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Cumulative Fuel System Life Cycle and Durability Testing of Hydrogen Containers

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16. Abstract

The goal of this Task Order was to assess the proposed test conditions used for evaluating life cycle durability of high-pressure hydrogen fuel containers. The key test conditions evaluated were representative high and low cycling temperatures, static hold time at high pressure, and the number of pneumatic hydrogen cycles required to reveal poor durability performance of high-pressure hydrogen fuel containers. The results of this Task Order provide data for NHTSA's assessment and consideration as it evaluates the safety performance of compressed hydrogen fuel systems and the need for enhancement of appropriate FMVSS. In addition, the results of this Task Order provide data to assist in resolving several open issues and concerns in the pneumatic test sequence of the 2009 edition of the Expected-Service Performance Verification Test procedure in the SAE Technical Information Report (TIR) 2579 for Fuel Systems in Fuel Cell and Other Hydrogen Vehicles.

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1.0 INTRODUCTION

Hydrogen-fueled vehicles offer the promise of significantly reducing the amount of pollutants expelled into the environment. Furthermore, hydrogen can be generated from a number of diverse energy sources, including hydrocarbon, nuclear, solar, and wind, helping address energy security as well as environmental concerns. While its use is promising from both of these perspectives, hydrogen is a challenging fuel that, for practical vehicle range, must be stored as a highly compressed gas, as a cryogenic liquid, or in chemical metal hydride form such as sodium borohydride. Fuel cell vehicles that are fueled by hydrogen also present the challenge of how to safely transfer power from the fuel cells to motors through high-voltage buses. Hydrogen vehicles introduce hazards that are different from those of conventionally fueled vehicles that consumers, mechanics, fire safety personnel, the public, and engineers already know and understand. Nevertheless, the public demands that hydrogen vehicles be no more hazardous to own and operate than conventional gasoline or diesel fueled vehicles.

The National Highway Traffic Safety Administration promotes the safety of vehicles through several means, including setting and enforcing safety performance standards for motor vehicles and associated equipment through regulations such as those set forth in the Federal Motor Vehicle Safety Standards (FMVSS). Recognizing the hazards and issues associated with use of hydrogen fuel, NHTSA is undertaking risk assessment studies to quantify potentially unsafe conditions, developing performance tests to address these conditions, and evaluating procedures to help ensure that hydrogen-fueled vehicles exhibit an equivalent level of safety to that of conventionally fueled vehicles.

Toward this end, NHTSA has awarded a contract to a team, led by Battelle, to evaluate various technical aspects of the safety of hydrogen fueled vehicles. Seven task orders have been awarded to date under that contract. The purpose of Task Order 2, Cumulative Fuel System Life Cycle and Durability Testing, is to assess test conditions representative of the life cycle of hydrogen vehicle fuel containers. This Task Order is led by Powertech Labs as a subcontractor to Battelle. This document summarizes the results of this Task Order including Task 2a, which examined the effects of high and low temperatures during pneumatic hydrogen pressure cycling; Task 2b, which examined the effects of high-pressure static hold time between pneumatic cycles; and Task 2c, which examined the effects of the number of pneumatic hydrogen pressure cycles on container durability.

1.1 Purpose and Scope of Document

The purpose of this report is to summarize the work performed, describe the results obtained, and assess those results under Task Order 2, Cumulative Fuel System Life Cycle and Durability Testing of the NHTSA Hydrogen Vehicle Fuel System Safety Research Program.

1.2 Task Order Goals and Objectives

The goal of this Task Order was to assess the test conditions used for evaluating life cycle durability of high-pressure hydrogen fuel containers. The objective of this effort was to assess the influence of key test conditions of high and low temperatures, static hold time at high pressure, and number of pneumatic hydrogen cycles on likelihood of damage to high-pressure hydrogen fuel containers. The results of this Task Order provide data for NHTSA's assessment and consideration as it evaluates the safety performance of compressed hydrogen fuel systems and the need for enhancement of appropriate FMVSS. In addition, the results of this Task Order provide data to assist in resolving several open issues and concerns in the 2009 edition of the Expected-Service Performance Verification Test procedure in the SAE Technical Information Report (TIR) 2579 for Fuel Systems in Fuel Cell and Other Hydrogen Vehicles [1].

2.0 WORK PLAN OVERVIEW

This section summarizes background information and technical rationale for the work conducted under this Task Order, as well as a description of the facilities, test equipment, and assessment methodology used for the work.

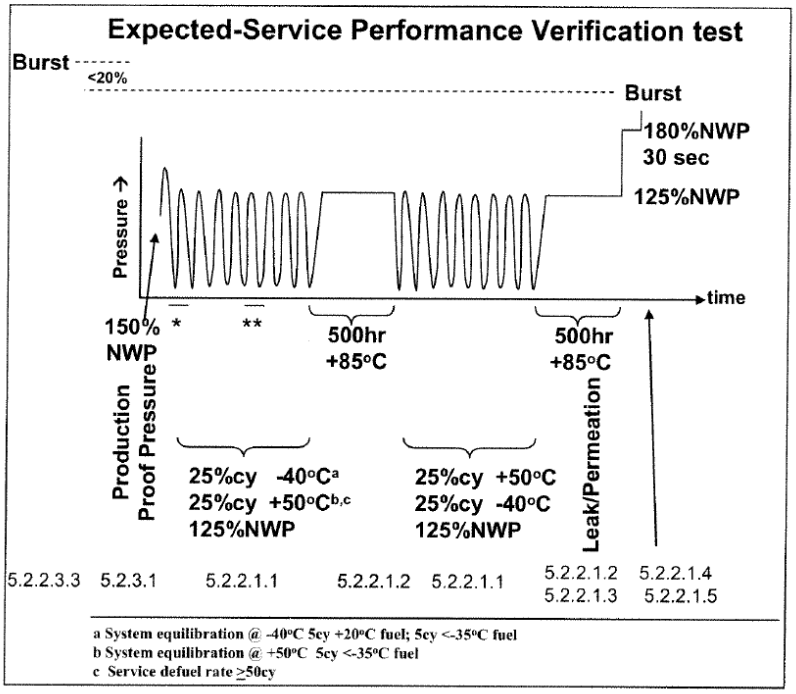
2.1 Background and Problem Statement

The SAE Technical Information Report 2579 for Fuel Systems in Fuel Cell and Other Hydrogen Vehicles [1] identifies tests that are intended to evaluate the cumulative, compounded stress of various exposures of the fuel system to pneumatic hydrogen fueling and defueling, and parking during ambient temperature conditions, including durability of the fuel system after drop and chemical exposure. This TIR is intended for use during the precommercial period of technology development and vehicle evaluation. Industry is currently conducting research to evaluate these test methods in order to ensure they are appropriate and practical.

The test program conducted under this Task Order has been designed to generate data to assist NHTSA and SAE International (Society of Automotive Engineers) in understanding container durability and reliability and in refining the Expected-Service Performance Verification Test procedure in the TIR. The experiments reported here are based on the evolving standard, which is being updated as new information is generated.

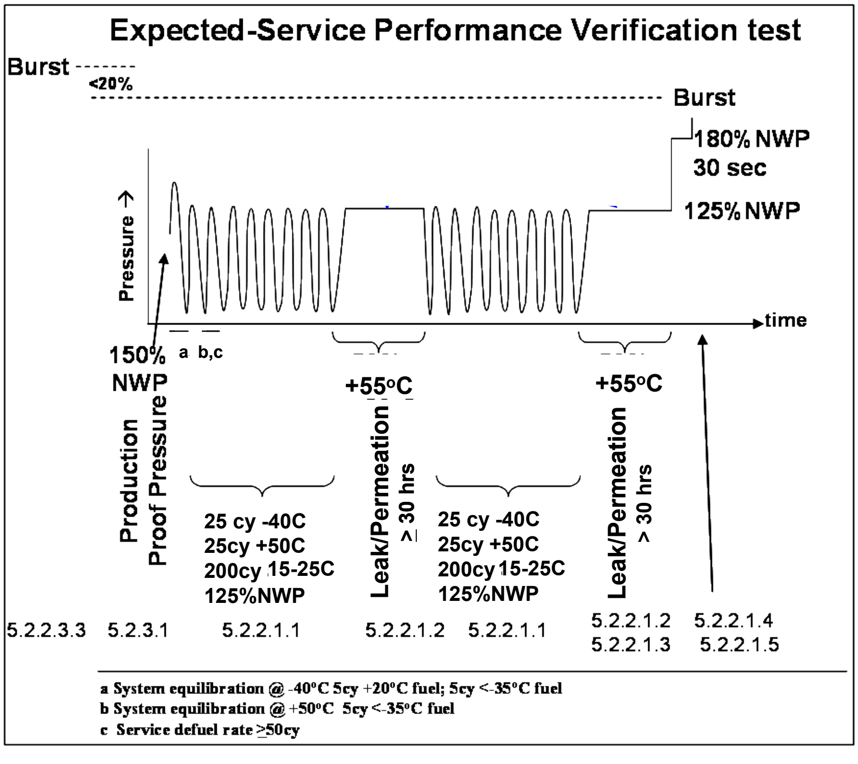
2.2 Technical Discussion and Rationale

SAE TIR J2579 was originally released in January 2008 [2]. That was the current version when the work plan for this project was originally written [3]. A revision to the TIR was released a year later in January 2009 [1]. Figure 1 below shows Figure 4 from the TIR, which is a pictorial summary of the pneumatic Expected-Service Performance Test from Section 5.2.2.1 of the TIR. A new draft version of the TIR was circulated following the November 2009 Fuel Cell Safety Working Group meetings with a modified test sequence [4]. The revised draft of the pneumatic test sequence is shown in Figure 2. Several significant areas have been affected, three of which were evaluated in this test program.



a System equilibration @ -40°C 5cy +20°C fuel; 5cy <-35°C fuel
b System equilibration @ +50°C 5cy <-35°C fuel
c Service defuel rate ≥50cy

Figure 1. Test sequence of SAE TIR J2579, as it was published in January 2009 [1].



a System equilibration @ -40°C 5cy +20°C fuel; 5cy <-35°C fuel
b System equilibration @ +50°C 5cy <-35°C fuel
c Service defuel rate ≥50cy

Figure 2. Test sequence of the draft SAE TIR J2579 following the November 2009 working group meetings [4]. Among other changes, the temperature and high-pressure static hold duration were revised.

SAE TIR J2579 has been based on industry experience and discussion. There is little data available to confirm recommended test methods. Some of the acceptance criteria in the test sequence were openly debated without complete consensus of participants. Consequently, “reasonable” values have been used by the committee in draft test methods until more data or a more comprehensive rationale becomes available. Three key unresolved issues in the Expected-Service Performance Verification Test are

- High and low test temperatures during pneumatic pressure cycling;
- Duration of high-pressure hydrogen static hold and achievement of steady-state permeation between pressure cycle series; and
- Total number of pneumatic pressure cycles.

Accordingly, the testing in this Task Order was organized into three distinct tasks, each generating data to assist in addressing one of these unresolved issues in the pneumatic test sequence in the draft revision of SAE TIR J2579 illustrated in Figure 3. Task 2a within this Task Order examined the high and low test temperatures. Task 2b examined the high-pressure static hold duration, and Task 2c examined the number of pneumatic pressure cycles. In each case testing was conducted to assess the influence of different test conditions on potential damage to the cylinder and on the ability of the test to discriminate durability of the container. Following is a discussion of the background for each test. Section 3 describes test procedures, test data, and assessment of test results.

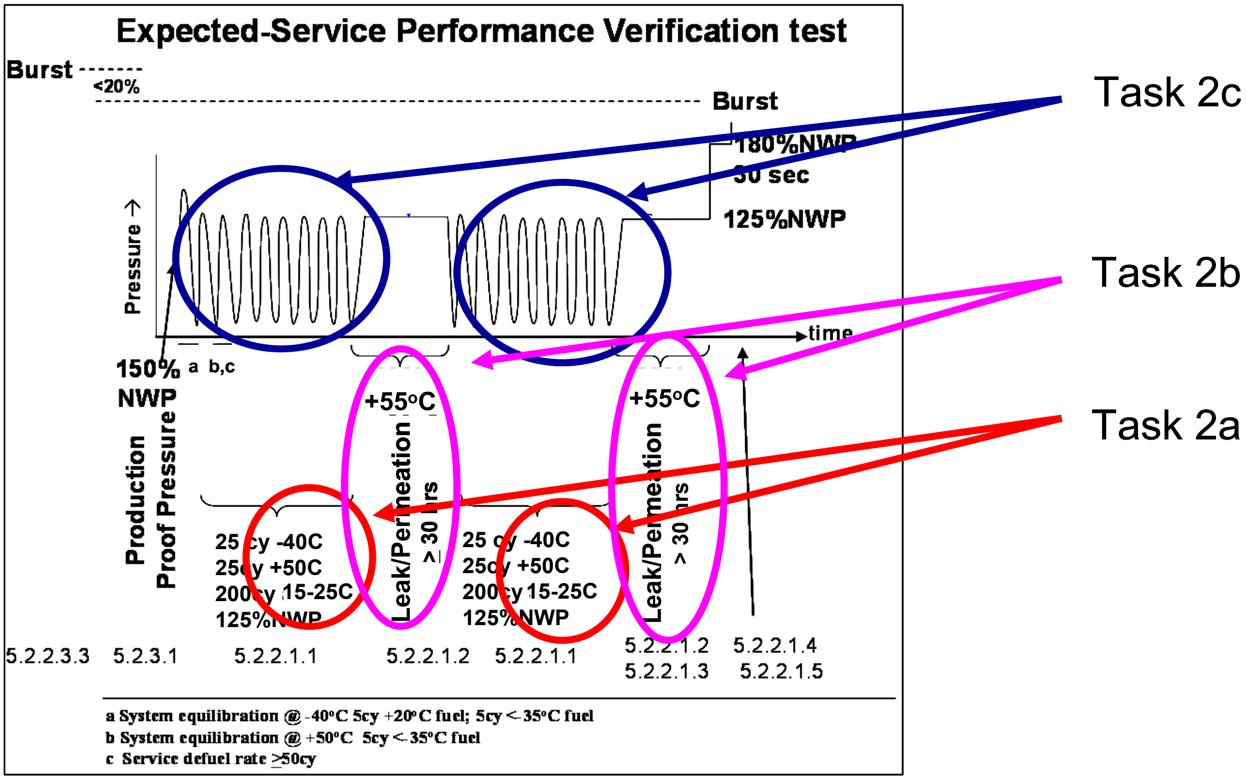


Figure 3. Illustration of the test conditions examined in Subtasks 2a, 2b and 2c.

2.2.1 Task 2a – High and Low Test Temperature Pneumatic Cycling Test Conditions Background

High-pressure gas diffuses in and out of fuel container liners during pneumatic cycling. Over many cycles at extreme conditions, this cyclic diffusion may damage the liner, particularly non-metallic liners, causing blistering and cracking, leading to excessive permeation or leakage. This form of damage may be influenced by the maximum and minimum temperatures experienced during fueling and during normal fuel use in vehicle operation (container defueling).

The maximum and minimum test temperatures recommended for pneumatic cycling in SAE TIR J2579 were chosen by the TIR committee to be +50°C and –40°C. These values were originally selected based on the rationale they are the highest and lowest *ambient* temperatures a vehicle is likely to encounter. The high temperature value has been discussed within the Fuel Cell Safety Working Group for several years, with values as high as +85°C and as low as +40°C proposed by committee members. A new approach to defining the test temperature has been adopted by the SAE J2601 Interface Working Group [5]. The peak fuel system gas and component temperature has been defined in the Working Group as +65°C based on testing data generated by automotive original equipment manufacturers (OEMs) on combined effects of ambient conditions, solar radiation, and gas temperature heating. This temperature is significantly higher than the current +50°C in SAE TIR J2579. The work of this committee has not defined how frequently this may be experienced over the life of the vehicle fleet or the total duration of exposure over a vehicle's lifetime.

The effects of low temperature extremes are different than those of high temperatures. Cycling at low temperature may affect the performance of the container liners by both increasing the permeation rate and by causing physical damage such as cracking or pulling away from the boss interface. The current published TIR test sequence evaluates the performance of the container at the lower temperature of –40°C. This temperature has been determined by industry to be the minimum expected *ambient* temperature these containers are likely to experience in service. However, most vehicle manufacturers have indicated within the SAE Fuel Cell Interface Working Group that they do not currently intend that their vehicles be operated at temperatures this low. Vehicle operation at –40°C will be very limited and at much reduced performance. Full vehicle operation is supported only down to temperatures of –25°C. A reduced "limp home" mode performance may be supported at –40°C.

Current fuel system designs cannot be tested at –40°C without violating the OEM-imposed fuel system operational limits. An operating temperature of –25°C has been proposed as an alternative low temperature for normal fuel cell and fuel system operation.

2.2.2 Task 2b – High-Pressure Hydrogen Static Hold Duration Background

The current long-term static high-pressure hydrogen "parking performance" test has been eliminated from the pneumatic Expected-Service Performance Verification test sequence and added to the hydraulic Durability Performance Verification test sequence as a traditional accelerated stress rupture (ASR) test.

A new leak permeation test performed at +55 °C has been proposed, as illustrated in Figure 2. The duration of this test is currently specified to be a minimum of 30 hours, but it must be continued until steady-state permeation is achieved. The rationale for this specification is that Type 4 containers may take several hundred hours to achieve steady-state permeation, whereas a Type 3 container will require only time for the O-rings and seals to fully saturate with hydrogen, before steady-state permeation values are reached. However, the containers will be pressurized to some degree during early cycling phases of the test sequence, so that time necessary to achieve steady-state permeation may be less in the static phase of this complex series than in an isolated permeation test.

The 30-hour minimum exposure was selected based on a single known failure of a Type 3 metal-lined container in less than 30 hours when subjected to static high-pressure hydrogen. This incident occurred at Powertech Labs on a prototype container design using a novel liner forming process. The liner material had originally been subjected to coupon-style hydrogen embrittlement testing and met those requirements. A finished container was then hydrogen-gas cycled 1,000 times at ambient temperature with no leakage detected. Following the gas cycle test, a static high-pressure hydrogen test was performed. In less than 30 hours, all gas escaped from the container. Further examination determined that micro-cracking of the metallic liner occurred due to hydrogen embrittlement. This result indicated that the high-pressure hydrogen exposure during gas cycling is too brief to evaluate this type of failure mode. For this reason, a static high-pressure hydrogen hold has been added to the SAE TIR J2579 pneumatic test sequence. An assessment of degradation of liners during testing, such as cracking, would help determine if the test duration is sufficient to help detect hydrogen compatibility issues. Neither the duration of this static high-pressure hydrogen hold nor the temperature of this test has been validated.

In addition, a rationally defined test procedure for determining steady-state permeation was also needed to ensure consistent results between test laboratories. A comparison of the permeation levels measured on test containers would help determine the minimum duration of testing required to achieve steady-state permeation.

Powertech has observed that the liner of a Type 4 container may buckle during depressurization at the conclusion of a pressure test as shown in Figure 4 below. The hydrogen trapped between the liner and composite has a higher pressure than the internal liner pressure, creating an inward force. This can cause the liner to buckle when a container that is saturated with hydrogen is vented completely down to atmospheric pressure. Because the containers should not be vented to atmosphere in service, this is not a realistic service condition and does not suggest damage or failure under the conditions being examined in this task.

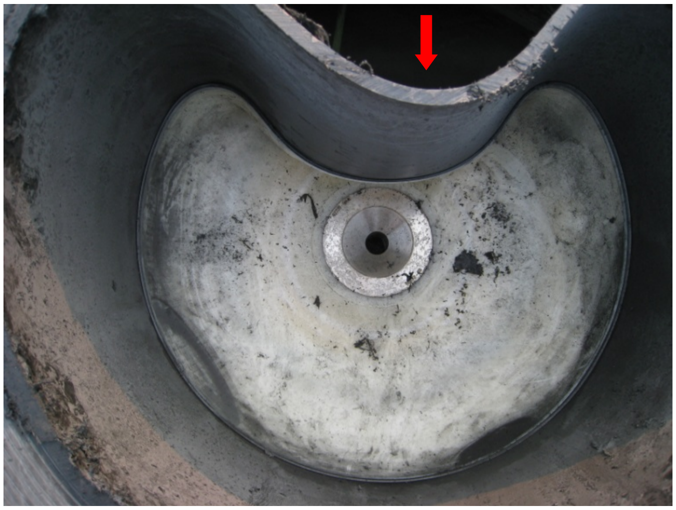


Figure 4. The liner buckling evident in this Type 4 container occurred when it was vented to atmospheric pressure following a pressure test.

2.2.3 Task 2c – Pneumatic Pressure Cycle Count Background

The pneumatic hydrogen pressure cycle count in the TIR Expected-Service Performance Test was defined by dividing the lifetime vehicle mileage range by refueling mileage range, with a minimum of 500 refueling cycles. In contrast, the maximum pressure cycle lifetime of containers is assumed by the TIR to be 1,833 cycles. No testing has been done to determine if applying 500 pneumatic cycles during this Expected-Service Performance Verification test is sufficient to discriminate the fuel system's durability over its presumed lifetime of 1,833 cycles. Testing to determine the number of pressure cycles necessary to assess durability of a container over its life cycle would provide confidence in the specified approach. In addition, confirmation that testing to less than the complete lifetime of 1,833 cycles would have the added benefit of reducing the duration of the test sequence and the cost of conducting the testing.

2.3 Facilities and Equipment

Powertech has designed and constructed two parallel apparatus for conducting the pneumatic Expected-Service Performance Test sequence in accordance with SAE TIR J2579. Each apparatus consists of the following equipment.

- Environmental chamber for the test container or fuel system
- Hydrogen gas pre-cooler
- Hydrogen gas flow control system (inlet and outlet)
- Hydrogen compression to 100 MPa
- High-pressure hydrogen storage (88 MPa)
- Low-pressure hydrogen storage (1 MPa)

The schematic in Figure 5 shows the most significant components of the apparatus.

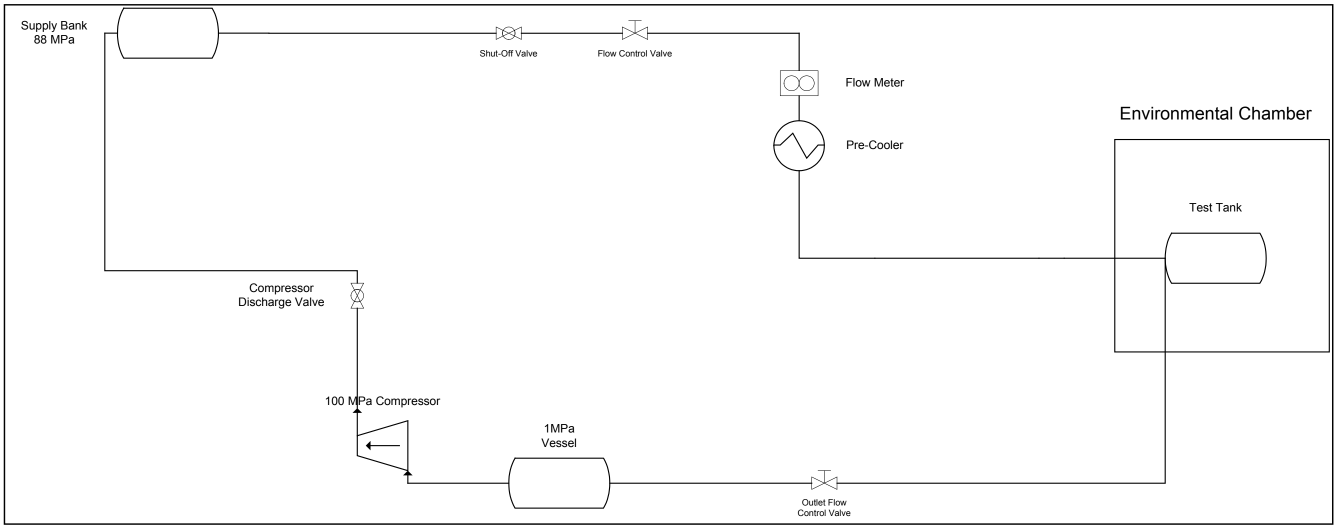


Figure 5. Schematic of the Powertech Labs apparatus for the SAE J2579 pneumatic testing.

Figure 6 and Figure 7 are photographs of the test equipment.

Figure 6. Powertech Labs SAE J2579 Pneumatic Test Facility.

Figure 7. Powertech Labs SAE J2579 Pneumatic Test Facility Inlet Flow Control Panel.

2.4 Instrumentation and Data Collection

All data was collected using National Instruments Data Acquisition hardware and Powertech custom data logging software (using the LabVIEW formatting).

All test containers were instrumented with an internal gas temperature sensor (K-type thermocouple), an end plug temperature sensor (K-type thermocouple), and a gas pressure sensor (Stellar model GT1600-15000). Ambient temperature measurements were made using a K-type thermocouple.

Data sampling rates during cycle testing were 2 Hz, although only the maximum and minimum value of each data channel were recorded during each cycle. The first 5 cycles and final 5 cycles had data recorded continuously at 2 Hz.

Data sampling during the hold portions of the testing occurred once per minute.

Permeation measurements were made using Powertech's Agilent 3000 gas chromatograph. Samples were measured and data stored as a ppm value of hydrogen in the background gas (nitrogen). Permeation samples were taken as often as required to obtain a clear indication of breakthrough and achievement of steady-state permeation.

Liner examination was visual and utilized a borescope. Video recordings of the borescope examinations were made, and still images were used for evaluation criteria and reporting requirements.

The pressure and temperature data collected during the testing were stored and plotted for informational purposes and possible future analysis.

2.5 Data Analysis Methodology and Evaluation Criteria

Container durability and degradation were assessed by measuring changes in permeation rate and by visual inspection of the liner. Permeation data were assessed through tabular and graphical comparisons. Liner deterioration was assessed via comparisons of sequential still photographs showing the same location of the liner during each examination. The change in permeation of each individual container was plotted against its point in the test sequence to show overall change. The liner examination data were treated in the same fashion.

Typically, a failure of a container is clear and not subject to interpretation. Liner deterioration is clearly shown by changes from the as-manufactured pristine condition, as evidenced by blistering, cracking, and pulling away from the boss. Small changes in permeation observed due to variations in ambient conditions were at least an order of magnitude lower than a permeation change observed due to container deterioration, such that the two causes were easy to distinguish.

3.0 TESTING, RESULTS, AND ASSESSMENT

This section of the report summarizes the testing, results, and assessment for each series of tests conducted in the project by subtask.

3.1 Task 2a – Assessment of High- and Low-Temperature Pneumatic Cycling Test Conditions

3.1.1 Temperature Selection

The goal of high- and low-temperature testing in Task 2a was to compare the effects of pneumatic cycling at different temperatures on liner permeation and physical damage, such as blistering, cracking, or pull-away from the boss interface. This testing provides data to support selection of the high and low temperatures used for pneumatic hydrogen cycle testing. If either temperature extreme affects permeation or causes physical damage, then further investigation may be required to determine a suitable high and/or low test temperature that is representative of field conditions and that verifies durability of the containers throughout their life cycle, without being excessive. If container performance is affected little by the choice of high and/or low temperature, then a nominal value, such as expected maximum and minimum ambient temperatures, may be suitable as the high and low temperatures for pneumatic cycle testing.

The effects of high- and low-temperature pneumatic cycling have been shown previously to be more severe on plastic liners and on the interface between the liner and end boss of Type 4 containers, than on metallic liners in Type 3 containers. Lower heat transfer in plastic liners may lead to higher peak gas temperature at the boss of Type 4 containers during fueling. Consequently, testing in this subtask was performed only on Type 4 containers.

3.1.2 Test Procedure

This experiment evaluated pneumatic hydrogen cycle testing at four temperatures, two high and two low, shown in Table 1. A separate container was used for each test condition. The containers were monitored for degradation via pre- and post-test permeation measurements and internal visual examination, as well as intermediate permeation measurements following 50 and 100 cycles.

Table 1. High- and Low-Temperature Pneumatic Cycle Test Matrix.

Test	Container Ambient Temperature	Number of Cycles
2a1-1	+50°C	200
2a1-2	+65°C	200
2a2-1	-40°C	200
2a2-2	-25°C	200

The following steps were performed on each of four Type 4 containers, one for each of the test temperatures listed in Table 1.

1. The container was filled to 70 MPa at a settled temperature of +15°C.
2. The container was placed in a permeation chamber and held until a steady-state permeation was achieved. The resulting value was recorded as the baseline pretest value for that specific container. This baseline value represents a short-duration steady state value and will differ from the value shown during cylinder qualification tests which require 500 hours minimum hold time.
3. The container was then depressurized to 35 MPa at +15°C, and placed in the appropriate environmental chamber to soak at the test temperature shown in Table 1.
4. Following soaking at the test temperature for at least 24 hours, 50 pneumatic hydrogen pressure cycles were applied to the container.
5. The container was then returned to +15°C and repressurized to 70 MPa. This was followed by a 72-hour permeation measurement.
6. The container was again depressurized to 35 MPa at +15°C, soaked at the test temperature, and cycled another 50 cycles to complete a total of 100 cycles.
7. Another repressurization and permeation test was then performed.
8. The container was again depressurized, soaked, and cycled 100 cycles to complete a total of 200 cycles.
9. The final repressurization and permeation test was then performed.
10. The container was slowly depressurized to atmospheric pressure.
11. The container was visually inspected with a borescope for any liner damage.
12. The two high-temperature test containers were sectioned for further inspection.

The first 50 cycles were intended to simulate 10 percent of the 500 pressure cycles in the SAE TIR J2579 Expected-Service Performance Verification Test in Figure 2. The first 100 cycles simulate 10 percent of a 1,000 cycle test, and the 200 cycles simulate more than 10 percent of a container lifetime of 1,833 cycles.

The incoming hydrogen was cooled to –35°C or below, as specified in SAE TIR J2579.

During high-temperature cycling, the depressurization rate was kept constant to mimic fuel cell vehicle operation. However, the fueling of the container in the +65°C environment was slowed considerably to avoid exceeding the upper container gas temperature limit of +85°C. Similarly, the defueling rate was restricted in the –40°C environment to avoid greatly exceeding the lower gas temperature limit of –40°C.

3.1.3 Test Containers

Four Type 4 70-MPa containers were tested according to the above procedure. The containers were identical in construction and were approximately 90L capacity. Each container was instrumented with an internal gas temperature sensor, a container surface temperature sensor, a test plug temperature sensor [to evaluate thermally activated pressure relief device (TPRD) temperature], and a pressure sensor. The containers were purchased by Powertech from Lincoln Composites (Lincoln, NE).

3.1.4 Results and Assessment

The test results are summarized in Table 2. The baseline permeation values for the four containers were very low, as expected. Permeation rates grew after the first 50 cycles. Only the container cycled at +65°C had a final permeation rate above the SAE TIR 2579 150 cc/hr allowable permeation criterion. Both containers that were pneumatically cycled at high temperature had visible damage; those pneumatically cycled at low temperature did not.

Table 2. Measured permeation rates from the containers before, during, and after the pressure cycling and Post-test Inspection Observations

Temperature	Permeation Rate (cc/hr)				Visual Inspection
	baseline (before cycling)	after 50 cycles	after 100 cycles	after 200 cycles	
+65°C	1.7	201	302	279	The liner pulled away from the end boss at the dome, with significantly more separation on the +65 °C container. The liner blistered.
+50°C	8.5	109	133	123	
-25°C	1.4	87	83	100	No damage was evident during the visual inspection.
-40°C	4.6	78	84	108	

3.1.4.1 High-Temperature Pneumatic Cycling Results

To illustrate the fluctuations of pressure and temperature during cycling, the temperatures and pressure during first few cycles of the test at +50°C test are shown in Figure 8.

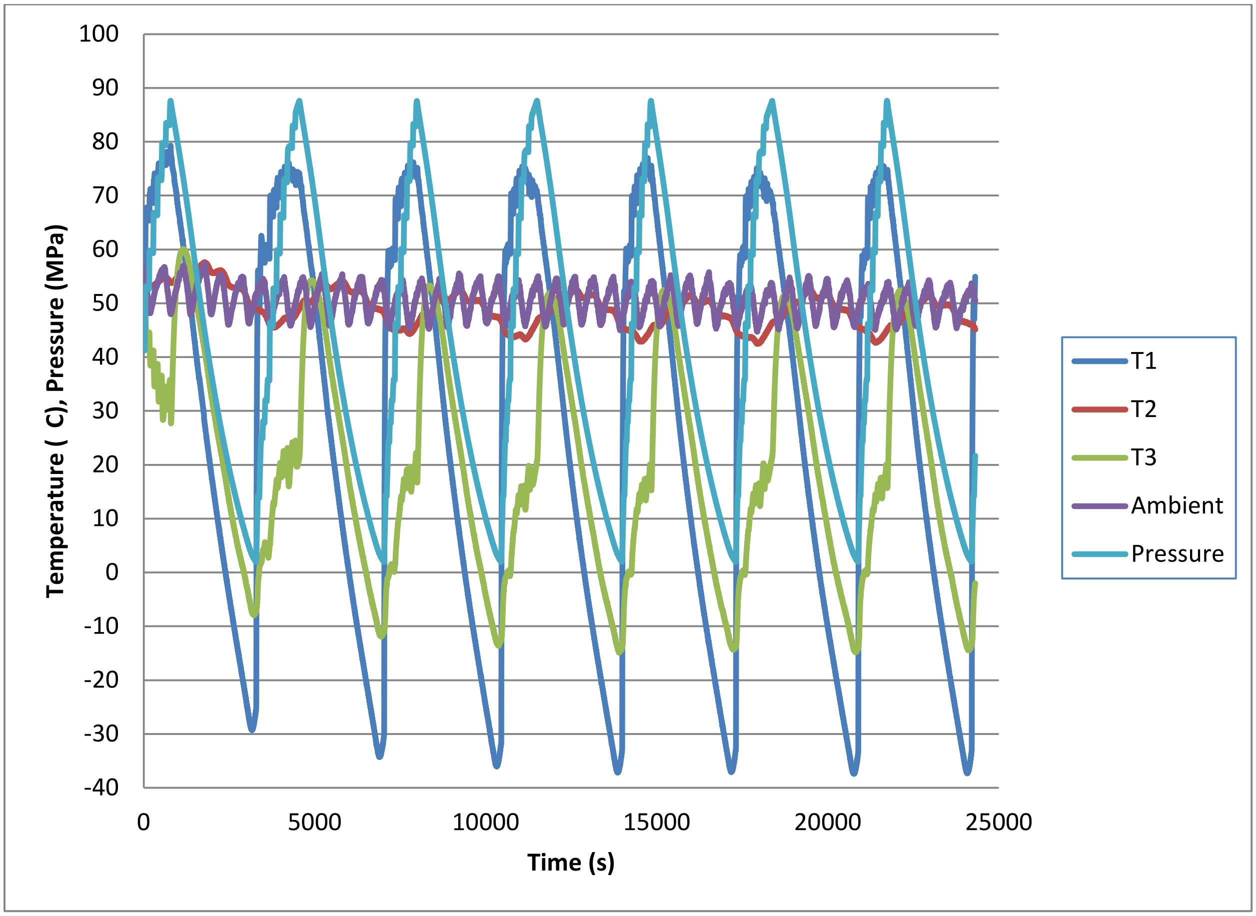


Figure 8. Temperature and pressure data from 7 of the first 50 test cycles at +50°C. T1 was gas temperature in the container, T2 was the container surface temperature, and T3 was temperature measured at the simulated TPRD.

The permeation rates from both containers following high-temperature pneumatic cycling were dramatically increased above the baseline rates. The container that was pneumatically cycled at +50°C showed a permeation rate of 122.85 cc/hr following the cycling, which is much greater than the average measured permeation rate, although still below the allowable rate of 150 cc/hr. The container pneumatically cycled at +65°C showed a permeation rate of 278.89 cc/hr, which is 70 times the average measured permeation rate and well above the allowable rate.

No damage was visible in high-temperature test containers when their liners were inspected using a borescope. The containers were then sectioned and the liners were visually inspected. This inspection showed that there was minor separation of the liner-boss interface on both high temperature test containers at the rear dome (opposite the gas inlet/outlet), with significantly more separation on the +65°C container. These gaps may be one of the causes of the observed increases in permeation. Liner bubbling was present in both containers, with more bubbling occurring on the rear dome section. Prior investigation experience suggests that the bubbling likely did not contribute to the observed increases in permeation. A normal liner end boss interface is shown in Figure 9, and the interiors of the cycled containers are shown in Figure 10, Figure 11, and Figure 12.

Figure 9. Normal interface between a liner end and the boss.

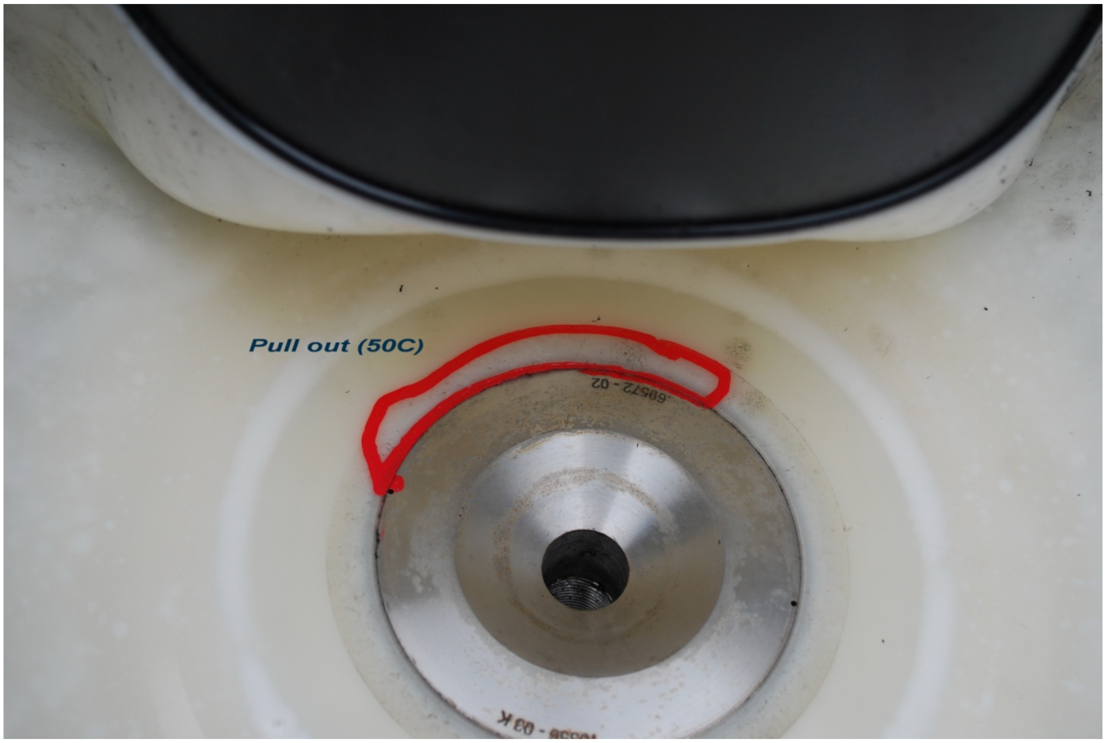


Figure 10. Interface between the liner and the boss of the container that was cycled at +50^{o}C. The red shows where the liner pulled away from the boss.

Figure 11. Red circles show blisters in the liner of the container that was cycled at +50°C.

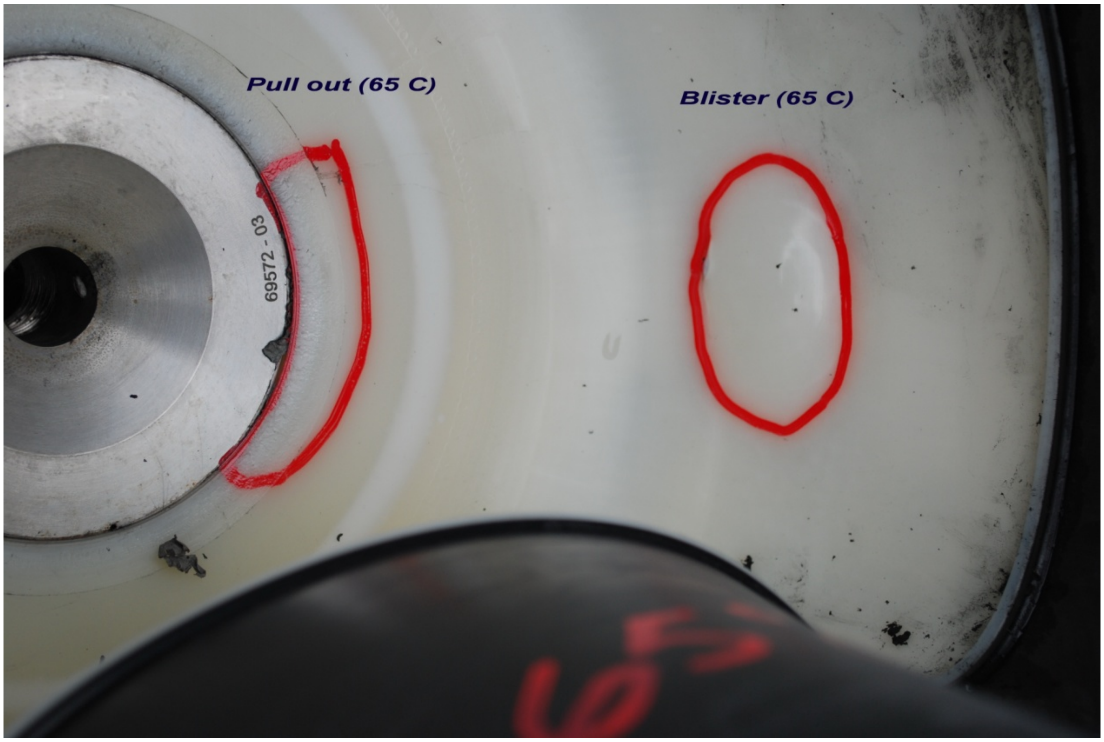


Figure 12. +65°C Liner-End Boss Interface.

The purpose of this task was to determine if there were differences in container performance when cycled at +50°C versus +65°C. The results demonstrated that damage occurred in both cases and that the damage was greater at +65°C, due to greater end-boss pull away and greater permeation. This experiment demonstrated that exposures to either of these temperatures may be damaging and that the higher temperature may be more damaging. This does not suggest that the containers are not suitable for hydrogen service. The results suggest that more work is needed to (1) determine the actual maximum temperature conditions in service and their frequency and duration over the life of a container and (2) determine a high temperature for this test that is representative of service conditions, ensuring a sufficient level of container durability throughout its life-cycle, without being excessive.

3.1.4.2 Low-Temperature Pneumatic Cycling Results

The test results show that permeation rates from both containers after low-temperature pressure cycling were increased over the baseline rates, although less than those cycled at the high temperatures. The container cycled at −40°C showed a permeation rate of 108.25 cc/hr following the cycling, which is 35 times the average baseline permeation rate, but below the allowable rate of 150 cc/hr. The container cycled at −25°C showed a permeation level of 100.25 cc/hr, which is approximately the same as that observed in the −40°C cold temperature container. The limitation on depressurization rate necessary to limit gas temperature to approximately −40°C resulted in the internal gas temperature being almost identical in the two containers throughout the testing. This likely contributed to the similarity in permeation results.

The data suggest that pneumatic cycling to 200 cycles at either of the two low temperatures did not appear to induce damage to the containers. No damage was evident during the visual inspection, suggesting that observed increases in permeation rate were likely caused by minor influences of cold temperature cycling on the seals and the liner. The increase in permeation rate was likely more influenced by prolonged exposure to hydrogen and pressure cycling in general, and possible permeation/leakage from tanks seals/valves.

Hydrogen vehicle fuel system components are generally rated to operate at temperatures down to −40°C. Maintaining a temperature of −40°C in the container during pneumatic cycling necessitated that the valves, seals, regulators and other test equipment components operate at temperatures well below −40°C. While the container itself operated well, the test equipment components did not, creating substantial difficulty in conducting efficient, repeatable testing.

Three aspects of the results of this subtask suggest that −25°C could be considered as a suitable low temperature for pneumatic cycling in the SAE TIR J2579 Expected-Service Performance Verification Test. First, as noted previously, OEMs indicate that it is unlikely that fuel cell vehicles will be expected to operate fully at ambient temperatures down to −40°C. (Full vehicle operation is supported only down to temperatures of -25°C. A reduced “limp home” mode performance may be supported at −40°C.) Secondly, no damage was observed in the liners from tests conducted at −25°C or from tests conducted at −40°C. Lastly, pneumatic cycling at −40°C is extraordinarily challenging and expensive to perform because the test apparatus must operate at temperatures well below that in order to maintain an ambient temperature of −40°C.

3.2 Task 2b – Assessment of High-Pressure Hydrogen Static Hold Duration

3.2.1 Hold Duration Evaluation

The goal of Task 2b was to determine a minimum time required to (1) detect issues in hydrogen compatibility, such as liner cracking or degradation, and (2) ensure that steady-state permeation results are comparable among test labs, container sizes, and container types. This assessment would help to reduce the total time required to perform the test sequence.

To perform this assessment, this test was conducted in accordance with the sequence shown in Figure 2, such that containers were cycled pneumatically 250 times, held at high static pressure, cycled another 250 cycles, then held again at high static pressure. Only the duration of the hold periods was varied, to assess the influence of hold duration on the container durability.

The temperature during high-pressure hydrogen static hold was +55°C as specified in the TIR. The low temperature during pneumatic cycling was selected to be −25°C for reasons described in the previous section of this report. The high temperature during pneumatic cycling was selected to be +50°C currently specified in the TIR. This minimized the likelihood that cycling at high temperature would influence hold time results.

Because static pressure hold duration could affect both Type 3 and Type 4 containers, both types were evaluated.

3.2.2 Test Procedure

Two separate tests were performed, the first on two Type 4 containers and the second on a single Type 3 container.

Table 3. Test Matrix

Test Number	Container Construction	Number of Cycles	Temperature for Holds, °C	Duration of Holds, Hours
2b1-1	Type 4	500	55	30
2b1-2	Type 4	500	55	100
2b2-1	Type 3	500	55	500

The steps of the experimental procedure were

1. The container was filled to service pressure of 70 MPa at a settled temperature of +15°C.
2. The container was placed in a permeation chamber and held until a steady state permeation rate was reached. This pre-cycling permeation rate was captured as the baseline value for that specific container.
3. The container was pneumatically pressure cycled 250 times, in the sequence illustrated in Figure 2. The low and high temperatures were −25°C and +50°C, respectively.

4. The permeation rate was measured at ambient temperature.
5. The container was pressurized to 125 percent of service pressure at +55°C and held for the time specified in Table 5. The permeation rate was measured during this time.
6. Pressure cycling was resumed for another 250 cycles, for a total of 500 cycles, again following the sequence illustrated in Figure 2. A second permeation test followed the 500th cycle.
7. The container was again pressurized to 125 percent of service pressure at +55°C and held for the duration specified in Table 5.
8. Following the final permeation test, the containers were slowly vented to atmosphere and the liners were visually inspected.

The hydrogen entering the container during pressure cycling was cooled to −35°C or below, as specified in the TIR.

The change in permeation values for each container throughout the course of the cycling was used to identify any degradation in leak-tightness of the container. The internal visual examination at the conclusion of the test sequence was performed to detect if there was any significant change in the liner condition, such as blistering or cracking.

3.2.3 Test Containers

This experiment required three 70-MPa hydrogen containers, two Type 4 and one Type 3. The two Type 4 containers were approximately 90 L capacity, and the single Type 3 container was approximately 60 L capacity. Each container was instrumented with an internal gas temperature sensor, a container surface temperature sensor, a test plug temperature sensor (to evaluate TPRD temperature) and a pressure sensor. The Type 3 containers were purchased by Powertech from Dynetek Industries and the Type 4 containers were purchased from Lincoln.

3.2.4 Results and Assessment

The test results are summarized in Table 6. Both Type 4 containers and the one Type 3 container were cycled a total of 500 cycles. There were two high-pressure hydrogen static holds, one after 250 pneumatic cycles and the other after 500 pneumatic cycles.

The baseline permeation values for the two Type 4 containers were very low, as expected. The permeation rate for the Type 3 container was barely measurable, likely resulting from permeation from the O-ring seals and the valve.

Table 4. Permeation rates from the containers before, during, and after the pressure cycling.

Container — hold duration	Steady-State Permeation Rate (cc/hr)				
	baseline (before cycling)	after 250 cycles		after 500 cycles	
		ambient temperature	+55°C hold	ambient temperature	+55°C hold
Type 4 — 30 hours	10.3	11	102.3	13.7	109.1
Type 4 — 100 hours	3.9	9.1	106.2	14.3	118.7
Type 3 — 500 hours	0.8	1.1	2.6	1.3	2.4

3.2.4.1 Type 4 Container Results

The permeation rates of the two Type 4 containers during the first-high pressure hydrogen static hold were increased from the baseline value, but this increase is attributed to the increased temperature during the permeation measurements. The ambient permeation reading of each container was taken prior to heating to +55°C and the values were similar to those from the baseline, showing no damage to the container. Notably, the readings were consistent between both containers, showing no change between the 30-hour hold and the 100-hour hold. Steady-state seemed to be achieved as soon as temperature equalization occurred and the hold period began.

Similar results were achieved during the second high-pressure hydrogen static hold. A very small increase in permeation values was noted in both containers, but was well below the allowable limit of 150 cc/hr and remained constant throughout the hold period. The increase was less than 2% of the allowable permeation rate.

The liners of both containers were inspected after testing and no damage was visible. Both had evidence of liner buckling, likely caused during final depressurization for inspection. Images of both containers are shown in Figure 13 and Figure 14. No liner-boss separation was visible.

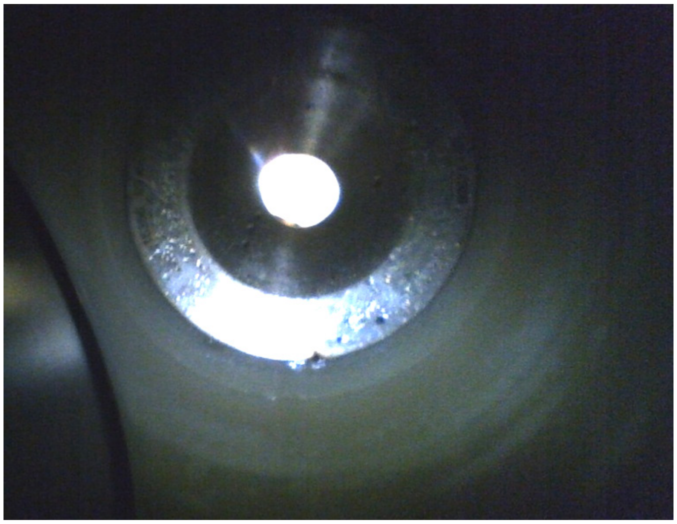


Figure 13. Container 1 (>30hr Hold Time) Following 500 Cycles and Two Time-at-Pressure Exposures.

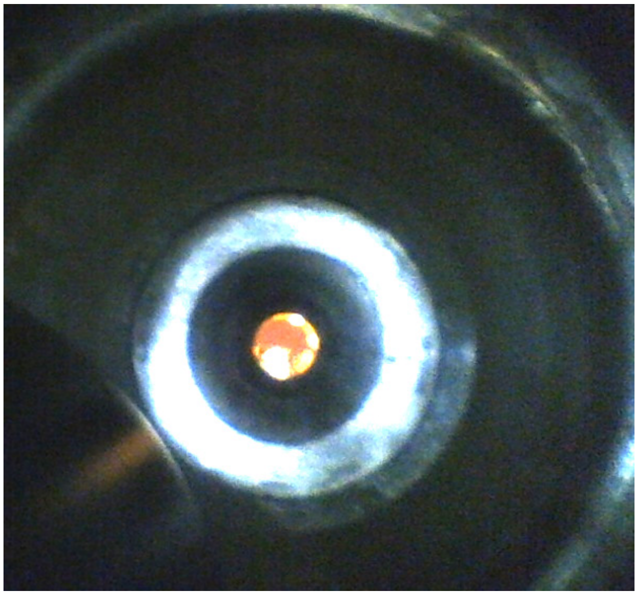


Figure 14. Container 2 (>100hr Hold Time) Following 500 Cycles and Two Time-at-Pressure Exposures.

These results suggest that the minimum 30-hour hold requirement for the high-pressure hydrogen static hold appears sufficient for evaluating Type 4 container performance, as long as the containers under examination have achieved steady-state permeation during previous cycling. This ensures that the materials are completely saturated and no longer under flux from hydrogen migration.

When this project began, the published draft of SAE TIR J2579 did not define steady-state permeation nor provide guidance for its determination. The SAE Fuel Cell Safety Working Group is adding language to the draft to define steady-state permeation to ensure consistency across different test facilities and measurement methods. Steady-state permeation is currently defined as three consecutive permeation readings within ±10 percent.

3.2.4.2 Type 3 Container Results

The permeation rate in the Type 3 container during the first high-pressure hydrogen static hold showed no increase from the baseline value. Steady-state appeared to be achieved as soon as temperature equalization occurred and the hold period began. The hold time was extended to 500 hours and no sign of increased permeation or liner damage was evident. The same results were achieved during the second high-pressure hydrogen static hold. Again the hold was extended to 500 hours with no change in permeation rates. The liner of the container was inspected following the test. There was no evidence of any deterioration, cracking, pitting, or damage.

The Type 3 container in the present test had a liner compatible with hydrogen, and 500-hour exposure to hydrogen at high pressure and temperature had no effect on its performance or structure. The high-pressure static hold time results of Task 2b suggest that a minimum 30-hour hold requirement was sufficient for assessing Type 3 container durability, when used in conjunction with other hydrogen compatibility test assessments.

3.3 Task 2c – Total Pneumatic Pressure Cycle Count

The goal of Task 2c was to determine the minimum number of pneumatic test cycles necessary to assess the durability of containers for their intended maximum lifetime of 1,833 fueling cycles. This assessment was made by cycling separate containers to 500, 1,000, and 1,833 cycles and assessing degradation. If no difference was found in permeation values, and no liner degradation or materials compatibility issues was discovered, then the data would suggest that fewer cycles are sufficient to assess life cycle durability. However, any change in container performance at test cycles greater than 500 and less than 1,833, evidenced by liner degradation or a change in container leak-tightness, may suggest that more cycling is necessary to assess safety performance.

Because pneumatic pressure cycles could affect the performance of both Type 3 and Type 4 containers, both types were evaluated.

3.3.1 Test Procedure

Three separate tests were performed, each on one Type 3 and one Type 4 container.

Table 5. Test Matrix

Test	Container Ambient Temperature During Cycling	Hold Time	Total Cycle Number
2c-1	−25°C and +50°C	100 hours	500
2c-2	−25°C and +50°C for first 500 cycles, then room temperature	100 hours	1,000
2c-3	−25°C and +50°C for first 500 cycles, then room temperature	100 hours	1,833

The steps of the experimental procedure were

1. The container was filled to 70 MPa at a settled temperature of +15°C.
2. The container was placed in a permeation chamber and held until a steady-state permeation rate was reached. This pre-cycling permeation rate was the baseline value.
3. The container was pressure cycled 250 times, in the sequence illustrated in Figure 2. The low and high extreme temperatures were −25°C and +50°C.
4. The permeation rate was measured at ambient temperature.
5. The container was taken to the hold temperature of +55°C and held for the time specified in Table 5.
6. Pressure cycling was resumed for another 250 cycles, for a total of 500 cycles, again following the temperatures of Figure 2.
7. The permeation rate was measured at ambient temperature.
8. The container was taken to the hold temperature of +55°C and held for the time specified in Table 5.
9. If necessary, additional ambient pressure cycles were performed.
10. The permeation rate was measured at ambient temperature.
11. Following the final permeation test, the containers were slowly vented to atmosphere and the liners were visually inspected.

The hydrogen entering the container during pressure cycling was cooled to −35°C or below, as specified in the standard.

Change in permeation values for each container was measured throughout the course of the cycling to identify any degradation in leak-tightness of the container. Internal visual examination was performed at the conclusion of the test sequence to detect any significant change in the liner condition, such as blistering or cracking.

3.3.2 Test Containers

This experiment required six 70-MPa hydrogen containers. The three Type 4 containers were approximately 90L capacity, and the three Type 3 containers were approximately 60L capacity. Each container was instrumented with an internal gas temperature sensor, a container surface temperature sensor, a test plug temperature sensor (to evaluate TPRD temperature), and a pressure sensor. Type 3 containers were purchased by Powertech from Dynetech Industries and Type 4 containers were purchased from Lincoln Composites.

3.3.3 Results and Assessment

The test results are summarized in Table 6. The Type 4 containers and the Type 3 containers were cycled as specified in Table 6 for the number of prescribed pressure cycles. The baseline permeation values for the Type 4 containers were very low, as expected. The barely measureable permeation rates recorded for the Type 3 containers likely resulted from permeation from the O-ring seals and the valve.

Table 6. Ambient temperature permeation leakage rates from the containers before, during, and after the pressure cycling.

	Steady-State Ambient Temperature Permeation Level (cc/hr)			
Test	Baseline	After 250 cycles	After 500 cycles	Final
Task 2c-1 Type 3	0.6	1.0	1.8	N/A
Task 2c-1 Type 4	10.1	17.2	16.7	N/A
Task 2c-2 Type 3	0.5	0.9	1.7	(1,000 cycles) 2.1
Task 2c-2 Type 4	8.8	13.4	15.4	14.2
Task 2c-3 Type 3	0.5	1.2	2.2	(1,833 cycles) 2.1
Task 2c-3 Type 4	9.2	14.1	18.2	18.1

3.3.3.1 Type 4 Container Results

The permeation rates of the Type 4 containers following the first 250 cycles were above the baseline value, but only by a small amount.

The same results were achieved following the second high-pressure static hold exposure. A very small increase in permeation values was noted in both containers, but was well below the allowable limit of 150 cc/hr and remained constant throughout the hold period. The small increase is attributed to the cycling and was less than 2 percent of the allowable permeation rate.

Permeation tests performed following the additional ambient cycles (up to 1,000 and 1,833) showed no increase in permeation rate with increased number of cycles. The rates remained well below the allowable limit.

The liners of all three Type 4 containers were inspected after testing and no difference between them was visible. All had evidence of liner buckling.

The results indicate that the liner performance was the same, whether cycling to 500, 1,000, or 1,833 cycles, suggesting that 500 pneumatic cycles is sufficient to demonstrate the performance of Type 4 containers. The results suggest that high or low temperatures could affect performance, but ambient temperature cycling appeared to have little effect. This in turn suggests that cycling should be performed at maximum and minimum temperatures. These results are based on limited test samples of the same construction and materials.

3.3.3.2 Type 3 Container Results

The permeation rate during the first measurement of the Type 3 containers showed no increase from the baseline value. This was true of the second reading and the final post-cycling measurement. There was no evidence of any deterioration. No cracking was observed and no obvious pitting or damage of any kind was detected.

Similar to the Type 4 container, the results indicate that the liner performance was the same, whether cycling to 500, 1,000, or 1,833 cycles, suggesting that 500 pneumatic cycles is sufficient to demonstrate the performance of Type 3 containers. While temperature effects appeared minimal, consistency would suggest that cycling of Type 3 containers should be performed at the same maximum and minimum temperatures as Type 4 containers. Again, these results are based on limited test samples of the same construction and materials.

4.0 SUMMARY OF CONCLUSIONS

The goal of this Task Order was to assess the test conditions used for evaluating life cycle durability of high-pressure hydrogen fuel containers. The objective of this effort was to assess the influence of key test conditions, namely high and low temperatures, high-pressure static hold time, and number of pneumatic cycles, on the likelihood of damage to high-pressure hydrogen fuel containers. The results of this Task Order provide data for NHTSA's assessment and consideration as it evaluates the safety performance of compressed hydrogen fuel systems and the need for enhancement of appropriate FMVSS. In addition, the results of this Task Order provide data to assist in resolving several open issues and concerns in the Expected-Service Performance Verification Test procedure in the SAE TIR 2579 for Fuel Systems in Fuel Cell and Other Hydrogen Vehicles [1].

SAE TIR J2579 has been based on industry discussion and limited testing results. Some of the acceptance criteria in the test sequence were openly debated without complete consensus. Consequently, a "reasonable" value has been inserted by the committee in the draft document until more data or a better rationale becomes available.

The high-temperature pneumatic cycling results of Task 2a demonstrated that pneumatic cycling conducted at +50°C and cycling conducted at +65°C both caused damage to Type 4 containers in the form of increased permeation and liner pull-away from the boss. The damage was greater at +65°C, in the form of more boss pull-away and greater permeation. This testing demonstrated that exposures to either of these temperatures may be damaging and that the higher temperature may be more damaging. This does not suggest that the containers are not suitable for hydrogen service. The results suggest that more work is needed to (1) determine the actual maximum temperature conditions in service and their frequency and duration over the life of a container and (2) determine a high temperature for this test representative of service conditions that ensures a sufficient level of container durability throughout its life cycle, without being excessive.

The low-temperature pneumatic cycling results of Task 2a demonstrated little difference in container performance when tested at −25°C and at −40°C. Three aspects of the results of this subtask suggest that −25°C could be considered as a suitable low temperature for pneumatic cycling in the SAE TIR J2579 Expected-Service Performance Verification Test. First, OEMs indicate that it is unlikely that fuel cell vehicles will be expected to operate fully at ambient temperatures down to −40°C. (Full vehicle operation is supported only down to temperatures of −25°C. A reduced "limp home" mode performance may be supported at −40°C.) Secondly, no damage was observed in the liners from tests conducted at −25°C or from tests conducted at −40°C. Lastly, pneumatic cycling at −40°C is extraordinarily challenging and expensive to perform because the test apparatus must operate at temperatures well below in order to maintain an ambient test temperature of −40°C.

The high-pressure static hold time results of Task 2b suggest that a minimum 30-hour hold requirement after cycling appears sufficient for achieving steady-state permeation and evaluating Type 4 container durability, as long as the containers have achieved steady-state permeation in previous cycling. No damage was observed in Type 4 containers subjected to high-pressure static hold periods of 30, 100, and 500 hours. The SAE Fuel Cell Safety Working Group is adding language to the draft to define steady-state permeation to ensure consistency across different test facilities and measurement methods. Steady-state permeation is currently defined as three consecutive permeation readings within ±10 percent.

The high-pressure static hold time results of Task 2b suggest that a minimum 30-hour hold requirement was sufficient for assessing Type 3 container durability, when used in conjunction with other hydrogen compatibility test assessments. The Type 3 container in the present test had a liner compatible with hydrogen, and 500-hour exposure to hydrogen at high pressure and temperature had no effect on its performance or structure.

The pneumatic cycle count results of Task 2c suggest that a minimum of 500 cycles is sufficient to assess the durability of Type 4 and Type 3 containers. No damage was found and no difference was observed in durability performance between cylinders subjected to 500, 1,000, and 1833 cycles.

5.0 REFERENCES

1. Society of Automotive Engineers. (2009). , “Technical Information Report for Fuel Systems in Fuel Cell and Other Hydrogen Vehicles.(SAE TIR J2579 – JAN2009). Warrendale, PA: SAE International. .

2. Society of Automotive Engineers. (2009).Technical Information Report for Fuel Systems in Fuel Cell and Other Hydrogen Vehicles. (SAE TIR J2579 – JAN2008). Warrendale, PA: SAE International

3. Gambone, L., & Stephens, D. (2008, November 19,, in progress) Work Plan for Cumulative Fuel System Life Cycle and Durability Testing. (Contract No. DTNH22-08-D-00080 Task Order 2, in progress). Washington, DC: National Highway Traffic Safety Administration.

4. Society of Automotive Engineers. (2009). Technical Information Report for Fuel Systems in Fuel Cell and Other Hydrogen Vehicles. (SAE TIR J2579, Revised Proposed Draft Nov. 2009). Warrendale, PA: SAE International

5. Society of Automotive Engineers. (2010). Fueling Protocols for Light Duty Gaseous Hydrogen Surface Vehicles. (SAE J2601 – MAR2010) Warrendale, PA: SAE International

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