

# DRAFT

## OICA Proposal for UN-GTR HTC V

– for consideration at SGS-8

The following text pertains to PART B, Section 5.1

Revisions to proposal provided to SGS-5 are marked in gray

### 5.1 Hydrogen Storage system:

This section specifies the requirements for the integrity of the compressed hydrogen storage system. The hydrogen storage system consists of the high pressure storage container(s) and closures of openings into the high pressure storage container(s). Closures include the temperature-activated pressure relief device(s) (TPRD), check valve(s), shut-off valve(s) and all components, fittings and fuel lines between the storage container(s) and the closure device(s) that isolate high pressure hydrogen from the remainder of the fuel system and the environment. A check valve prevents reverse flow in the vehicle fill line. A shut-off valve between the storage container and the vehicle fuel system defaults to the closed position when unpowered.

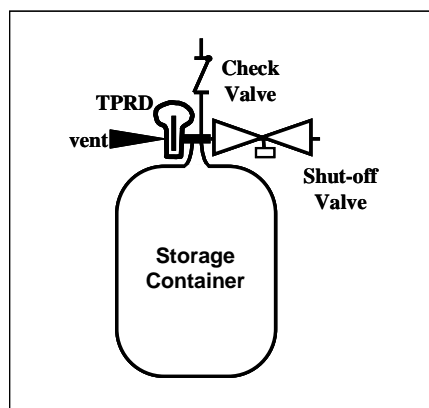


Figure 5.1.1 Generic Hydrogen Storage System

Qualification requirements for on-road service include:

- 5.1.1 Material Requirements
- 5.1.2 Storage System Performance Test Requirements
- 5.1.3 Storage System Production Requirements

The test elements within these performance requirements are summarized in Table 5.1.

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**Table 5.1.1**  
**Overview of Performance Qualification Test Requirements**

<b>5.1.1 Material Test Requirements</b>
<b>5.1.2 Storage System Performance Test Requirements</b>
<b>5.1.2.1 Verification Tests for Baseline Metrics (parallel hydraulic tests)</b> 5.1.2.1.1 Baseline Initial Burst Pressure 5.1.2.1.2 Baseline Initial Pressure Cycle Life
<b>5.1.2.2 Verification Test for Performance Durability (sequential hydraulic tests)</b> 5.1.2.2.1 Proof Pressure Test 5.1.2.2.2 Drop (Impact) Test 5.1.2.2.3 Surface Damage Test 5.1.2.2.4 Chemical Exposure and Ambient Temperature Pressure Cycling Test 5.1.2.2.5 High Temperature Static Pressure Test 5.1.2.2.6 Extreme Temperature Pressure Cycling 5.1.2.2.7 Residual Proof Pressure Test 5.1.2.2.8 Residual Strength Burst Test
<b>5.1.2.3 Verification Test for Expected On-road Performance (sequential hydraulic and pneumatic tests)</b> 5.1.2.3.1 Proof Pressure Test (hydraulic) 5.1.2.3.2 Ambient and Extreme Temperature Gas Pressure Cycling Test (pneumatic) 5.1.2.3.3 Extreme Temperature Static Gas Pressure Leak/Permeation Test (pneumatic) 5.1.2.3.4 Residual Proof Pressure Test (hydraulic) 5.1.2.3.5 Residual Strength Burst Test (Hydraulic)
<b>5.1.2.4 Verification Test for Fail-Safe Performance</b> 5.1.2.4.1 Fire Test 5.1.2.4.2 Penetration Test

## 5.1.1 Material Test Requirements

Materials used in storage systems (as illustrated in Figure 5.1.1) must comply with requirements of 6.4.1. Manufacturers must maintain relevant information relevant on suitability of materials for the system design.

## 5.1.2 Storage System Performance Test Requirements

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The hydrogen storage system will be qualified to the performance test requirements specified in 5.1.2. The specifications for test procedures in 5.1.2 are provided in Section 6.4.2.

The storage system does not have to be re-qualified if the subsystem components are exchanged for components with comparable function, fittings, and dimensions, and meet comparable component performance qualification specifications. A change in the TPRD hardware, its position of installation and/or venting lines requires re-qualification with 5.1.2.4.1, the fire test.

These criteria apply to qualification of new storage systems for original use in vehicles

## 5.1.2.1 Verification Tests for Baseline Metrics

5.1.2.1.1 Baseline Initial Burst Pressure Test: The test is designed to provide a baseline metric for assurance that vessels manufactured in the future correspond to the vessels used to qualify for on-road service. The manufacturer will determine the nominal (average) burst pressure of new storage containers (tanks),  $BP_0$ , and will document it with measurements and statistical analyses that establish the average burst pressure of production units and establish that the burst pressure of production units is controlled to  $> 90\% BP_0$  and  $> 180\%$  of NWP.

$BP_0$  will be verified in design qualification testing by hydraulically pressurizing until burst three (3) randomly selected new vessels from a sample of at least ten (10) vessels (6.4.2.1 test procedure). All vessels tested must have burst pressures  $\geq 180\%$  NWP and within 10% of  $BP_0$ ; if not,  $BP_0$  is reset to the highest burst pressure measured that is greater than the original  $BP_0$  supplied by the manufacturer. The resultant  $BP_0$  is used to satisfy requirements of 5.1.2.2.8 and 5.1.2.3.5 for design qualification (performance verification) and also to satisfy requirements of 5.1.3 (Production Batch quality control).

Note: 5.1.2.1.1 is a draft proposal, not a fully considered and consensed recommendation from OICA. 5.1.2.1.1 is subject to revision; it remains under discussion and re-consideration.

5.1.2.1.2 Baseline Pressure Cycle Life (Leak Before Break) Test. The test is designed to provide assurance that vessels manufactured in the future correspond to the vessels used to qualify for on-road service. The manufacturer will determine the nominal (average) hydraulic pressure cycle life of new containment vessels,  $PCL_0$ , and will document it with measurements and statistical analyses used for that determination; alternatively, the manufacturer may simply specify  $PCL_0$  to be 2 times the number of cycles required for 5.1.2.2.

$PCL_0$  will be verified by randomly selecting three (3) vessels from a sample of at least ten (10) vessels and hydraulically pressure cycling them for 11,000 cycles (2 times the minimum number of cycles required for all production units) or until leak occurs (6.4.2.2 test procedure). If no leak occurs within 11,000 cycles, then  $PCL_0$  is equated to 11,000. If all 3 vessels do not have a pressure cycle life within 25% of  $PCL_0$ ,  $PCL_0$  is set to the highest cycle life measured that is greater than the original  $PCL_0$  supplied by the manufacturer. (6.4.2.2 test procedure.)  $PCL_0$  is used to satisfy requirements of 5.2.3.2 (Production Batch quality control).

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Note: 5.1.2.1.2 is a draft proposal, not a fully considered and consensed recommendation from OICA. 5.1.2.1.2 is subject to revision; it remains under discussion and re-consideration.

## 5.1.2.2 Verification Test for Performance Durability (Hydraulic sequential tests)

All production hydrogen storage systems must have the capability to perform as specified during the following sequence of tests, which are illustrated in Figure 5.1.2.2 . At least one system must be tested to demonstrate the performance capability.

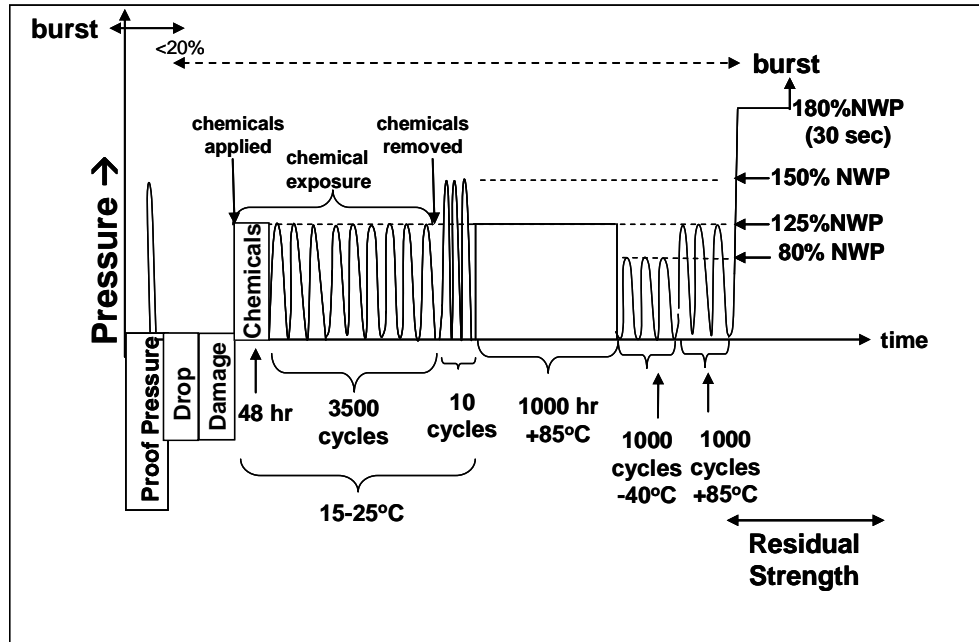


Figure 5.1.2.2 Verification Test for Performance Durability

- 5.1.2.2.1 Proof Pressure Test. A storage container will be pressurized to 150%NWP. (6.4.2.3 test procedure)
- 5.1.2.2.2 Drop (Impact) Test. The storage container(s) will be dropped at several impact angles (6.4.2.4 test procedure)
- 5.1.2.2.3 Surface Damage Test. The storage container will be subjected to specified surface damage (6.4.2.5 test procedure)
- 5.1.2.2.4 Chemical Exposure and Ambient-Temperature Pressure Cycling Tests. The storage container will be exposed to chemicals typical of worst-case on-road exposures. After 48 hours of exposure, the container will undergo 3500 pressure cycles to 125% NWP at ambient temperature (15C-25C) with the last 10 cycles done to 150% NWP. Chemical exposure will be maintained throughout the 125% NWP pressure cycles. (6.4.2.6 and 6.4.2.2 test procedures); chemical exposure will be discontinued after the 3500 cycles.

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- 5.1.2.2.5 High-Temperature Static Pressure Test. The storage system will be subjected to 125% NWP and +85°C for 1000 hr. (6.4.2.7 test procedure)
- 5.1.2.2.6 Extreme-Temperature Pressure Cycling Test. The storage container will undergo 1000 pressure cycles to 125% NWP at 85°C and 1000 pressure cycles to 80% NWP at -40°C. (6.4.2.2 test procedure)
- 5.1.2.2.7 Residual Proof Pressure Test. The storage container will under a proof pressure test to 180% NWP. (6.4.2.3 test procedure)
- 5.1.2.2.8 Residual Burst Strength Test . The storage container will undergo a hydraulic burst test to verify that the burst pressure is within 20% of the baseline burst pressure determined in 5.1.2.2.1. (6.4.2.1 test procedure)

### 5.1.2.3 Verification Test for Expected On-road Performance (Pneumatic and hydraulic sequential tests)

All production hydrogen storage systems must have the capability to perform as specified during the following sequence of tests, which are illustrated in Figure 5.1.2.3 . At least one system must be tested to demonstrate the performance capability.

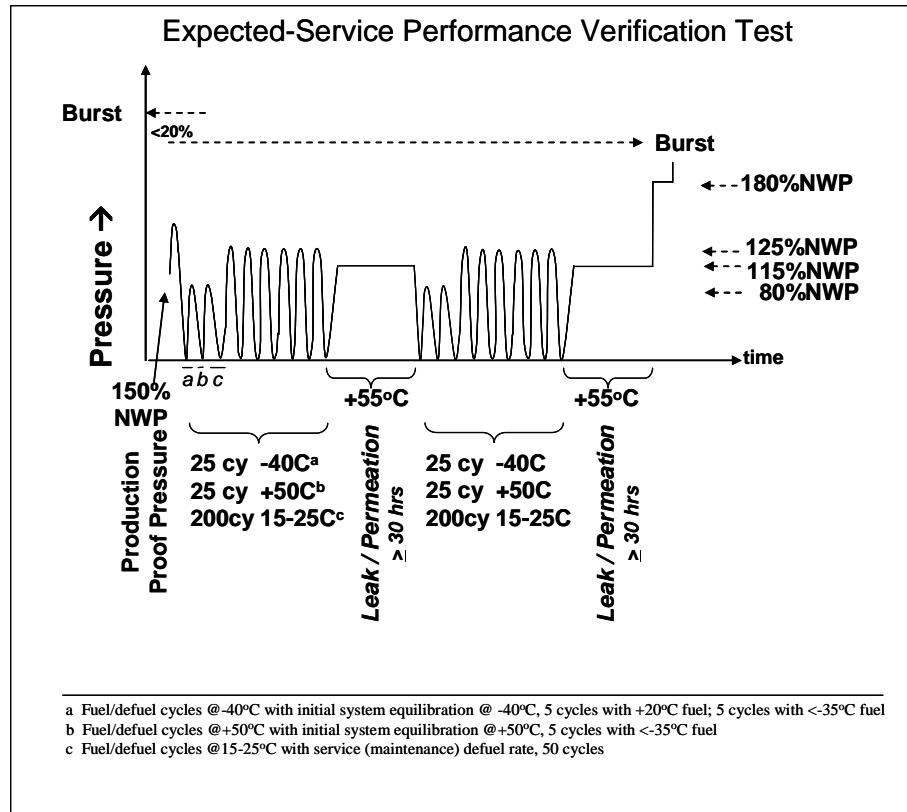


Figure 5.1.2.3 Verification Test for Expected On-Road Performance (pneumatic and hydraulic sequential tests)

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- 5.1.2.3.1 Proof Pressure Test: A system will be pressurized to 150%NWP. (6.4.2.2 test procedure)
- 5.1.2.3.2 Fueling Performance Verification Test: Extreme and Ambient Temperature Gas Pressure Cycling Test (pneumatic). The system will be pressure cycled (repeatedly filled and defueled to <2MPa) using hydrogen gas for 500 cycles. Half of the cycles will be performed before exposure to static pressure (5.1.2.3.3) and half of the cycles will be conducted after the initial exposure static pressure (5.1.2.3.3). In each case, 10% of cycles will be at 50C and 95% relative humidity and cycled to 125%NWP, 10% at -40C and cycled to 80%NWP, and the remainder at ambient temperature (15C-25C) and cycled to 125%NWP. The hydrogen gas fuel temperature will be <-35C. Five of the cycles will be performed after temperature equilibration at 50C and 95% relative humidity, and five cycles after equilibration at -40C; an additional five cycles will be performed with >20C fuel after ambient-temperature equilibration at -40C. Fifty of the cycles will be performed using the maintenance defueling rate. (6.4.2.8 test procedure)
- 5.1.2.3.3 Parking Performance Verification Test: Extreme Temperature Gas Permeation/Leak Test (pneumatic). The system will be held at 55C and 115%NWP with hydrogen gas until steady-state permeation or 30 hours, whichever is longer. The test will be performed after 250 pressure cycles are conducted in 5.1.2.3.2 and again at the completion of 5.1.2.3.2. The maximum allowable discharge from the compressed hydrogen storage system is 150 ml/min for standard passenger vehicles. (6.4.2.9 test procedure) [The maximum allowable discharge for systems in larger vehicles is  $R * 150 \text{ Ncc/min}$  where  $R = (V_{\text{width}}+1) * (V_{\text{height}}+0.5) * (V_{\text{length}}+1) / 30.4$  and  $V_{\text{width}}$ ,  $V_{\text{height}}$ ,  $V_{\text{length}}$  are the vehicle width, height, length (m), respectively.]
- If the measured permeation rate is greater than 0.005 mg/sec (3.6 cc/min), then a localized leak test shall be performed to ensure no point of localized external leakage is greater than 0.005 mg/sec (3.6 cc/min). (6.4.2.10 test procedure)
- 5.1.2.3.4 Residual Proof Pressure Test. The storage container will be pressurized to 180%NWP and held 30 seconds without burst. (6.4.2.3 test procedure)
- 5.1.2.3.5 Residual Strength Burst Test (hydraulic): The storage container will undergo a hydraulic burst test to verify that the burst pressure is  $\geq 80\% \text{ BP}_0$ , the baseline initial burst pressure determined in 5.1.2.1.1. (6.4.2.1 test procedure)

## 5.1.2.4 Verification Test for Fail-Safe Conditions

At least one system must be subjected to the following fail-safe conditions and demonstrate the absence of rupture.

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5.1.2.4.1 Fire Test. A hydrogen storage system will be pressurized to NWP and exposed to fire. A temperature-activated pressure relief device will release the contained gases in a controlled manner. (Test procedure TBD)

## 5.1.4 Markings

Tank label will contain the NWP, date of manufacture, and date of expected 25 year lifetime.

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#### The following text pertains to PART B, Section 6.4.1

#### Revisions are marked in gray

#### 6.4 Test Procedures for Hydrogen Storage

##### 6.4.1 Material Qualification

6.4.1.1 Plastic liner tensile test. For containers with plastic liners, two plastic liners shall be tested at -40°C in accordance with ISO 527-2. The tensile yield strength and ultimate elongation shall be within the manufacturer's specifications.

6.4.1.2 Plastic liner softening temperature test. For containers with plastic liners, the softening temperature of polymeric materials from finished liners shall be determined based on the A50 method in ISO 306. The softening temperature shall be  $\geq 100^{\circ}\text{C}$ .

6.4.1.3 Glass transition temperature test. For containers with composite wraps, the glass transition temperature of resin materials shall be determined in accordance with ASTM D3418. Test results shall be within the manufacturer's specifications.

6.4.1.4 Resin shear strength test. For containers with composite wraps, resin materials shall be tested on a sample coupon representative of the over-wrap in accordance with ASTM D2344. After boiling in water for 24 hours the minimum shear strength of the composite shall be 13.8MPa.

6.4.1.5 Coating test. For containers with external environmental coatings, coatings shall be evaluated as follows:

- a) adhesion strength based on ISO 4624; the coating shall exhibit an adhesion rating of 4.
- b) flexibility based on ASTM D522 Method B with a 12.7 mm mandrel at the specified thickness at -20°C; the coating shall exhibit no apparent cracks
- c) impact resistance in accordance with ASTM D2792. The coating at room temperature shall pass a forward impact test of 18 J.
- d) water exposure based on ASTM G154 using an exposure of 1000 hours. There shall be no evidence of blistering. The adhesion shall meet a rating of 3 when tested in accordance with ISO 4624.
- e) salt spray exposure in accordance with ASTM B117 using an exposure of 500 hours. There shall be no evidence of blistering. The adhesion shall meet a rating of 3 when tested in accordance with ASTM D3359.



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## 6.4.1.6 Metal hydrogen compatibility.

### a) Steel

In all applications where steel comes in contact with hydrogen, hydrogen compatibility should be demonstrated. Steels that meet requirements of 6.3 and 7.2.2 of ISO 9809-1:1999 are recognized as hydrogen compatible for low stress applications.

The following steels are recognized as suitable for high pressure hydrogen gas applications: SUS316L, AISI316L, AISI316 and DIN1.4435; all must have  $\geq 12\%$  nickel composition and  $\leq 0.1\%$  magnetic phases by volume. These steel applications may not include welds.

Other steels must be qualified for high pressure hydrogen gas applications by meeting the following performance-based test requirements:

TBD Test procedures are expected to be developed in 2010 by industry standards organizations and presented for inclusion in 6.4.1.6

### b) Aluminum

Aluminum alloys that meet the requirements of 6.1 – 6.2 of ISO 7866:1999 are recognized as hydrogen compatible for low stress applications.

The following aluminum alloys are recognized as suitable for use in contact with hydrogen in the hydrogen storage system, as defined in Figure 2, or in any other high-stress applications in contact with hydrogen: A6061-T6, A6061-T62, A6061-T651 and A6061-T6511. These aluminum applications may not include welds.

Other aluminum alloys must be qualified for high pressure hydrogen gas applications by meeting the following performance-base test requirements:

TBD Test procedures are expected to be developed in 2010 by industry standards organizations and presented for inclusion in 6.4.1.6.

## 6.4.2 Storage System Performance Test Requirements (Procedures for 5.1.2 Tests)

6.4.2.1 Burst Test (Hydraulic). The burst test shall be conducted at ambient temperature using a non-corrosive fluid. The rate of pressurization shall be  $\leq 1.4$  MPa/s for pressures higher than 150% of the nominal working pressure. If the rate exceeds 0.35 MPa/s at pressures higher than 150% NWP, then either the container shall be placed in series between the pressure source and the pressure measurement device, or the time at the pressure above a target burst pressure shall exceed 5 seconds. The burst pressure of the container shall be recorded.

6.4.2.2 Pressure Cycling Test (Hydraulic). The test shall be performed in accordance with the

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following procedure:

- a) Fill the container with a non-corrosive fluid.
- b) Stabilize the temperature of the container at the specified temperature and relative humidity at the start of testing; maintain the environment and container skin temperature at the specified temperature and relative humidity for the duration of the testing. The container interior temperature may vary from the environmental temperature during testing.
- c) Pressure cycle between  $<2$  MPa and the target pressure at a rate not exceeding 10 cycles per minute for the specified number of cycles.
- d) Maintain and monitor the temperature of the hydraulic fluid within the containerl at the specified temperature.

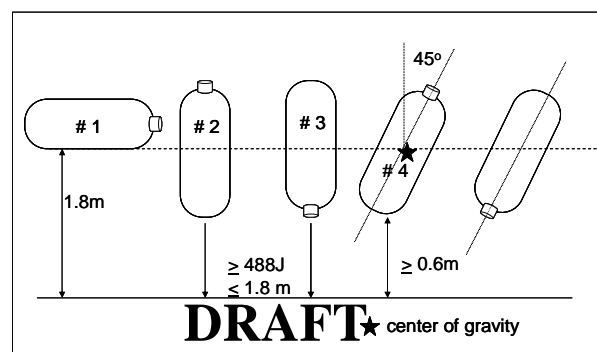
6.4.2.3 Proof Pressure Test (Hydraulic). The system should be pressurized smoothly and continually until the target test pressure level is reached and then held for at least 30 seconds. The component should not leak or suffer permanent deformation. All mechanical components should be functional after completion of the test.

6.4.2.4 Drop (Impact) Test (Unpressured). One or more storage containers will be drop tested at ambient temperature without internal pressurization or attached valves. All drop tests may be performed on one tank, or individual impacts on a maximum of 3 tanks. The surface onto which the tanks are dropped should be a smooth, horizontal concrete pad or similar flooring. The tank(s) should be tested in the following sequence:

- a) Drop once from a horizontal position with the bottom 1.8 m above the surface onto which it is dropped.
- b) Drop once onto each end of the tank from a vertical position with a potential energy of not less than 488J, but in no case should the height of the lower end be greater than 1.8 m.
- c) Drop once at a 45 ° angle, and then for non-symmetrical and non-cylindrical tanks rotate the tank through 90 ° along its longitudinal axis and drop again at 45 ° C with its center of gravity 1.8 m above the ground. However, if the bottom is closer to the ground than 0.6 m, the drop angle should be changed to maintain a minimum height of 0.6 m and a center of gravity of 1.8 m above the ground.

No attempt should be made to prevent the bouncing of tanks, but the tanks may be prevented from falling over during the vertical drop test described in b) above.

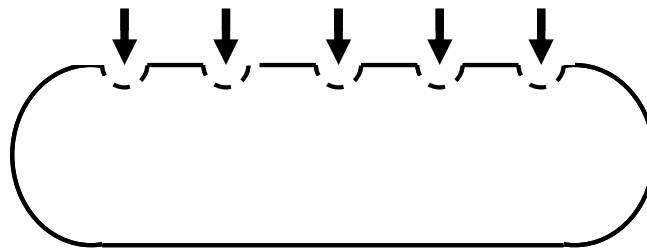
Following the drop impact, the containment vessel subjected to the 45° impacts should then be subjected to further testing as specified in 5.2.2. The vessel(s) subjected to horizontal and vertical drop impacts, if different from the vessel subjected to a 45° drop impacts, should be subjected to 1000 hydraulic ambient-temperature pressure cycles per the test procedure defined in 6.4.2.2.



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6.4.2.5 Surface Damage Test (Unpressured). The test should proceed in the following sequence:

- a) Surface Flaw Generation: Two longitudinal saw cuts are made on the bottom outer surface of the unpressurized horizontal storage container along the cylindrical zone close to but not in the shoulder area. The first cut will be at least 1.25 mm deep and 25 mm long toward the valve end of the vessel. The second cut will be at least 0.75 mm deep and 200 mm long toward the end of the tank opposite the valve.
- b) Pendulum Impacts: The upper section of the horizontal storage container should be divided into five distinct (not overlapping) areas 100 mm in diameter each (see Figure). After 12 hrs preconditioning at  $-40\text{ }^{\circ}\text{C}$  in an environmental chamber, the center of each of the five areas should sustain impact of a pendulum having a pyramid with equilateral faces and square base, the summit and edges being rounded to a radius of 3 mm. The center of impact of the pendulum should coincide with the center of gravity of the pyramid. The energy of the pendulum at the moment of impact with each of the five marked areas on the containment vessel should be 30J. The tank should be secured in place during pendulum impacts and not under pressure.



**"Side" View of Tank**

6.4.2.6 Chemical Exposure Test. Each of the 5 areas of the unpressured vessel preconditioned by pendulum impact (6.4.2.5b) should be exposed to one of five solutions: 1) 19% (by volume) sulfuric acid in water (battery acid), 2) 25% (by volume) sodium hydroxide in water, 3) 5% (by volume) methanol in gasoline (fluids in fueling stations), 4) 28% (by volume) ammonium nitrate in water (urea solution), and 5) 50% (by volume) methyl alcohol in water (windshield washer fluid).

Orient the test vessel with the fluid exposure areas on top. Place a pad of glass wool approximately 0.5 mm thick and 100 mm in diameter on each of the five preconditioned areas. Apply an amount of the test fluid to the glass wool sufficient to ensure that the pad is wetted across its surface and through its thickness for the duration of the test.

The exposure of the vessel with the glass wool should be maintained for 48 hrs with the vessel held at 1.25% NWP (applied hydraulically) and ambient temperature ( $15\text{C} - 25\text{C}$ ) before the vessel is subjected to further testing. When chemical exposure is discontinued, the glass wool should be removed and the exposed area should be rinsed with water.

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6.4.2.7 Static Pressure Test (Hydraulic). Pressurize the storage system to the target pressure in temperature-controlled chamber held at the target temperature  $\pm 5^{\circ}\text{C}$ .

6.4.2.8 Gas Pressure Cycling Test (Pneumatic). At the onset of testing, stabilize the storage system at the specified temperature, relative humidity and fuel level at least 24 hrs, if necessary in a temperature-controlled chamber. Maintain the specified temperature and relative humidity within the test environment throughout the remainder of the test. (When required in the test specification, the system temperature should be stabilized at the external environmental temperature between pressure cycles.) Pressure cycle between  $< 2$  MPa and the specified maximum pressure. Control the fill rate to a constant 3-minute pressure ramp rate; control the temperature of the hydrogen fuel dispensed to the vessel to the specified temperature. Control the defueling rate to no less 2g/sec or the intended vehicle's maximum fuel-demand rate. Conduct the specified number of pressure cycles. If devices and/or controls are used in the intended vehicle application to prevent an extreme internal temperature, the test may be conducted with these devices and/or controls (or equivalent measures).

6.4.2.9 Gas Permeation Test (Pneumatic). A storage system shall be fully filled with hydrogen gas (full fill density equivalent to 100% NWP at 15 °C is 113% NWP at 55 °C) and held at 55°C in a sealed container. The total steady-state discharge rate due to leakage and permeation from the storage system shall be measured.

6.4.2.10 Localized Gas Leak Test (Pneumatic). A bubble test (or alternative method with sufficient accuracy) may be used to fulfill this requirement. The following guidance is provided for conducting the bubble test:

- a. The exhaust of the shutoff valve (and other internal connections to hydrogen systems) may be capped for this test (as the test is focused at external leakage). At the discretion of the tester, the test article may be immersed in the leak-test fluid or leak-test fluid applied to the test article when resting in open air. Bubbles can vary greatly in size, depending on conditions. In general, the tester should estimate the gas leakage based on the size and rate of bubble formation.
- b. Note: Visual detection of unacceptable leakage should be feasible. When using standard leak-test fluid, the bubble size is expected to be approximately 1.5 mm in diameter. For a localized rate of 0.005 mg/sec (3.6 cc/min), the resultant allowable rate of bubble generation is about 2030 bubbles per minute. Even if much larger bubbles are formed, the leak should be readily detectable. For example, the allowable bubble rate for 6 mm bubbles would be approximately 32 bubbles per minute.

If the permeation test conducted in 6.4.2.9 yields a total discharge less than the specified allowable localized leak, then localized leak testing is not necessary as the total system leakage is already below the localized leak requirement.

6.4.2.11 Proof Leak Test (Pneumatic). A bubble test (or alternative method with sufficient accuracy) to verify local leakage should be conducted as follows: a) The exhaust of the shutoff valve (and other internal connections to hydrogen systems) may be capped for this test (as the test is focused at external leakage).

At the discretion of the tester, the test article may be immersed in the leak-test fluid or leak-test fluid applied to the test article when resting in open air. Bubbles can vary

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greatly in size, depending on conditions. In general, the tester should estimate the gas leakage based on the size and rate of bubble formation. b) When using standard leak-test fluid, the bubble size is expected to be approximately 1.5 mm in diameter and the resultant allowable rate of bubble generation is about 2030 bubbles per minute. Even if much larger bubbles are formed, the leak should be readily detectable. For example, the allowable bubble rate for 6 mm bubbles is still approximately 32 bubbles per minute.

6.4.2.9 Fire Test (pneumatic). TBD