking test of two composite IBCs from both Manufacturer A and Manufacturer B:

1. The IBC was filled with water and sand to achieve the maximum permissible gross mass.
2. A load spreading base of laminated plywood sheets was cut to size such that it fit within the top load bearing area, and of a sufficient height as to not interfere with the top lifting eyes. A large steel plate, nominally 6.4 cm (2.5 in.) thick, with lifting eye bolts in each corner suitable to lift the test load safely, was used as the load carrier. Steel boxes containing lead as well as rubber mats were loaded onto the steel plate to achieve a target mass of 1.8 times the maximum number of fully loaded IBCs that may be stacked on a bottom IBC per the manufacturer’s design.
3. String potentiometers were attached at all four corners to monitor any displacement resulting from frame deformation.
4. The IBC was subjected to the test load for a duration of 24 h.
5. A visual inspection of the IBC was performed and any permanent deformation of the structural equipment or release of the IBC contents was noted.

3.5 Leakproofness test

The following sequence of steps was followed in performing a leakproofness test of three IBCs from each of Manufacturer A, Manufacturer B and Manufacturer C.

1. The IBC was instrumented with a pressure transducer and an airline fitting for pneumatic pressurization.
2. A torque of 102 N-m (75 lbf-ft) was applied initially to the closures as per instructions found either on the enclosures or in the manufacturer's literature.
3. An air pressure of 20 kPa (3 psi) was applied to the body of the IBC for 10 min.
4. In the event a leak was detected, hot melt glue was applied to seal relief valves in the vented closures and prevent venting, at which point pressurization resumed. If leakage was still present at the main closure threads, the closures were further torqued to 136 N-m (100 lbf-ft) and pressurization was resumed.
5. A visual inspection of the IBC was performed for leaks throughout the test by coating any seams, joints, fittings and closures with a soap solution and observing for bubbles. Sensory methods of leak detection were also used by listening and feeling for leaks.

3.6 Hydraulic pressure test

The following sequence of steps was followed in performing a hydraulic pressure test of three IBCs from each of Manufacturer A, Manufacturer B and Manufacturer C.

1. The IBC closure was instrumented with a pressure transducer and a fitting for hydraulic pressurization.
2. Hot melt glue was applied to seal pressure relief valves in the vented closures to prevent venting.
3. A torque of 102 N-m (75 lbf-ft) was applied initially to the closures as per instructions found either on the enclosures or in the manufacturer's literature.
4. The IBC was filled and the maximum hydraulic pressure as marked on the IBC (100 kPa (15 psi) for all IBCs) was applied to the body of the IBC for 10 min.
5. In the event a leakage was detected, the main closures were further tightened to 136 N-m (100 lbf-ft) and pressurization was resumed.
6. A visual inspection of the IBC was performed for leaks throughout the test by inspecting for free flowing droplets at seams, joints, fittings and closures.

3.7 Drop test

For the drop test, two drop orientations were selected for testing. The standard requires that the point of impact is the part of the base that would cause the most damage to the IBC. This is typically assumed to be the corner, however, while corner drops may cause the most apparent damage, flat drops may result in greater shock loadings, thus both orientations were investigated in this study.

The drop height was determined to be 1.9 m from the standard based an assumed product specific gravity of 1.9, calculated from information within the UN marking and a test at packing group level II when the test is performed with water. As the cold drop tests were performed with a glycol water mix with a specific gravity of 1.1, the drop height was reduced to 1.72 m. Also as results were obtained and due to the limited number of samples available to be dropped, lower drop heights of 1.2 m and 0.8 m were included.

3.7.1 Flat drop test

The following sequence of steps was followed in performing flat drop tests of one IBC from Manufacturer C and one IBC from Manufacturer A, at ambient temperatures. This is contrary to the standard which specifies a temperature of -18°C; however, it was done to simplify test setup and acquire preliminary results.

1. The empty IBC was weighed to identify a tare mass. It was then filled with water to brim full capacity, reweighed, and then water was removed to achieve 98% of load capacity.
2. Hot melt glue was applied to seal pressure relief valves in the vented closures to prevent venting.
3. The IBC was confirmed to be level using a standard carpenter's level. The height was measured using a 1.9 m rod placed under each of the four corners in turn, and the IBC was released from the confirmed height.
4. A visual inspection of the IBC was performed and any permanent deformation of the structural equipment or release of the IBC contents was noted.

3.7.2 Corner drop test

The following sequence of steps was followed in performing corner drop tests of one of the Manufacturer C IBCs and one of the Manufacturer B IBCs at ambient temperatures. This is contrary to the standard which specifies a temperature of -18°C; however, it was done to simplify test setup and acquire preliminary results.

1. The empty IBC was weighed to identify a tare mass. It was then filled with water to brim full capacity, reweighed, and then water was removed to achieve 98% of load capacity.
2. Hot melt glue was applied to seal pressure relief valves in the vented closures to prevent venting.
3. Corner drops were completed by lifting the IBC from a single lift point such that the impact point would be on a corner adjacent to the bottom fittings. The 1.9 m distance was measured between the impact surface and the lowest point on the hanging IBC and the IBC was released from the confirmed height.
4. A visual inspection of the IBC was performed and any permanent deformation of the structural equipment or release of the IBC contents was noted.

3.7.3 Cold drop test

The test procedures described in Sections 3.7.1 and 3.7.2 were repeated with the IBCs cooled to -18°C as specified in the standard. Note that water was replaced with equal parts water/ethylene glycol mix for the cold drop tests.

3.8 Vibration test

The following sequence of steps was followed in performing a vibration test of one IBC from each of Manufacturer A, Manufacturer B and Manufacturer C.

1. The empty IBC was weighed to identify a tare mass. It was then filled with water to brim full capacity, reweighed, and then water was removed to achieve 98% of load capacity.
2. A torque of 102 N-m (75 lbf-ft) was applied initially to the closures as per instructions found either on the enclosures or in the manufacturer's literature.
3. The IBC was hoisted into position on a 1.5 m x 1.5 m steel support plate bolted to a single 178 kN hydraulic actuator providing vertical vibration. The lifting crane and vertical strapping was left in place as a safety precaution while the IBC was constrained from lateral motion by the installation of four aluminum angle retaining walls. A 30 g uniaxial accelerometer was installed on each IBC to provide acceleration response during the test.
4. A ±12.5 mm (±0.5 in.) displacement sine wave was applied at 1 Hz and gradually increased until accelerometer values in excess of 1 g were achieved.
5. The shim verification test was employed, whereby a flat shim was passed between the IBC and actuator plate, to demonstrate an unloading condition. The frequency and amplitude were adjusted until the shim could be passed between the IBC and support plate with at least two corners of the IBC leaving the test platform.
6. Once an unloading condition had been achieved, the vibration profile continued to be applied for 60 min.
7. A visual inspection of the IBC was performed for leaks throughout the test by inspecting for free flowing droplets at seams, joints, fittings and closures.

3.9 Materials properties test

In addition to the performance tests used to evaluate the observable physical condition of the IBCs, Fourier transformed infrared spectroscopy (FTIR), hardness testing, scanning electron microscopy (SEM) and differential scanning calorimetry (DSC) were used to evaluate the composition and condition of the inner bottles of the IBCs.

All material properties analyses were performed by the NRC-AST Polymer Bioproducts team located at Boucherville, QC. The test samples provided were taken from the inner bottles in locations showing significant visual degradation.

3.9.1 Fourier transformed infrared spectroscopy (FTIR)

In addition to allowing the identification of the various materials involved in the construction of the IBC, FTIR is used to detect possible chemical modifications of samples due to ultraviolet or chemical exposure.

Analyses were performed using a Thermo Scientific Nicolet iS50 bench mounted spectrometer with a PIKE diamond attenuated total reflectance (ATR) accessory. The bench is equipped with a Polaris infrared source, an automated Mickelson interferometer and deuterated triglycine sulfate (DTGS) detector. Spectra were acquired through 64 scans, from 4,000 and 525 cm^{-1}, at a resolution of 4 nm.

Acquisitions were performed on surfaces exposed to light and/or chemicals, and on protected surfaces for comparison purpose. Each spectrum was acquired at least twice, on different spots, to check the reproducibility. Analysis of the spectra was done using the Omnic and Omnic Specta software from Thermo Scientific.

Spectra were acquired for the following samples:

- Container plastic walls, internal part, external and internal surfaces;
- Black closure gasket (Manufacturer B), exposed and protected surfaces;
- Red closure gasket (Manufacturer A), exposed and protected surface; and
- 2-in. bung and gasket (Manufacturer C).

3.9.2 Hardness

Hardness measurement was performed on rubbery and elastomeric materials to quantify their elastomeric properties. The results should be compared to the required value for the application or the initial values from the material provider.

Analysis was performed using a Shore A2 calibrated equipment (with calibration verified every five measurements) mounted on a semi-automated measurement setup. 10 measurements were performed for each surface.

The analysed samples were:

- Black closure gasket (Manufacturer B) exposed surface;
- Black closure gasket (Manufacturer B) unexposed surface;
- Red closure gasket (Manufacturer A) exposed surface;
- Red closure gasket (Manufacturer A) unexposed surface; and
- 2-in. bung gasket (Manufacturer C).

3.9.3 Scanning electron microscopy (SEM)

SEM is used to detect micro degradation (cracks or disintegration) of surfaces, due to mechanical and environmental exposures (e.g., temperature, chemical, or ultraviolet).

Analyses were performed using a field emission gun (FEG) - SEM Hitachi system. The samples were prepared by cutting and embedding on a support, prior to platinum coating, with pictures acquired at magnifications of x50, x100, x250 and x500.

The analysed samples were:

- Container wall external surface;
- Black closure gasket (Manufacturer B) exposed surface;
- Red closure gasket (Manufacturer A) exposed surface;
- 2-in. bung damaged collar (Manufacturer C); and
- 2-in. bung gasket (Manufacturer C).

3.9.4 Differential scanning calorimetry (DSC)

DSC is used to measure the transition temperatures (glass transition temperature, melting temperature and crystallization temperature) of polymers and to determine the current thermal history of the samples.

Analyses were performed using a Q2000 thermal analysis system, and the data were analysed with the TA Universal Analysis software. The same systematic method was applied for each sample, with a first heating ramp from 25°C to 300°C at 10°C/min, an isothermal plateau for 5 min, a cooling ramp from 300°C to 25°C at 10°C/min, and a final heating ramp from 25°C to 300°C at 10°C/min.

Thermograms were acquired for the following samples:

- Container plastic walls, internal part, external and internal surfaces;
- Black closure gasket (Manufacturer A) exposed and protected surfaces;
- Red closure gasket (Manufacturer B) exposed and protected surface; and
- 2-in. bung surface and gasket (Manufacturer C).

4 Test results and analysis

4.1 Overview

IBCs produced by three manufacturers were subjected to different combinations of the specified tests, as shown in Table 5. IBCs were randomly selected from those that were made available to the NRC.

Test	Manufacturer and IBC identifier																								
	A							B							C										
	1	2	3	4	5	6	7	1	2	3	4	5	6	7	1	2	3	4	6	7	8	9	11	13	16
Bottom lift	T	T	T					T	T	T					T	T	T								
Top lift			T							T					T	T	T								
Stacking	T	T						T	T						T	T	T								
Leakproofness		T		T		T				T	T		T		T			T	T						
Hydraulic pressure		T		T		T				T	T		T		T			T	T						
Flat drop (ambient)				T														T							
Flat drop (cold)												T										T			
Corner drop (ambient)														T						T					
Corner drop (cold)					T																T		T	T	
Vibration							T		T																T
Materials				T			T		T	T											T				

Table 5: IBC samples tested (T)

4.2 Bottom lift test

A total of nine IBCs were subjected to the bottom lift test – three IBCs from each of Manufacturer A, Manufacturer B and Manufacturer C.

The weight of each IBC was measured and adjusted to the required 125% of maximum permissible gross weight as shown in Table 6.

IBC	Max permissible gross weight (kg)	Measured gross weight (kg)	Actual test weight (kg)
A1	2,242	2,242	2,803
A2	2,242	2,243	2,804
A3	2,242	2,244	2,805
B1	2,242	2,244	2,805
B2	2,242	2,243	2,804
B3	2,242	2,242	2,803
C1	2,510	2,513	3,141
C2	2,510	2,512	3,140
C3	2,510	2,513	3,141

Table 6: Measured weights of IBCs for bottom lift test

After the tests the IBCs were visually inspected, with none of the IBCs exhibiting any sign of permanent structural deformation or release of the IBC contents.

4.3 Top lift test

A total of five IBCs were subjected to the top lift test – one from each of Manufacturer A and Manufacturer B, and three from Manufacturer C.

A mass equivalent to the maximum permissible gross mass was assembled and hung with chains off the top of the IBC using two I-beams, chains and a plywood base.

The configuration of the lifting assembly, with a 45° angle check, and additional mass added for the 45° top lift of Manufacturer C IBCs. The configuration of the additional mass was unchanged from the straight top lift test.

The weight of each IBC was measured and adjusted to the required 200% of maximum permissible gross weight as shown in Table 7.

IBC	Target weight (kg)	Measured weight (kg)
C1	5,010	5,080
C2	5,010	5,105
C3	5,010	5,097

Table 7: Measured weights of Manufacturer C IBCs for top lift test

After the top lift tests the Manufacturer C IBCs were visually inspected, with none of the three IBCs exhibiting any sign of permanent deformation or release of the IBC contents after being subjected to both straight and 45° lifts.

The configuration of the additional mass added for the top lift as well as the lifting assembly configuration for the 45° top lift of Manufacturer A and Manufacturer B IBCs was similar to that for the Manufacturer C IBCs. A mass equivalent to the maximum permissible gross mass was assembled and hung with chains off the top of the IBC using two I-beams, chains and a plywood base.

The weight of each IBC was measured and adjusted to the required 200% of maximum permissible gross weight as shown in Table 8.

IBC	Target weight (kg)	Measured weight (kg)
A3	4,486	4,637
B3	4,486	4,564

Table 8: Measured weights of Manufacturer A and Manufacturer B IBCs for top lift test

After the straight top lift tests the Manufacturer A and Manufacturer B IBCs were visually inspected, with neither of the two IBCs exhibiting any sign of permanent deformation or release of the IBC contents. However, after the 45° top lift tests the Manufacturer A and Manufacturer B IBCs were visually inspected, with both of the IBCs experiencing a failure of one of the lifting eyes during one of the two lifts. Subsequently, due to a shortage of metal frames, further top lift tests were suspended to preserve IBCs for subsequent testing.

It was noted that the orientation of the shackles used to lift the IBCs was potentially a contributing factor to the damage of the lifting eyes. In each of the two 45° top lift tests performed on IBCs A3 and B3, where the pin of the shackle was oriented upwards, an eye failed. In each of the two 45° top lift tests performed on IBCs A3 and B3 where the pin of the shackle engaged with the lifting eye of the IBC, the eye bent slightly inwards but did not pull out. In such cases the deformation was such that another IBC would likely have been able to be nested during stacking. This would likely be considered as a pass of the test, although any permanent deformation could also be considered a test failure depending upon the degree of damage. This failure mode of the steel frames was unexpected and may require further study.

No damage to the inner bottles was noted and no release of contents was observed. Damage to the frames in the test cases where a lifting eye failed completely was to such an extent to declare a failure on the basis that such severe deformation would prevent proper nesting of stacked IBCs, as well as the loss of the lifting location.

4.4 Stacking test

A total of seven IBCs were subjected to the stacking test – two IBCs from each of Manufacturer A and Manufacturer B, and three IBCs from Manufacturer C.

The maximum number of fully loaded IBCs allowed for stacking was one. A reproduction of the base of the IBC was manufactured by partially disassembling a separate identical frame. A concrete mixture was poured into the concave bottom section, lined with a section of tarp. Steel boxes containing lead as well as rubber mats were added to this resulting flat base to achieve a target mass of 1.8 times the mass that will be supported by the IBC during transport, equivalent to 4,518 kg.

After the stacking tests the Manufacturer C IBCs were visually inspected, with none of the three IBCs exhibiting any sign of permanent deformation or release of the IBC contents. Plastic frames deflected an average of 5 mm with a maximum deflection under load of 10 mm. Based on the criteria specified in the standard, the stacking tests on Manufacturer C IBCs are deemed a pass.

The maximum number of fully loaded IBCs allowed for stacking was two. A load spreading base of laminated plywood sheets was cut to size such that it fit within the top load bearing area, and of a sufficient height as to not interfere with the top lifting eyes. A large steel plate, nominally 6.4 cm (2.5 in.) thick, with lifting eye bolts in each corner suitable to lift the test load safely, was used as the load carrier. Steel boxes containing lead as well as rubber mats were loaded onto the steel plate to achieve a target mass of 8,860 kg, as specified on the nameplate as the maximum stacking load. This is approximately 10% greater than 1.8 times the mass of similar IBCs that would be stacked.

After the stacking tests the Manufacturer A and Manufacturer B IBCs were visually inspected, with none of the four IBCs exhibiting any sign of permanent deformation or release of the IBC contents. Steel IBC frames showed no significant deformation or damage. Deflection measurements were on the order of 1.6 mm maximum, which is within the potential for measurement errors. Based on the criteria in the standard, the stacking tests on Manufacturer A and B IBCs are deemed a pass.

4.5 Leakproofness test

4.5.1 Initial leakproofness test

A total of nine IBCs were subjected to the leakproofness test – three IBCs from each of Manufacturer A, Manufacturer B and Manufacturer C.

Within each set of three IBCs from each manufacturer, two IBCs had the air fitting and pressure transducer installed in the closure while in the third IBCs they were installed on the top face of the container.

To seal pressure relief valves in the vented closures and prevent venting, hot melt glue was applied. All three IBCs from Manufacturer C did not incorporate pressure relief valves into the closures, while IBCs A2 and B3 had pressure relief fittings which had been glued prior to the start of testing.

Maximum pressures and pass/fail results observed in the leakproofness tests are shown in Figure 1. Detailed results from the leakproofness tests are given in Table 9. Initial leakage pressures and location are specified in addition to any subsequent leakage and location after action was taken to remedy the initial leakage, where applicable.

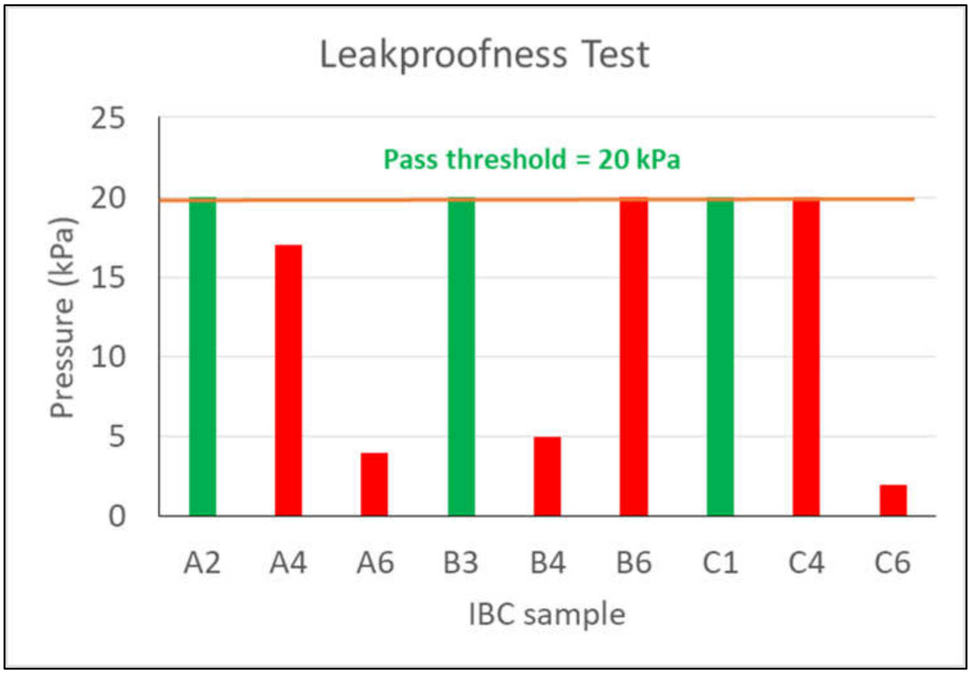

Figure 1: Initial results of leakproofness test prior to any modification

IBC	Initial leakage		Subsequent leakage		Comment	Pass / fail
	Pressure (kPa)	Location	Pressure (kPa)	Location		
A2	None detected	NA	None detected.	NA	No leakage observed at 20 kPa.	Pass
A4	17	Relief valve	None detected after closure replacement and hot melt.	NA	Leakage past base of relief valve at approximately 17 kPa. Repeated air test with a spare closure, leakage at approximately 5 kPa at the relief valve threads. The valve was hot melted and no leakage was observed at 20 kPa.	Pass following modification
A6	4	Relief valve	20	Closure	Leakage past the relief valve at 4 kPa. Hot melted the relief valve parts and repeated air test, slow leakage heard past the closure at 20 kPa.	Fail
B3	None detected	NA	None detected.	NA	No leakage observed.	Pass

IBC	Initial leakage		Subsequent leakage		Comment	Pass / fail
	Pressure (kPa)	Location	Pressure (kPa)	Location		
B4	5	No gasket present	17	Base of pressure relief, then leakage past main closure.	No gasket was present in closure, leakage occurred at 5 kPa. Closure was replaced with a spare and test repeated, with leakage past the pressure relief base at less than 17 kPa. The valve was hot melted and leakage was observed past the main closure. Closure re-torqued to 136 N-m and leakage continued.	Fail
B6	20	Relief valve	None detected.	NA	Leaking past relief valve at 20 kPa. Valve was hot melted and no leakage observed at 20 kPa.	Pass following modification
C1	None detected	NA	None detected	NA	No leakage observed.	Pass
C4	20	2-in. closure and fitting	None detected.	NA	Leakage at 20 kPa through the 2-in. bung closure and at the threads to the insert for the closure. Gasket for the insert was pinched and damaged. Parts were replaced and test repeated with no leakage observed.	Pass following modification
C6	2	Both 2-in. closures	20 kPa	Main 6-in. closure.	Leakage past two 2-in. closures at 2 kPa. Evidence of chemical leakage on bungs was observed. Replaced with spare bungs and test continued, with leakage at pressure past closure. Re-torqued the closure to 136 N-m and leak still audible at closure at 20 kPa.	Fail

Table 9: Results and observations from leakproofness testing

Leakage was assessed by spraying the closure and various other fittings with a soap solution. Due to the large threads on the closures of the Manufacturer A and Manufacturer B IBCs, it is not always possible to check the main closure for leakage using this method as the threads are quite large and it is difficult to look under the closure itself for evidence of bubbles. Sensory methods of leak detection were also used by listening and feeling for leaks.

It was also noted that a slow reduction in pressure with the air supply shut off did not appear to be a reliable leak detection option as the containers relax slightly during the test. In one case the test duration was extended and the relaxation mostly subsided, indicating no leak was present. It was also noted that larger leaks were sometimes missed using the soapy water method, as the air simply blows out the soap film.

The majority of IBCs tested showed at least some air leakage in an unmodified state. The first set tested, IBCs A2 and B3, had already had pressure relief fittings sealed with hot melt glue at the start of testing. The remaining IBCs did not have pressure relief fittings sealed. All of the air leakage observed past relief valves was below the rating for the valves, which was 34.5 kPa (5 psi) as per markings on the valves. Pressure relief valves leaked both at the threads to the closure as well as directly through the valve itself in some instances. The ability to seal the valves with hot melt varied as threads stood proud of the interior cap surface on orange closures but were recessed from the surface on black closures.

The Manufacturer C IBCs had no relief valves installed and it was observed that four of the six 2-in. bungs installed leaked directly through the closure material. Three of those four may have been over torqued at some point as cracks appeared at the top of the closure, while all appeared to be quite worn and weathered upon visual inspection. Two of the bungs on IBC C6 appeared to have chemical residues on their exterior, implying that leakage may have occurred while they were in service.

There were no instances of leakage at the bottom discharge valves of any of the IBCs.

4.5.2 Repeat leakproofness test

One requirement in industry is to replace gaskets in closures if they are found to be leaking. Using this approach, a set of new gaskets was ordered so that samples which had failed could be retested with new gaskets installed. Installation instructions were not provided, thus gaskets were first installed “dry”, tested, and then if leaks were observed the gaskets were lubricated with a light coating of WD40. As some IBCs were consumed in other tests, only six remained intact for the repeat leakproofness test.

Maximum pressures and pass/fail results observed in the repeat leakproofness tests are shown in Figure 2. All six IBCs tested were ultimately able to pass the leakproofness test, with only one of the six requiring additional torque on the closure to do so.

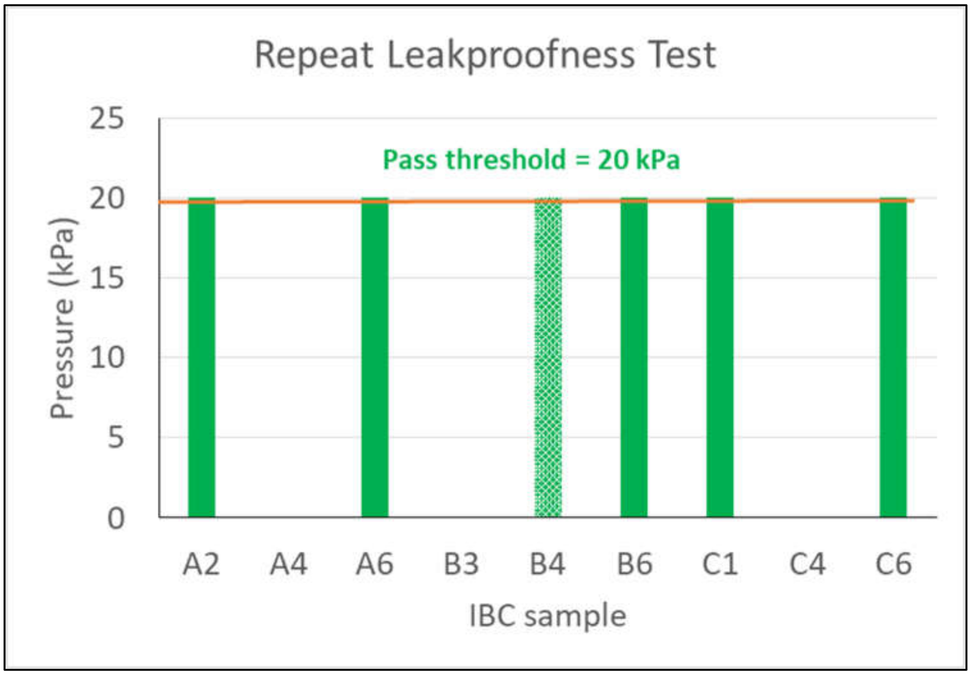

Figure 2: Results of repeat leakproofness test

IBCs A2 and A6 passed the leakproofness test with new gaskets installed on the closures with 102 N-m (75 lbf-ft) of torque.

IBC B4 leaked with the closure torqued to 102 N-m (75 lbf-ft), but it passed with a new gasket and the closure torqued to 136 N-m (100 lbf-ft).

IBC B6 passed the leakproofness test with a new gasket installed and the closure torqued to 102 N-m (75 lbf-ft) torque.

IBC C1 passed the leakproofness test with a new gasket installed, at 102 N m (75 lbf-ft) torque.

IBC C6 leaked at full pressure with a new but “dry” gasket. The gasket was lubricated and a pass result was obtained at 102 N m (75 lbf-ft) of torque.

4.6 Hydraulic pressure test

4.6.1 Initial hydraulic pressure test

A total of nine IBCs were subjected to the hydraulic pressure test – three IBCs from each of Manufacturer A, Manufacturer B and Manufacturer C.

The hydraulic pressure test was performed following the leakproofness test on the same IBCs. Within each set of three IBCs from each manufacturer, two IBCs had the air fitting and pressure transducer installed in the closure while in the third IBC they were installed on the top face of the container. Maximum pressures and pass/fail results observed in the hydraulic pressure test are shown in Figure 3. Detailed results from the hydraulic pressure tests are summarized in Table 10.

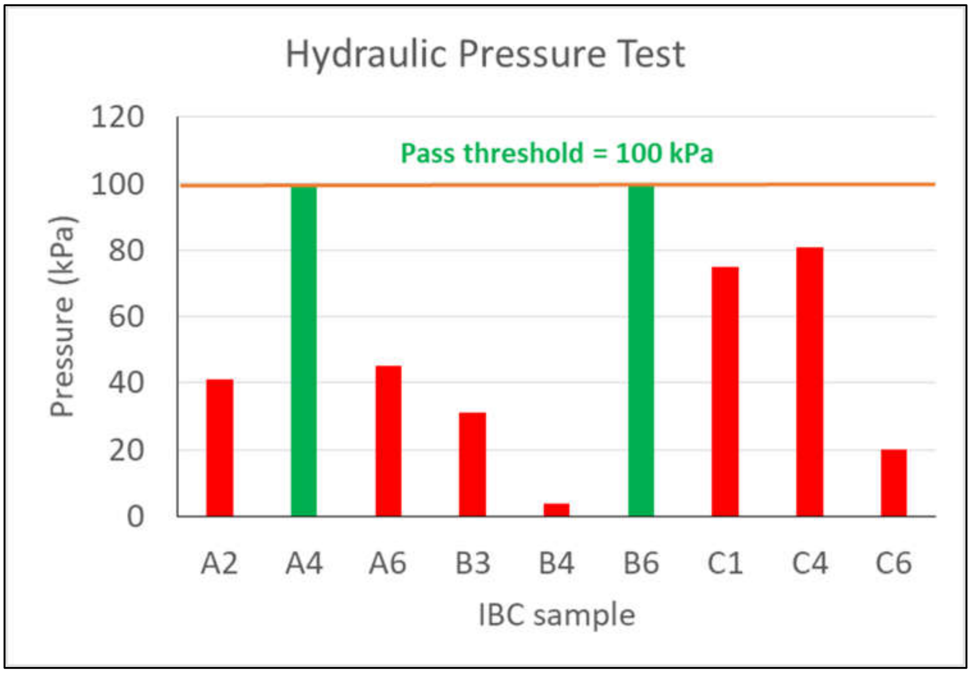

Figure 3: Hydraulic pressure test initial leakage pressures and pass/fail results observed

IBC	**Initial leakage**		**Subsequent leakage**		**Comment**	**Pass / fail**
	Pressure (kPa)	**Location**	**Pressure (kPa)**	**Location**		
A2	41	Closure	34	Closure	Leakage past the closure observed at approximately 41 kPa. Closure tightening to 136 N-m resulted in leakage at 34 kPa.	Fail
A4	None detected	NA	None detected	NA	No leakage at 100 kPa. Test was extended to observe pressure stability. Note that quick pressure drops were observed during the testing. It is believed this is due to the frames suddenly relaxing or moving such that the walls expand and reduce pressure. No leaks were found.	Pass
A6	45	Closure	45	Closure	Leakage past the closure observed at approximately 45 kPa. Closure tightening to 136 N-m did not stop the leak.	Fail

IBC	Initial leakage		Subsequent leakage		Comment	Pass / fail
	Pressure (kPa)	Location	Pressure (kPa)	Location		
B3	31	Closure	65	Closure	Leakage past the closure observed at approximately 31 kPa. Closure tightening to 136 N-m stopped leakage temporarily, with a resumption at 65 kPa.	Fail
B4	4	Closure	4	Closure	Leakage past the closure observed at approximately 4 kPa. Closure tightening to 136 N-m did not stop the leak. Close observation of the closure area on the container showed a small deformation that likely led to the quite low result obtained.	Fail
B6	None detected	NA	None detected	NA	No leakage observed.	Pass
C1	62	Closure	76	Closure	Leakage past the closure observed at approximately 62 kPa. Closure tightening to 136 N-m stopped leakage temporarily, with a resumption at 76 kPa	Fail
C4	Trapped air <81	2-in. bung	81	Closure	Air leakage at bung discovered, replaced part with spare and continued test. Leakages past the closure observed at approximately 81 kPa.	Fail
C6	20	Closure	20	Closure	Air leakage past the closure observed at approximately 20 kPa. Closure tightening to 136 N-m did not stop the leak.	Fail

Table 10: Results and observations from hydraulic pressure testing

It was noted that some of the Manufacturer A and Manufacturer B IBCs had the closure area lower than the container edges thus even brimful filling allowed some air to remain within. As water pressure was applied, the containers deformed upwards allowing that trapped air to move into the closure area. Thus determination of leak pressures was somewhat more difficult as the trapped air would tend to leak first followed by water. The Manufacturer C IBCs with a domed top did not suffer this same phenomenon.

Manufacturer A and Manufacturer B IBCs did not arrive with steel frames, thus a set of two frames was used for all the hydraulic testing. This was done in an effort to keep some frames in an undamaged state for the planned drop tests. Hydraulic testing was observed to cause permanent deformation of the side walls of the steel frames. Since the steel frames are not the main focus of this study this was deemed an acceptable practice for this test.

Only two of nine IBCs passed the hydraulic tests: IBCs A4 and B6. Deformation was observed on one of the Manufacturer A inner bottles from the pressure application. Leakage observed was typically at the closures.

Hydraulic pressure failures were generally at or above the pressures of any failures observed with the leakproofness test, as expected. Pressure relief valves, where present, were rendered inoperable for all hydraulic tests, as their rating was only for 34.5 kPa (5 psi).

The last set of three IBCs (one from each manufacturer) tested with the closures left in original condition performed essentially the same as those which were tested with pressure fittings mounted in the closure. One of each arrangement passed the test, indicating that if properly installed, fittings through the closure are unlikely to invalidate the test results. IBC C6, with an unadulterated closure and no obvious defect on the sealing surface, leaked at 20 kPa, the second lowest pressure among the nine IBCs tested.

There was not an obvious pattern to the failure pressures, although tightening the closures did in general seem to improve the results. The average initial failure pressure was 55 kPa (8.0 psi). In no instances were failures seen at the bottom discharge valve.

4.6.2 Repeat hydraulic pressure test

The hydraulic pressure tests were repeated on six of the IBCs that failed the initial hydraulic pressure test, with closures that had new gaskets installed. Maximum pressures and pass/fail results observed in the hydraulic pressure test are shown in Figure 4. Detailed results from the hydraulic pressure tests are summarized in Table 11.

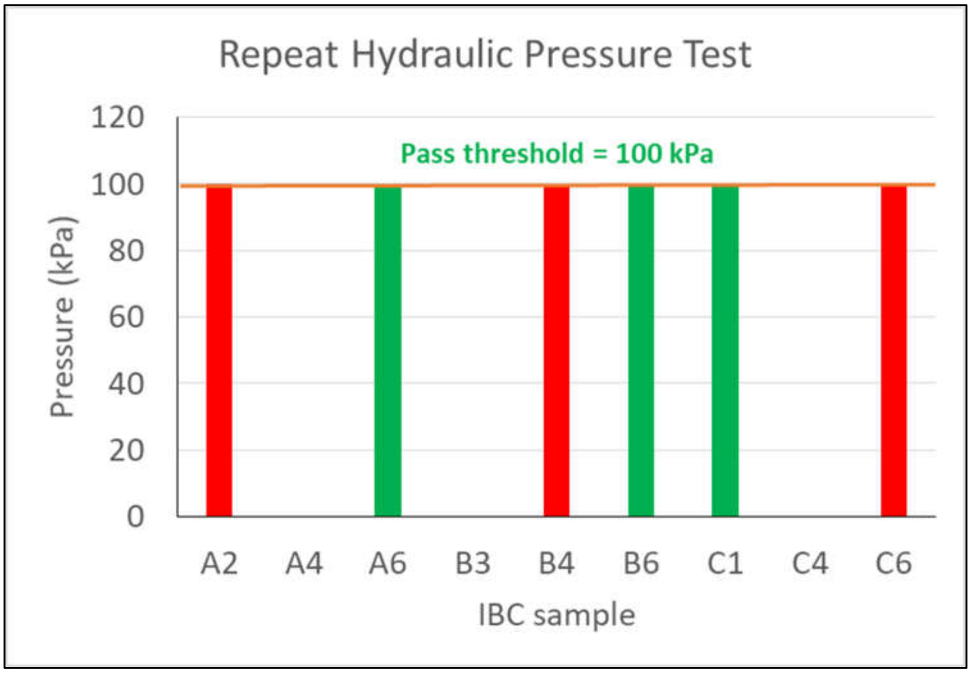

Figure 4: Results of repeat hydraulic pressure test

IBC	Original pass / fail	Comment	Repeat pass / fail
A2	Fail	Leak at bottom closure, handle opened	Fail
A6	Fail	No leakage with new gasket	Pass
B4	Fail	Leak through body of tank	Fail
B6	Pass	Initially failed with dry gasket	Pass
C1	Fail	No leakage with new gasket	Pass
C6	Fail	Leak past seal	Fail

Table 11: Results and observations from repeat hydraulic pressure testing

IBC A2 failed at full pressure with a new gasket applied but not lubricated. With lubrication IBC A2 failed due to bulging in the recessed area of the bottom discharge valve. The bulge forced the handle to open and the locking mechanism was damaged in the process.

IBC A6 passed the test.

IBC B4 developed a leak at the cap when it was torqued to 102 N-m (75 lbf-ft). With 136 N-m (100 lbf-ft) of torque applied to the closure, a leak developed through the body of the tank.

IBC B6 was fully pressurized and a small leak was visible at the main closure. The gasket was then lubricated, the test was repeated and a pass result was obtained.

IBC C1 leaked with a dry gasket. The gasket was then lubricated, the test was repeated and a pass result was obtained.

IBC C6 leaked at 7.5 psi with a dry gasket. The gasket was then lubricated, the test was repeated, and leakage past the gasket was observed at full pressure with a lubricated gasket.

4.7 Drop test

Two phases of drop testing were performed, the first at ambient temperature outdoors on 2021-03-12 and the second at -18°C in the climatic facility between 2021-06-25 and 2021-06-29. A total of four IBCs were subjected to the drop test at ambient temperature: one Manufacturer A and one Manufacturer C IBC were subjected to the flat drop test, and one Manufacturer B and one Manufacturer C IBC were subjected to the corner drop test. A total of six IBCs were subjected to the drop test at a temperature of -18°C: one Manufacturer B and one Manufacturer C IBC were subjected to the flat drop test, and one Manufacturer A and three Manufacturer C IBCs were subjected to the corner drop test.

It was decided, in consultation with TC staff, to complete a preliminary drop test at ambient temperatures rather than the -18°C temperature specified in the standard. This had the advantage of simplifying the test set up, as water can be used to fill the IBCs, and the drops can be completed on the NRC’s outdoor test pad rather than inside the climatic facility.

The drop test at ambient temperatures is considered less severe than a cold drop test at a temperature of -18°C. Cold temperatures are expected to result in embrittlement of plastic components, making them more fragile and susceptible to cracking under shock loading. Despite this, any failure of an IBC at ambient temperature should still be considered a completely valid test result. A pass on the other hand would be suspect, as the IBC was not as severely tested.

For the composite IBCs available for this test, used metallic frames that had undergone the air leakage and hydraulic pressure tests were utilized. These frames had been deformed by these tests, thus it can be argued that the test results are not necessarily representative of how a new frame would react. As only a limited number of metal frames could be obtained for the study, frames were reused in some cases to preserve those in good condition to be used in later tests.

The IBCs were weighed empty to get a tare mass, then filled brim full with water and reweighed. Water was then removed until 98% of the brim full capacity remained. The IBC's closures were modified by the addition of hot melt glue to any working parts of pressure relief valves, and closures were applied to the IBCs with 102 N-m (75 lbf-ft) of torque.

The ambient temperature drop test was completed outdoors on the NRC drop pad. The pad measures approximately 5 m by 14 m by approximately 0.5 m thick and is constructed of reinforced concrete. The pad is level and flat. IBCs were taken outside for the test at approximately 09:00 a.m. on a day with temperatures rising to 6°C. Testing was completed by 11:30 a.m., thus the final IBC temperatures were expected to be quite close to their original 20°C when removed from the climatic facility.

Flat drops were achieved by the use of a four-leg chain lifting sling apparatus. The hanging IBC was confirmed to be level using a standard carpenter's level. Shackles were added to each of the four lifting points on the IBCs so that the lift hooks could be easily inserted. The NRC drop hook was attached to the crane used to lift the IBCs with a large shackle. The drop height was determined to be 1.9 m from the standard, based on a product specific gravity of 1.9 and a test at packing group level II. The height was measured using a 1.9 m rod placed under each of the four corners in turn.

Cold drop tests were completed in the climatic facility and completed over a three day period. IBCs were filled with a 50/50 mix of water and ethylene glycol to prevent freezing at the test temperature of -18°C. The climatic facility floor consists of a two foot thick concrete slab, heavily reinforced with steel rebar. A temporary berm was set up to contain any spilled glycol in the event of failures, with an A-frame gantry crane placed in the berm area to allow lifting of the IBCs to the desired drop height. A quick release drop hook was again used to enable drops in either a flat or corner drop orientation. Fill level was determined by weighing the IBCs.

The initial drop height for the cold drop was determined to be 1.72 m from the standard, based on a specific gravity of 1.9 and a test at packing group level II, and correcting for a fill fluid specific gravity of 1.1 instead of 1.0. The height was measured using either a fixed rod or tape measure, set to the appropriate height, placed under each of the four corners of the IBC in turn.

Corner drops were completed by lifting the IBC from a single lift point such that the impact point would be on a corner adjacent to the bottom fittings. The appropriate distance was measured between the impact surface and the lowest point on the hanging IBC.

Detailed results for each of the drop tests, at ambient temperature and at a temperature of -18°C, are presented in Sections 4.7.1 to 4.7.9. Discussion of the results is provided in Section 4.7.11.

4.7.1 IBC A4 flat drop test at ambient temperature

Damage incurred by IBC A4 as a result of being subjected to a flat drop from 1.9 m included:

1. Severe deformation of the cushion under the inner bottle;
2. Cracking of the bottom steel frame at two weld points;
3. Further bulging of the side walls of the steel frame. These had bulged from previous hydraulic tests prior drop testing; and
4. Scraping of plastic material on side walls.

No release of contents was observed.

4.7.2 IBC C4 flat drop test at ambient temperature

Damage incurred by IBC C4 included cracking of the base across two adjacent corners with no release of contents observed as a result of being subjected to a flat drop from 1.9 m.

4.7.3 IBC B7 corner drop test at ambient temperature

Damage incurred by IBC B7 as a result of being subjected to a corner drop from 1.9 m included:

1. Crushing of the impacted corner;
2. Racking of the steel frame; and
3. Minor scraping of plastic material on side walls.

No release of contents was observed when the IBC came to rest upright. When intentionally set horizontal after the drop test, leakage was observed past the pressure relief valve.

4.7.4 IBC C7 corner drop test at ambient temperature

Damage incurred by IBC C7 as a result of being subjected to a corner drop from 1.9 m included:

1. Severe cracking of impacted corner of plastic frame; and
2. Severe cracking through to adjacent corner of frame (not adjacent bottom fittings).

Leakage past the closure was observed in the form of free flowing droplets as the IBC came to rest horizontally.

IBC C13 corner drop test at -18°C temperature

Damage incurred by IBC C13 as a result of being subjected to a corner drop test from 1.72 m included:

1. Severe cracking and damage to plastic frame;
2. Inner bottle severely cracked into multiple pieces; and
3. Immediate and complete leakage of the IBC contents.

4.7.5 IBC C8 corner drop test at -18°C temperature

Damage incurred by IBC C8 as a result of being subjected to a corner drop test from 1.2 m included:

1. Impacted corner folded inward on plastic frame;
2. Inner bottle severely cracked into multiple pieces; and

3. Immediate and complete leakage of the IBC contents.

4.7.6 IBC C9 flat drop test at -18°C temperature

Damage incurred by IBC C9 as a result of being subjected to a flat drop test from 0.8 m included:

1. Severe cracking and damage to plastic frame;
2. Inner bottle severely cracked into multiple pieces; and
3. Immediate and complete leakage of the IBC contents.

4.7.7 IBC C11 corner drop test at -18°C temperature

Damage incurred by IBC C11 as a result of being subjected to a corner drop test from 1.72 m included:

1. Severe cracking and damage to plastic frame;
2. Inner bottle severely cracked into multiple pieces; and
3. Immediate and complete leakage of the IBC contents.

4.7.8 IBC A5 corner drop test at -18°C temperature

Damage incurred by IBC A5 as a result of being subjected to a corner drop test from 0.8 m included:

1. Minor damage to steel frame; and
2. No leakage of IBC contents was observed.

Damage incurred by IBC A5 as a result of being subjected to a second corner drop, this time from 1.2 m (on the other undamaged corner adjacent to the closure) included:

1. Minor damage to steel frame;
2. Inner bottle severely cracked into multiple pieces; and
3. Immediate and complete leakage of the IBC contents.

4.7.9 IBC B5 flat drop test at -18°C temperature

Damage incurred by IBC B5 as a result of being subjected to a flat drop test from 0.8 m, included;

1. No visible damage to frame;
2. Crushing and cracking of black pillow pad; and
3. No leakage of IBC contents was observed.

4.7.10 Drop test discussion

Only one of the IBCs leaked contents immediately following the ambient temperature drop test. When IBC C7 was dropped onto its corner, it came to rest on its side and a continuous flow of droplets was observed past the main closure. This leakage did not appear to be related to the impact itself, and earlier hydraulic tests revealed that the closures may leak at quite low pressures. However, any continuous leakage after a drop test is considered a failed result. Following a rest period over a weekend, IBC B7, which was also subjected to the corner drop test but had come to rest upright, was tipped onto its side and it also leaked past the threads of the relief valve. Again, there was no apparent damage from the drop, and the authors suspect this is a separate issue related to closures and relief valves.

There was some discussion around the topic of pass-fail criteria at the time of the drops. Damage to the corner dropped IBC B7 metal frame was significant, but it is unknown if the IBC could be shipped in this condition. Witness opinions varied, but those present generally agreed that it would be unwise to continue shipping after such an event. Rather, the consensus was that the IBC would be set aside and its contents transferred to another IBC.

Similarly, the plastic IBC that was corner dropped experienced significant damage to the plastic outer frame, and there was some concern the inner bottle might slip free of its frame when it was being lifted. The crane operator was instructed to keep the IBC close to the ground to prevent a second unintentional drop. This indicates that the IBC is not suitable for further shipment and thus failed the drop test, as part of the criteria for a successful drop test, other than no loss of contents, is that there is no damage which renders the IBC unsafe to be transported.

All four tested IBCs were moved by fork truck approximately 300 m after the testing was complete, without incident.

IBCs that were flat dropped appeared to be in overall better condition than those which were corner dropped. There was damage to both the steel and plastic frames. Damage to the steel was not obvious until the frame was tilted so the bottom could be observed. This was done in an effort to see the location of liquid dripping onto the ground under the IBC. It appeared that the liquid observed was water contained within the steel frame itself and not a leak from the inner bottle. The leaked water was blackish in color suggesting it did not originate from inside the inner bottle, and there was no clear visible path from the inner bottle. It should be noted the same frames were previously used for hydraulic leak testing.

One possible method to determine if the IBCs are shippable after dropping would be to conduct a vibration test on them. The concern would be that broken frame members might rub against the inner bottle material causing it to wear thin or puncture, resulting in product loss. This could be quite valuable research, as the damage to the corner dropped steel frame did not appear unusual relative to other corner drops that have been conducted in the past. The extent of the damage was severe due to the high drop height. This may prove to be a difficult task as the IBCs are no longer symmetrical and this may induce significant lateral motion on the vibration platform.

4.8 Vibration test

A total of three IBCs were subjected to the vibration test – one from each manufacturer – IBCs A7, B2 and C16. All three IBCs were visually inspected to ensure they were free of defects that could affect the results. Note that IBC B2 had been previously subjected to the bottom lift and stacking tests.

Accelerometers were installed on either the metal or plastic IBC frames and each IBC was weighed empty, and then filled with water. The weight when filled with water was recorded and used to determine the 98% filled weight required for test performance as shown in Table 12. A torque of 102 N-m (75 lbf-ft) was then applied to the closures as per instructions found either on the closure or the manufacturer's literature, where available.

IBC	Empty IBC weight (kg)	Full IBC weight (kg)	98% of full IBC weight (kg)
A7	256	1,520	1,490
B2	255	1,493	1,463
C16	151	1,461	1,432

Table 12: Measured weights of IBCs for vibration test

The IBC was hoisted into place on the single 178 kN actuator, and restraints were installed to prevent the IBC from moving laterally on the steel plate as the sine wave displacement was applied. Restraints were installed in such a way as to allow access with the shim tool to confirm IBC unloading had been achieved.

A ±12.5 mm (±0.5-in.) displacement sine wave was applied at 1 Hz and the frequency was gradually increased until accelerometer values in excess of 1 g were achieved, at which point the shim verification test was employed. A flat shim was passed between the IBC and actuator plate, on two sides, to demonstrate an unloading condition. The frequency and amplitude were adjusted until the shim could be passed between the IBC and support plate with at least two corners of the IBC leaving the test platform.

Once an unloading condition had been achieved, the vibration profile continued to be applied for a further 60 min. In reviewing the acceleration data, it was evident that it could be used as an indicator as to when an unloading condition for the IBC had been achieved. For illustrative purposes and without reference to the actual numbers, Figure 5 shows the raw acceleration data (top) and the same data filtered with a low pass filter up to 20 Hz (bottom) collected from a trial run using IBC A7.

There are clearly three distinct sections for the acceleration data. Section 1 is a smooth sine wave that increases in acceleration amplitude as the frequency increases to achieve an acceleration value above 1 g. Section 2 shows acceleration values above 1 g where the IBC is hopping in an erratic fashion. It was not possible to pass the shim under the IBC in a continuous manner, around the IBC perimeter. Section 3 shows a consistent, large amplitude acceleration, at which point the large shim could be easily passed under the full perimeter of the IBC. This was the chosen acceleration amplitude for the test run of 60 min. The acceleration amplitudes recorded are high due to the impacts that occur as the IBC contacts the support plate after lifting off. The same result was observed for the Manufacturer B and Manufacturer C IBCs.

At the selected acceleration amplitude, the top face of Manufacturer A and Manufacturer B IBCs displayed resonant frequency behaviour where the top face was visibly deflecting up and down at the centre while the side walls were bowing inwards and outwards at 180° out of phase with the top face, sometimes referred to as “oil canning”. The Manufacturer C IBC did not display any observable resonant frequency deformation during the test.

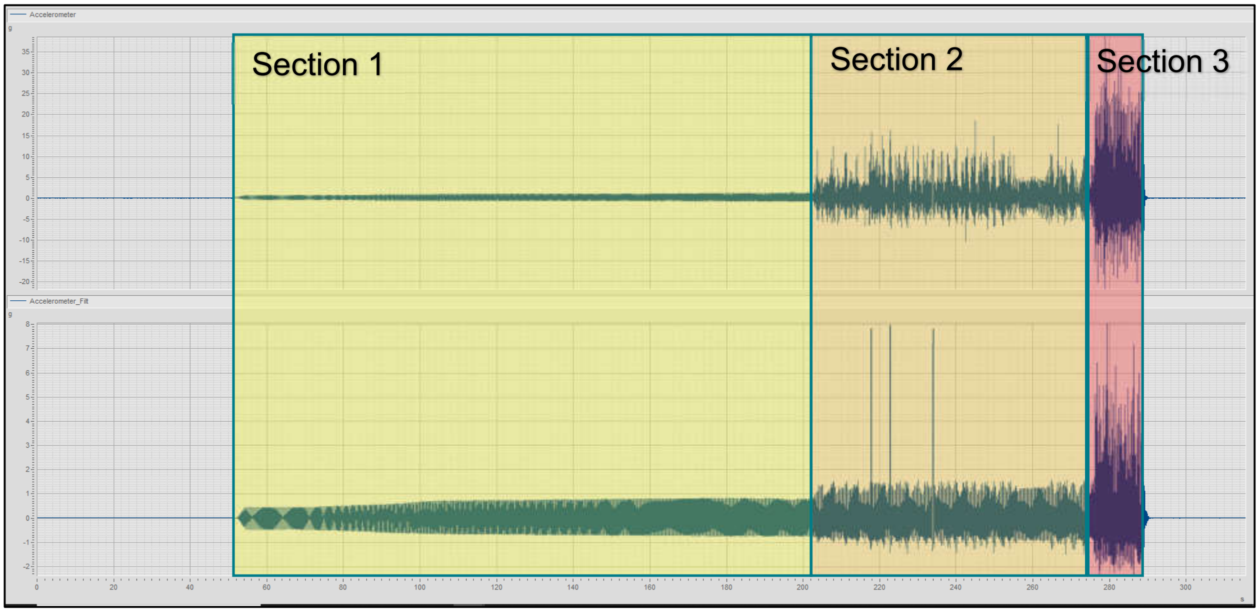

Figure 5: Trial run raw (top) and filtered (bottom) acceleration data

4.8.1 IBC A7 vibration

IBC A7 was subjected to the 60 min vibration exposure as described in Section 4.7.10 on 2021-09-28 with the following observations:

- After approximately 6.8 min, water leakage was observed through the closure of the IBC. The closure was removed, the gasket was replaced and the closure was re-torqued to the specification;
- Testing was resumed, and after a total of 9.4 min water leakage was again observed through the cap at which point the cap was removed, the pressure release valve was sealed with hot melt glue, and the closure was re-torqued to the specification;
- Testing was resumed, and after a total of 29 min a midpoint check found no leaks; and
- After a total of 42 min of vibration exposure, water leakage was observed and a crack was identified.

Upon further inspection, the failure was located at the outer lower corner of the indent on the long side opposite the drain valve in addition to crack initiations observed on the opposite side in the same location.

4.8.2 IBC B2 vibration

IBC B2 was subjected to the 60 min vibration exposure as described in Section 4.7.10 on 2021-09-29 with the following observations:

- After approximately 30 min a midpoint inspection found no leaks; and
- After a total of 62 min of vibration exposure, testing was terminated with no observed leaks.

Upon inspection, a crack initiation was observed in the identical location where IBC A7 had failed, though in this instance there was no observed water leakage. The outer metal frame also suffered damage, with multiple broken welds between the frame and side plates.

4.8.3 IBC C16 vibration

IBC C16 was subjected to the 60 min vibration exposure as described in Section 4.7.10 on 2021-10-01 with the following observations:

- After approximately 33 min a midpoint inspection found no leaks; and
- After a total of 66 min of vibration exposure, testing was terminated with no observed leaks.

Upon inspection, there was no observed damage to either the IBC frame or vessel.

4.9 Materials properties test

4.9.1 Fourier transformed infrared spectroscopy

According to the absorption bands attribution shown in Figure 6 and data base comparison, the sample container walls are made of low molecular weight polyethylene (PE). The spectra taken in the core of the wall show absolutely no trace of oxidation, with only absorption bands corresponding to the C-H bonds, characteristics of the PE backbone.

Internal and external surfaces, in comparison, show strong oxidation. This oxidation can be seen by the presence of O-H (hydroxyls) bands at 3,400 cm^{-1}, C-O (ethers) bands at 1,100 cm^{-1}, and C-O (esters and ketones) bands at 1,700 cm^{-1}. The shoulder on the left of the 2,900 cm^{-1} C-H band is the sign of a loss of symmetry in the backbone, proof of oxygen grafting.

Both surfaces were exposed to strong oxidation, probably due to exposure to water or stronger oxidizing agents for the internal surface, and ultraviolet and water exposure on the external surface. Such a level of oxidation is not surprising, nor dangerous, considering the thickness of the walls. Oxidized PE will lose mechanical properties and eventually start to degrade slightly, but as long as the core of the wall is not exposed, no significant failure is likely.

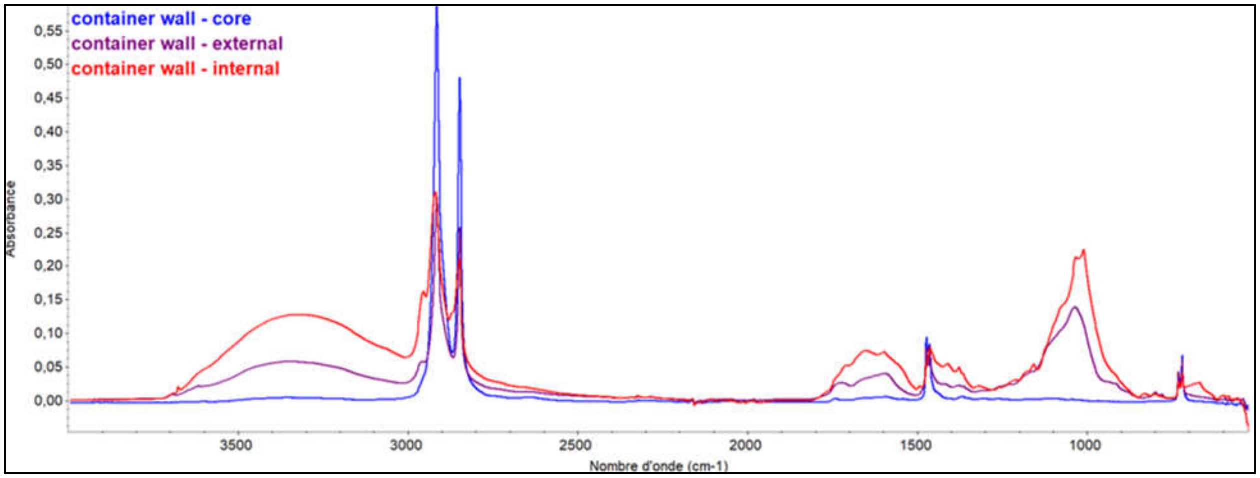

Figure 6: FTIR spectra of the core, internal surface and external surface of the inner bottle wall

The chemical structure of the two closure gaskets are determined to be different as shown by the absorption bands in Figure 7 and Figure 8. The strong presence of carbon black in the black closure gasket (Manufacturer B) makes the interpretation of the absorption bands difficult. While both gaskets seem to be mainly fluoro based, the red closure gasket (Manufacturer A) probably integrates a significant silicon backbone.

The red closure gasket (Manufacturer A) does not exhibit any kind of chemical modification on any surfaces and was barely modified from a chemical point of view.

The black closure gasket (Manufacturer B) exhibits considerably less hydroxyls on the exposed surface in comparison to the protected internal surface. This could possibly indicate drying or degradation of the surface exposed to the IBC for closure. If this modification cannot be quantitatively expressed, such significant modification of the chemical structure has to be further analysed.

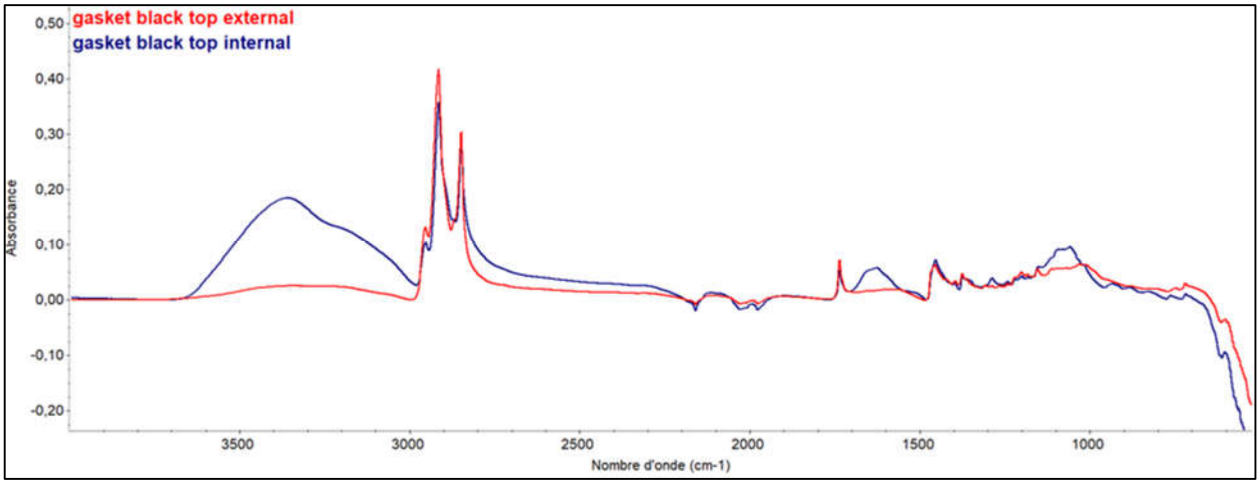

Figure 7: FTIR spectra of the exposed (external) and protected (internal) surfaces of the black top gasket (Manufacturer B)

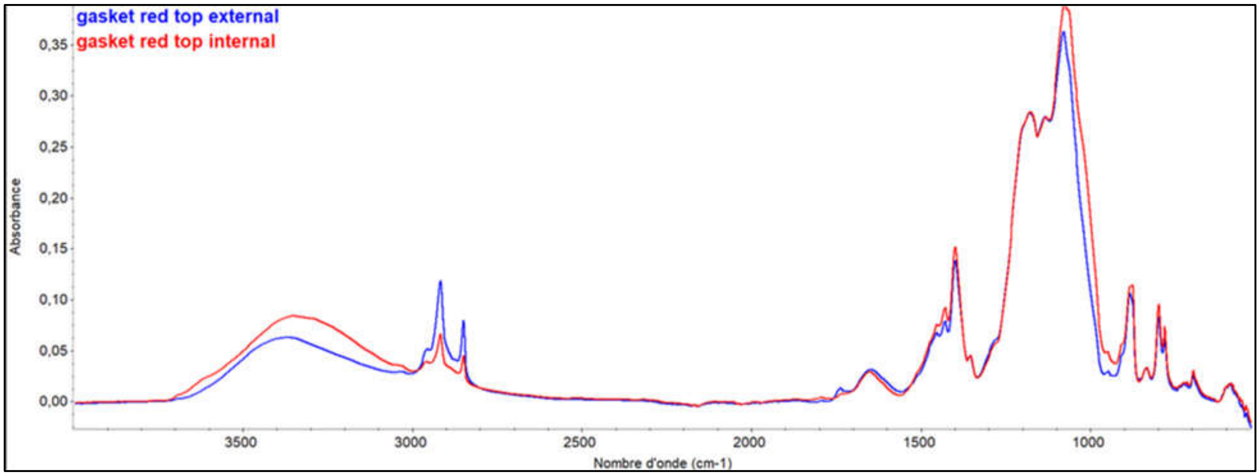

Figure 8: FTIR spectra of the exposed (external) and protected (internal) surfaces of the red top gasket (Manufacturer A)

The chemical structure of the Manufacturer C 2-in. bung was identified as oxidized polyethylene, with the absorption bands shown in Figure 9. The oxidation state is comparable with the external surface of the container walls.

Although the seal is more complicated to analyse, the structure seems to correspond to a highly oxidized rubber, probably polyethylene propylene diene. The exact initial composition is difficult to estimate as the oxidation state is very advanced. A seal in such condition should be replaced, as the oxidation will lead to disintegration and porosity.

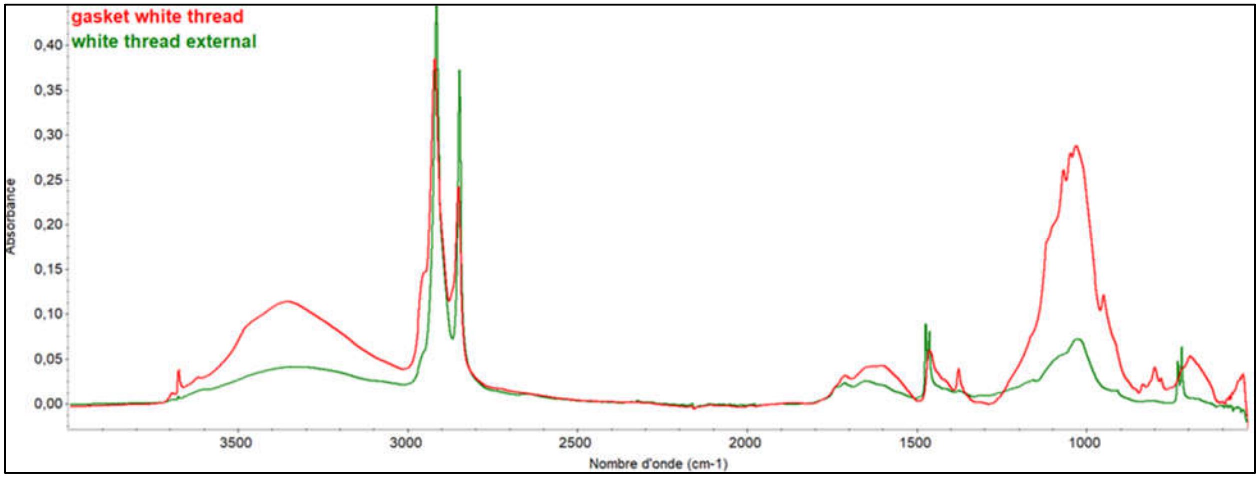

Figure 9: FTIR spectra of the 2-in. bung surface and gasket

4.9.2 Hardness

No significant variations were detected for the internal and external surfaces of the black closure gasket (Manufacturer B) and red closure gasket (Manufacturer A). The elastomeric properties seem untouched, with hardness values and the standard deviation from each of the ten measurements shown in Table 13.

Sample and location	Shore A average	Measurement SD
Manufacturer A closure gasket external surface	50.6	4.1
Manufacturer A closure gasket internal surface	48.6	1.6
Manufacturer C closure gasket external surface	73.4	1.4
Manufacturer C closure gasket internal surface	74.3	0.9
2-in. bung gasket	84.2	1.5
2-in. bung gasket	83.6	0.8

Table 13: Closure gasket hardness measurement average and standard deviation

The 2-in. bung gaskets have similar properties, with quite significant hardness, probably due to the effects of aging.

4.9.3 Scanning electron microscopy

Analysis of the SEM images taken of the external wall of the Manufacturer A container as shown in Figure 10 indicates considerable directional scratching which could be due to the handling or the production of the part. None of the scratches have led to any deep cracks and only negligible damage is visible. From a micro analysis point of view, the surface is still in perfect shape.

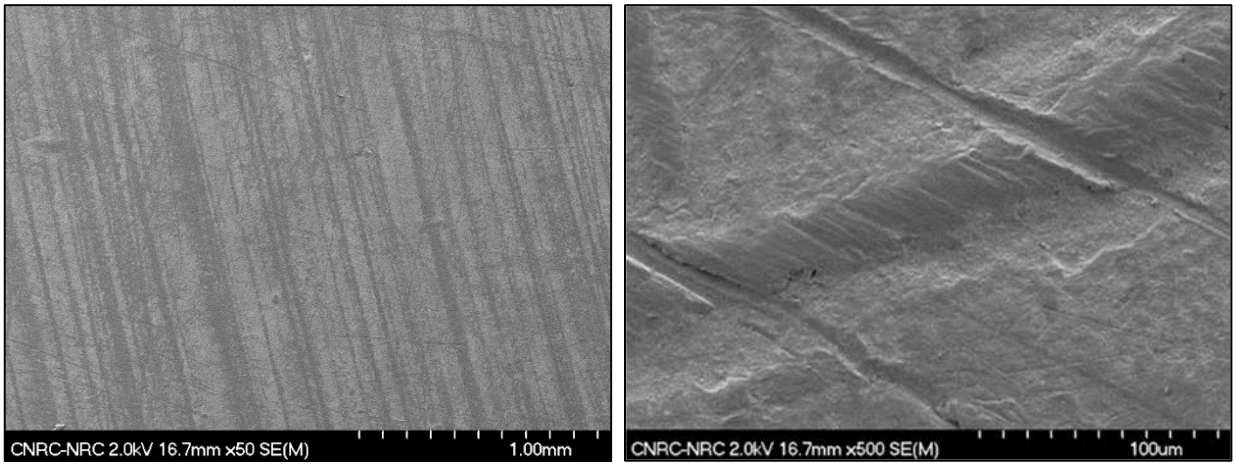

Figure 10: Manufacturer A container SEM images taken at x50 (L) and x500 (R)

Analysis of the SEM images taken of the Manufacturer B container closure gasket as shown in Figure 11 indicate that the surface is still homogeneous with only slight drying and damage visible. Some holes can be seen and close ups show irregular surfaces, signs of slight drying and tearing of the thermoset elastomer material. No clear traces of disintegration can be detected.

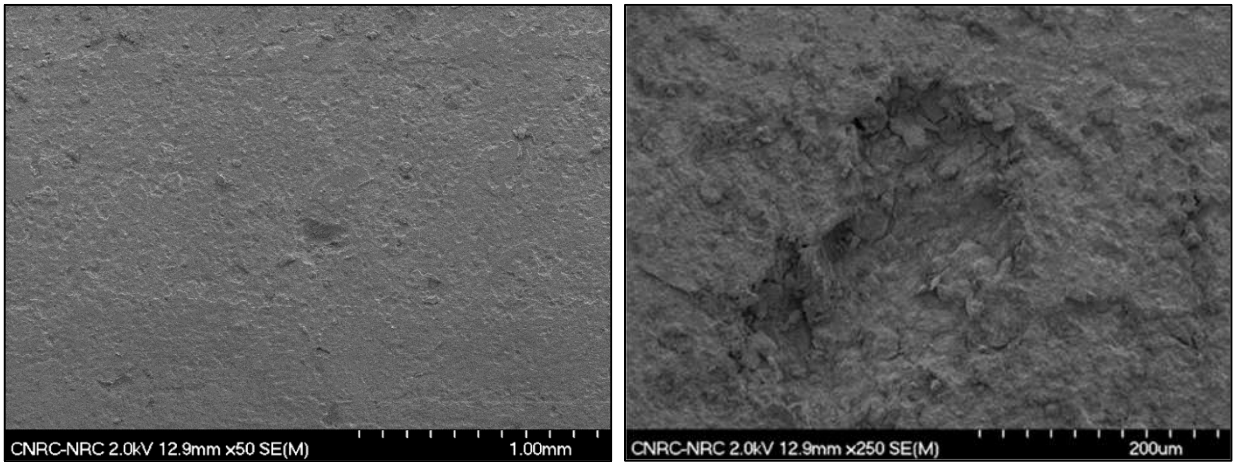

Figure 11: Manufacturer B closure gasket SEM images taken at x50 (L) and x250 (R)

Analysis of the SEM images taken of the Manufacturer A container closure gasket as shown in Figure 12 indicate that the surface of this gasket is relatively similar to the Manufacturer B container closure gasket, however with one difference. The Manufacturer A container closure gasket contains small porosities which can be detected on the bottom part of the ring structure (close to the internal radius), possibly due to the handling of the gasket after the removal from the top (extension). No disintegration can be seen around the porosity, which would support the hypothesis of the damage having occurred post removal. Other than these specific damages, the surface appears to be in good shape.

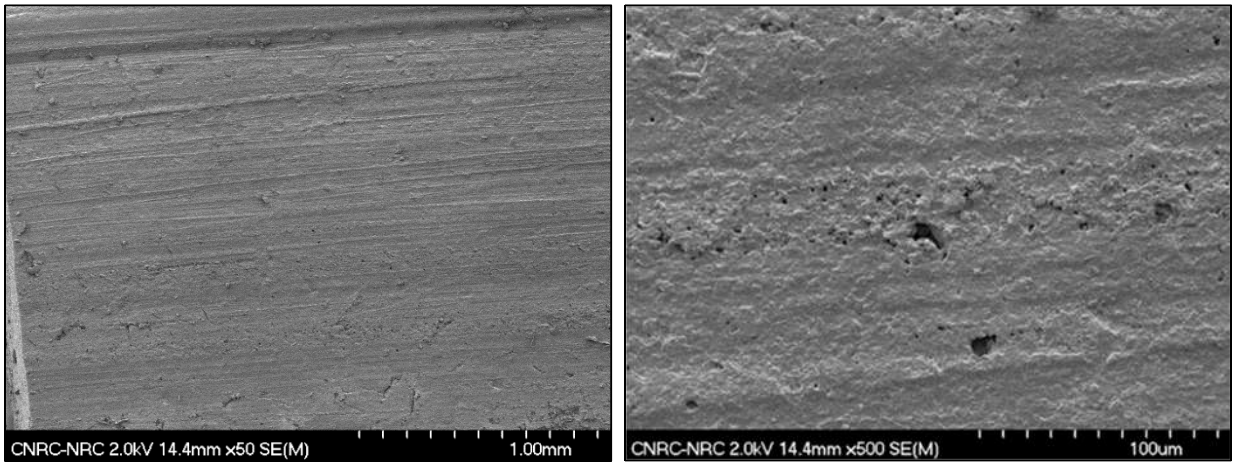

Figure 12: Manufacturer A closure gasket SEM images taken at x50 (L) and x500 (R)

SEM images of the damaged 2-in. bung collar from the Manufacturer C IBC as shown in Figure 13 were taken in the proximity of the failure location. The “hairy” surface is typical of a strong pulling of a polymer plastic part, with a large chunk of polymer being removed (large cracking) at this location. The force was perpendicular to the surface, and the “hairs” are the result of the mechanical separation of strongly intricate polymer chains. Close ups show that below those surfaces, additional cracks are clearly visible and are proof of a general failure of the structure under constraints. Such failure is not due to degradation of the polymer but to an excessive tightening of the thread (pressure applied on the collar) either repeatedly or for a long duration.

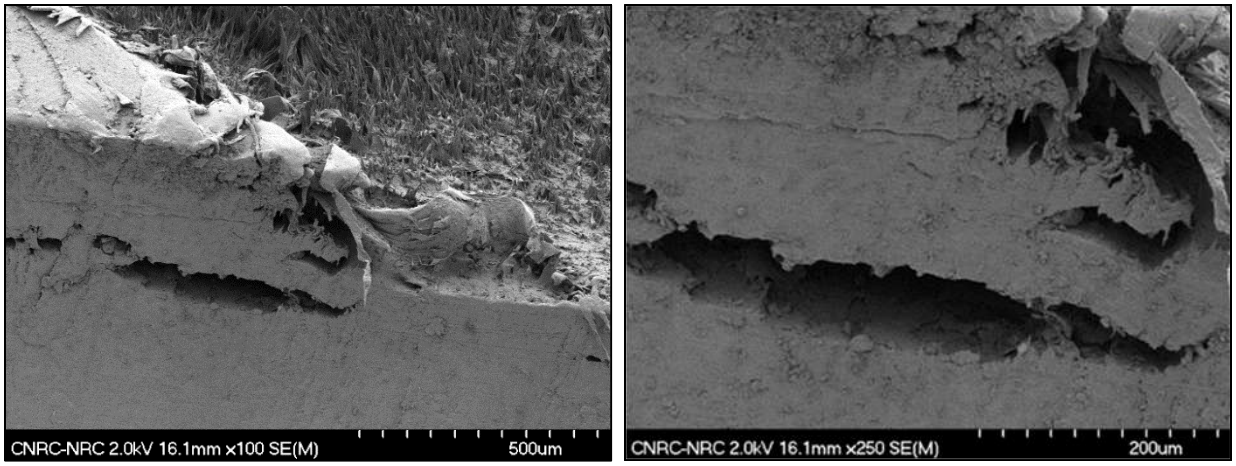

Figure 13: Manufacturer C 2-in. bung SEM images taken at x100 (L) and x250 (R)

SEM images of the damaged 2-in. bung seal from the Manufacturer C IBC shown in Figure 14 indicate significant drying and signs of disintegration. Large parts, especially on the edges, tend to degrade due to oxidation of the rubber structure, leading to a weakening of the thermoset network and the disintegration. As a result the seal will stiffen to an extent and lose its capacity to seal, which perhaps explains why more strength was required to seal the part, resulting in damage to the bung.

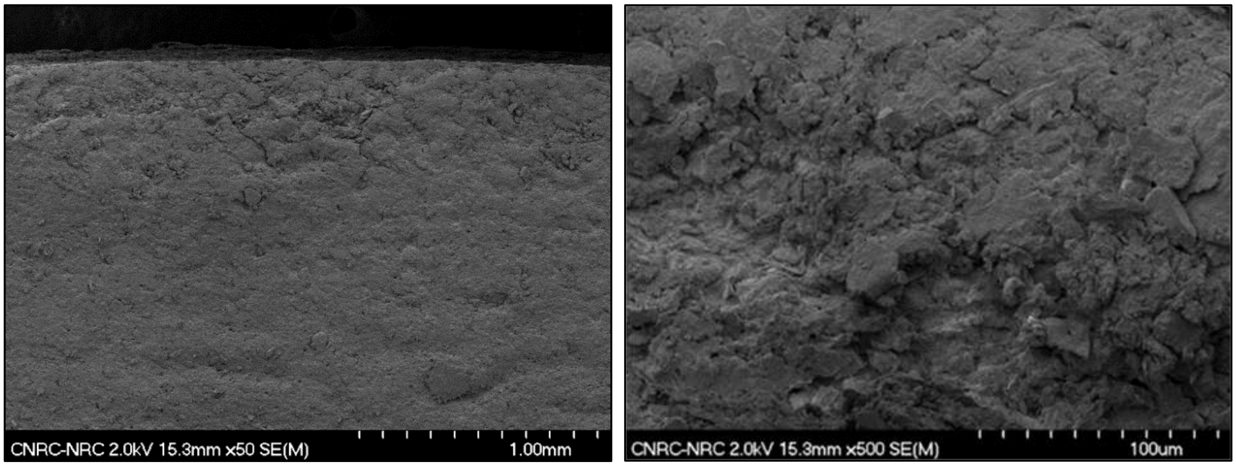

Figure 14: Manufacturer C 2-in. bung gasket SEM images taken at x50 (L) and x500 (R)

4.9.4 Differential Scanning Calorimetry

The thermal properties of the container walls and the 2-in. bung differ greatly as shown in Table 14. The measured glass transition (T_g) and melting (T_m) temperatures of the bung material are significantly higher than that for the container walls with the T_g at least 15°C above, and the melting temperature onset, 30°C above. This variation is mainly due to the type of PE used for the two parts. The walls are produced by rotational molding, in comparison with the bung which is produced by injection molding. Both materials are relatively high density PE, with defined melting, but the density is a little bit lower for the walls, with likely thinner but more numerous crystals. The variation can be due to the additives involved, as nucleating agents, with the walls exhibiting a higher crystallinity but a lower melting temperature. The interpretation is that there are more crystals present, but they are less stable.

Sample analysed	**First Heating Ramp**				**Cooling Ramp**				**Second Heating Ramp**			
	T_g (°C)	**T_m onset (°C)**	**T_m max (°C)**	**ΔH (J/g)**	**T_g (°C)**	**T_c onset (°C)**	**T_c max (°C)**	**ΔH (J/g)**	**T_g (°C)**	**T_m (°C)**	**T_m max (°C)**	**ΔH (J/g)**
PE Walls	-	122	128.5	144	68.5	112.5	115.5	121.5	-	121	127	141
2-in. bung	78	151	164.5	65	87	115.5	112	72.5	-	153.5	160.5	74.5
2-in. bung gasket	-	128	172	6.5	-	-	-	-	-	-	-	-
Red closure gasket	-	78.5	83.5	1.2	-	54	60.5	0.35	-	78	84.5	0.35
Black closure gasket	-	-	-	-	-	-	-	-	-	-	-	-

Table 14: Results from the DSC analysis on IBC material samples

Interestingly, while the container walls seem to exhibit a complete stable crystallization (similar ΔH for both ramps), the bung was not fully crystallized and was quenched too fast in production, as evidenced by the ΔH for the first ramp being significantly lower than for the second ramp. This could lead to residual tension in the bung, and could potentially explain the crack observed when placed under strain.

For a better understanding of the degradation of the materials analysed, ideally a comparison should be made with the fresh materials.

With respect to the elastomeric materials, the red closure gasket and the 2-in. bung gasket both exhibited melting processes during the first ramp. This melting was reproducible in the second ramp for the red closure gasket, but not for the 2-in. bung gasket. The indication is that the 2-in. bung gasket has undergone a non-reversible transformation involving a stress induction, which is characteristic of a degradation under a specific constraint and a compression fatigue. The stress is removed by heating, but the flexibility of such material is reduced. In the case of the red closure gasket, the transition is reversible, and represents a partial crystallization of the hard domains of the elastomeric structure. The ΔH in the second ramp being lower indicates that the seal was also subject to a compression fatigue, but the impact on the flexibility was less damaging.

No such effect is detected on the black closure gasket, where again a comparison ideally should be made with fresh materials.

5 Conclusions

A number of interesting results were observed during the course of testing the three manufacturer's IBCs. It is noteworthy that the Manufacturer C IBCs were tested as received. The Manufacturer A and Manufacturer B IBCs were received without their accompanying steel frames, thus a set of three older steel frames and three new steel frames were obtained for the testing. The plastic pillow pads were supplied with the inner bottles and are believed to be a similar age. A summary of the performance of the IBC test samples is contained in Table 15.

Test	**Manufacturer and IBC identifier**																								
	A							**B**							**C**										
	1	2	3	4	5	6	7	1	2	3	4	5	6	7	1	2	3	4	6	7	8	9	11	13	16
Bottom lift	✓	✓	✓					✓	✓	✓					✓	✓	✓								
Top lift			X							X					✓	✓	✓								
Stacking	✓	✓						✓	✓						✓	✓	✓								
Leakproofness		✓		X		X				✓	X		X		✓			X	X						
Hydraulic pressure		X		✓		X				X	X		✓		X			X	X						
Flat drop (ambient)				✓														X							
Flat drop (cold)												✓										X			
Corner drop (ambient)														X						X					
Corner drop (cold)					X																X		X	X	
Vibration							X		✓																✓

Note: IBC B5 passed the flat drop test (cold) from a height of 0.8 m vs 1.9 m

Table 15: IBC test samples pass (✓) / fail (X) designation after standardized test exposures

Bottom lift tests were uneventful, with all nine IBCs successfully completing the test without damage or reason for concern.

During the top lift test the Manufacturer C IBCs showed no leakage or damage. However the steel frames did suffer damage on the 45° top lift for the Manufacturer A and Manufacturer B IBCs. This was unexpected and outside the scope of this study, which is focusing primarily on the inner bottles. It is of concern, however, as the steel frames should be able to pass this test. Orientation of the shackle used to fasten the IBC to the lifting chains potentially contributed to the failure. Having the pin of the shackle oriented upwards seemed to promote the damage to the lifting eye. The shackle pulled through the steel eye, ultimately dropping the load. The loss of use of the lifting location and the amount of deformation at the lifting eye leads to the conclusion that further safe shipment of the IBC would not be possible. Further testing was halted in order to preserve the limited number of steel frames for other tests. In discussions with riggers from a local crane company the orientation of the shackle during a lift is not something that is standardized, their comment being that either orientation should be acceptable. This result could be an area for further study.

Leakproofness tests were performed on three of each manufacturer's IBCs; however, the Manufacturer A and Manufacturer B IBCs were tested by reusing the two damaged steel frames from the top lift test, again to preserve remaining frames for future testing. It is believed that this had no impact on the test results obtained. All types of IBCs had some air leakage at an air pressure lower than the required 20 kPa. All leaks observed were at the top closure systems, no leaks were observed at the bottom valves. Tests were conducted with pressure transducers installed in the closures for two of the three sets of IBCs, and mounted directly on the inner bottle wall for the third set of IBCs, with no significant difference in results noted. Closures are quite rigid and the method used to mount pressure transducers would not have compromised the closure integrity in any way. Leakage past pressure relief valves was noted at pressures much less than the relief rating. The Manufacturer C IBCs did not incorporate pressure relief valves but did have 2-in. drum style closures mounted. Leakage through these closures was observed. Upon visual inspection after testing it appears the closures had been over torqued. SEM and other mechanical tests performed support this conclusion with evidence that the closures may have residual stress from the initial fabrication processes. This is a topic that could warrant further investigation.

Upon retesting six samples with new gaskets installed, all IBCs were able to successfully pass the leakproofness test with only one requiring the higher torque application.

The hydraulic pressure tests were conducted immediately after the leakproofness tests. Only two of the nine IBCs tested were able to achieve the full test pressure without leaking. Exact leakage pressures were difficult to determine in some cases as entrapped air tended to move towards the closure areas at the start of each test. Some IBCs from Manufacturer A and Manufacturer B may have been subjected to negative pressures during their lifetimes as indicated by the concave shape of the inner bottle top. The lowering of the closure area allows air to be trapped at the inner bottle edges when filling to brimful. Low pressures can occur during unloading if the top seals are not vented properly, or it may occur due to thermal contraction if the empty IBC is cooled while sealed. In one instance, IBC C4, an air leak was discovered at a 2-in. bung closure that had been missed in the leakproofness test. It is believed that during the leakproofness test, the leak was such that air blew out the soap film without being detected. The bung was replaced and a leak at the main closure was subsequently witnessed.

The IBCs showed evidence that product had been in contact with the exterior of the inner bottles, indicating that leakage may have occurred while the IBC was used to contain dangerous goods.

Two of six IBCs which were retested using closures with new gaskets passed the tests where failures were first recorded. However, new failure modes were also observed on some IBCs once higher pressures were achieved, such as bulging causing a bottom valve to open, and a crack through the wall of the inner bottle. The one sample which was originally a pass, failed the test with a new dry gasket then passed with lubrication applied. It is evident from the results that gaskets must be lubricated before being installed. The topic of gasket lubrication is one which could be studied further

Stacking tests were successfully completed without incident. This test is primarily a test of the frames, and no failures were observed.

Drop tests were performed at ambient temperatures on four IBCs. Drops at ambient temperature are generally considered less severe, as plastic tends to become more brittle at lower temperatures. One flat drop and one corner drop were conducted on IBCs with steel frames and plastic frames. Some damage occurred on all IBCs dropped. The standard requires a judgement call by the test engineer on whether the IBC is safe for transport if damage to the IBC does occur, unless product leakage occurs, which is automatic grounds for failure.

The authors had some discussions on the significance of the level of damage observed. The standard requires that there be “no damage which renders the IBC unsafe to be transported, and no loss of contents”. The Manufacturer C IBC, when dropped on its corner, came to rest on its side. Loss of contents was evident thus this would be considered a failed test. Though landing on its base, the corner dropped steel frame Manufacturer A IBC was intentionally tipped onto its side after testing. Prior experience with corner drops shows the final resting position is somewhat random. When tipped to its side this IBC also leaked; however, both these leaks may be attributed to poor closure performance, which was observed during leakproofness and hydraulic pressure tests rather than the drop test.

Though this is not a requirement of the standard, one possible method to determine if the IBCs are safe for transport after dropping would be to conduct a vibration test on them. The concern will be if broken frame members rub against the inner bottle material causing it to wear thin or puncture resulting in product loss. This could be quite valuable research, as the damage to the corner dropped steel frame did not appear unusual relative to other corner drops that the NRC has witnessed on IBCs in the past. This may prove to be a difficult task as the IBCs are no longer symmetrical and this may induce significant lateral motion on the vibration platform. This approach is not presently in the standard.

Damage to the frame of the Manufacturer C IBCs was such that further shipment would not be advisable, thus the IBC was deemed to have failed the test. The composite frame damage was also significant, but further shipment may have been possible without leakage. Deflection at the corner is common with corner drops; however, the high mass rating of these IBCs resulted in greater deflection.

The external frames were damaged on both IBCs that were flat dropped. The bottom cushion on the steel frame IBC was also deformed significantly after the flat drop. This damage is not easily visible and it is quite likely that the IBCs would continue to be transported. The crushed cushion meant that proper bottom valve orientation was affected. The bottom frame member interfered with the attachment of a fitting to empty the IBC. In comparison, the crack in the Manufacturer C IBC base is much more obvious, and it is questionable if this IBC is still able to be handled safely. Thus the Manufacturer B IBC would be the only potential pass from this set of tests.

Vibration testing on one of each IBC design was conducted with one clear failure observed on the Manufacturer A IBC. Flexing at both top gussets along the long edges resulted in cracks forming and penetrating the inner bottle and allowing contents to leak. The Manufacturer B IBC did not leak after an hour of vibration, thus it passed the test. However, cracks were forming in a similar location and it is of little doubt that leakage would eventually occur if the test was extended. It is noteworthy that the one hour vibration test is of a similar duration to methods commonly used for testing single use IBCs for non-dangerous goods.

The three IBC types tested passed the bottom lift and stacking tests. The metal frames did not pass the top lift test, while the plastic IBC did pass this test. All three IBC types experienced failures in both the leakproofness and hydraulic pressure tests. While the installation of new gaskets improved the leakproofness and hydraulic pressure test results, multiple failures were still observed. Cold drop tests at the height for which the IBCs were rated were all failures. Of the few IBCs subjected to vibration testing, the plastic IBC passed the test, while one composite IBC failed by leaking and the second was developing cracks that would make it unsuitable for shipment. Overall, the three IBC types, which were beyond their prescribed period of use, did not continue to meet the performance test requirements of a new IBC design.

Project Team

The project team consisted of the following personnel:

- project manager: Al Hendley
- test engineers: Don Leblanc, Todd Garrett, Patrick Kehoe, Damien Maillard
- instrumentation technologists/technical support: Rick Lamb, Luc Levesque, Sybren Mulder, Leo Vanderlaan, Tony Hustoft, Wayne MacLeod
- report editors: Jon Preston-Thomas, Bruce Gaudet

Acronyms and abbreviations

ATR	attenuated total reflectance
DSC	differential scanning calorimetry
DTGS	deuterated triglycine sulfate
FEG	field emission gun
F.S.	full scale
FTIR	Fourier transformed infrared spectroscopy
g	acceleration due to gravity
IBC	intermediate bulk container
NRC	National Research Council of Canada
PE	polyethylene
SEM	scanning electron microscopy
TC	Transport Canada

References

[1] Canadian General Standards Board, *CAN/CGSB-43.146-2016, Design, manufacture and use of intermediate bulk containers for the transportation of dangerous goods, classes 3, 4, 5, 6.1, 8 and 9,* 2016.

[2] *Recommendations on the Transport of Dangerous Goods - Model Regulations - Volume 1, twenty-first revised edition,* New York and Geneva: United Nations, 2019.