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Final Report

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16. Abstract

The contractor's Fire Technology Department, under contract with NHTSA, performed two series of testing of compressed hydrogen storage systems to evaluate and provide feedback on the proposed fire test procedures. For the Base Period, four Type IV CHSS were subjected to the GTR NO. 13 fire test method. For the Option Period, three Type III standard CHSS and one Type IV conformable CHSS were subjected to the draft fire test method provided by NHTSA.

The contractor fabricated the standard burner and pre-test container. Prior to each test series, pre-test burner checkout testing was performed to determine the necessary propane gas flow rates to meet the temperature requirements. The pre-test container was instrumented with seventeen thermocouples on and around the tank to fully characterize the thermal exposure. The flame temperature requirements for the CHSS tests were set based on the results from the pre-test burner checkout test.

This final report contains the test results and the contractor lessons learned from conducting the physical fire tests. Additionally, the final report for the Closures Tests is included as Appendix A of this report.

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FIRE TESTS EXECUTIVE SUMMARY

The contractor's Fire Technology Department under contract with the National Highway Traffic Safety Administration (NHTSA) performed two series of testing of compressed hydrogen storage systems (CHSS) to evaluate and provide feedback on the fire test procedures. For the Base Period (9/29/2020 – 9/29/2021), four (4) Type IV CHSS were subjected to the fire test method prescribed in the United Nations Economic Council for Europe Global Technical Regulation (GTR) No. 13, Section 6.2.5, on May 19 and 20, 2021. For the Option Period (9/29/2021 – 3/28/2023), three (3) Type III standard CHSS and one (1) Type IV conformable CHSS were subjected to the draft test method provided by NHTSA on December 12-16, 2022, which was based upon GTR No. 13 for localized and engulfing bonfire testing of hydrogen containers. All testing was performed at the contractor's remote test site, located in Sabinal, Texas.

The contractor fabricated the standard burner and pre-test container. Prior to each test series, pre-test burner checkout testing was performed to determine the necessary propane gas flow rates to meet the temperature requirements. The pre-test container was instrumented with seventeen (17) thermocouples (TCs) on and around the tank to fully characterize the thermal exposure. The flame temperature requirements for the CHSS tests were set based on the results from the pre-test burner checkout test.

Each CHSS was secured above the standard burner, instrumented with temperature and pressure sensors, filled to the appropriate setting with hydrogen gas, and then subjected to the prescribed two-stage fire exposure until the pressure was vented. Flame temperatures, LPG flow rates to each burner zone, and CHSS pressure were recorded. Testing was documented with pre- and post-test photographs, and video from one angle.

This final report contains a summary of the Base Period and Option Period test results and the contractor lessons learned from conducting the physical tests which may benefit one. The final report for the Closures Tests is included as Appendix A of this report.

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1.0 INTRODUCTION

The objective of this research project was to evaluate and provide feedback on compressed hydrogen storage system (CHSS)[1] fire test methods over two contract periods. For the Base Period (9/29/2020 – 9/29/2021), four (4) Type IV CHSS[2] were subjected to the fire test method prescribed in the United Nations (UN) Economic Council for Europe (ECE) Global Technical Regulation (GTR) No. 13, Section 6.2.5, on May 19 and 20, 2021. For the Option Period (9/29/2021 – 3/28/2023), three (3) Type III standard CHSS and one (1) Type IV conformable CHSS were subjected to the draft test method provided by National Highway Traffic Safety Administration (NHTSA) which was based, in part, on GTR No. 13 for localized and engulfing bonfire testing of hydrogen containers (dated November 17, 2022), on December 12-16, 2022. This program was conducted by Southwest Research Institute's (SwRI®) Fire Technology Department for NHTSA.

The following sections summarize the test performance by period, and the overall lessons learned. The results presented in this report apply only to the materials tested, in the manner tested, and not to any similar materials or material combinations.

2.0 BASE PERIOD

The Base Period objective was to evaluate the test procedure and perform testing of four (4) 70 MPa, Type IV compressed hydrogen storage systems (CHSS) in accordance with ECE GTR No. 13, Section 6.2.5, *Test Procedures for Service Terminating Performance in Fire* (also known as ECE-TRANS-180a13e_TF0_Draft_19Mar2021). The contractor fabricated the standard burner and pre-test container, procured test samples, and performed the pre-test checkout and CHSS tests per GTR No. 13, Section 6.2.5.[3] Testing was performed on May 19 and 20, 2021. The following sections summarize the test samples selected for testing, and results of the pre-test checkout and CHSS tests.

2.1 Test Samples

The contractor conducted a study to identify CHSS manufacturers, and request pricing and lead time for four (4) CHSS with associated hardware. The customer defined the requirements and preferred specifications. The following specifications were the goal of the survey:

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[1] According to GTR No. 13, a Compressed Hydrogen Storage System (CHSS) is a system designed to store compressed hydrogen fuel for a hydrogen-fueled vehicle, composed of a container, container attachments (if any), and all primary closure devices (such as shut-off valve, check valve, and TPRD) required to isolate the stored hydrogen from the remainder of the fuel system and the environment.

[2] See CSA/ANSI HGV 2-2021 for definitions of Type III and Type IV CHSS assemblies.

[3] See GTR No. 13 for definition of standard burner and pre-test container.

Required	Preferred
• Type IV tank • Approx. $10,000 USD or less per CHSS and OTV/TPRD(s) • Delivery within 3 months from the contract award date (12/29/2020)	• 35 MPa service pressure • Length ≤1.65 m • TPRD on one side only • Manual tank valve

For the second stage of the project and due to contractual limitations, NHTSA and the contractor selected the following required characteristics: tank type, cost, and delivery timing.

The contractor contacted eight manufacturers and received pricing and lead time from five. It became apparent that 70 MPa service pressure CHSS are more prevalent for passenger vehicles, and most manufacturers did not have stock available for immediate or near-term purchase. A spreadsheet summarizing the market survey was submitted to NHTSA. Based on the results, the contractor proposed, and NHTSA approved, the tank design specified in Table 1. The CHSSs were received by the contractor on March 2, 2021.

Table 1. Base Period Tank Design Details.

Specification	Nominal Value
Length (without valve)	865 mm
Length of middle (without domes)[4]	480 mm
Diameter	376 mm
Water Volume	52 L
Weight	45 kg
Service Pressure	70 MPa at 15 °C

2.2 Pre-Test Checkout Tests

The pre-test burner checkout test was performed on May 18, 2021, at the contractor's remote test site in order to determine the burner flow rate settings for each fire stage that met the temperature requirements as shown in Table 2. The pre-test included a 320 mm diameter steel container (fabricated from 300 mm/12 in. Schedule 40 pipe with hemispherical tank ends, 1,650 mm long not including the smaller pipe section representing the valve, with a body length without the hemispherical tank ends of 1350 mm) instrumented with seventeen (17) thermocouples (TCs) on and around the tank to fully characterize the thermal exposure. The container surface TCs were within located within a 5 mm gap from the pre-test container surface, and the flame TCs were 25 ± 5 mm below the container bottom surface as shown in Figure 1.

[4] Length of Middle (between domes) is the length of the primarily flat region between the domes. See the length between TUL and TUR in Figure 1.

Table 2. Pre-test Burner Checkout Temperature Requirements (GTR NO. 13, Table 2c).

Fire Stage	Allowable Temperature Range on		
	Bottom of Container	**Sides of Container**	**Top of Container**
Localized	450 – 700 °C	< 700 °C	100 – 300 °C
Engulfing[5]	> 600 °C	N/A	260 – 750 °C

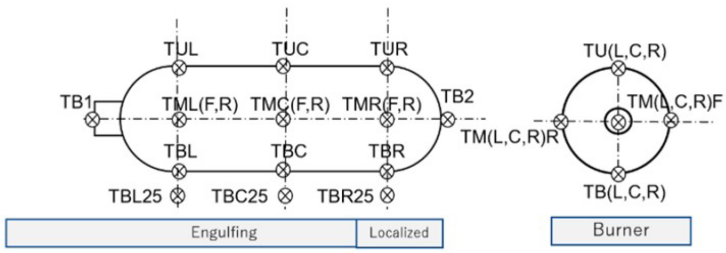

Figure 1. Pre-Test Container and Thermocouple Locations (GTR NO. 13, Figures 6h and 6i).

For the Localized Fire Stage, the flow rate was set to 30 Standard Liters Per Minute (SLPM) to meet the temperature requirements. For the Engulfing Fire Stage, a flow rate of 260 SLPM met the temperature requirements. Figure 2 shows an example of the flame and container surface temperatures for the pre-test burner checkout.

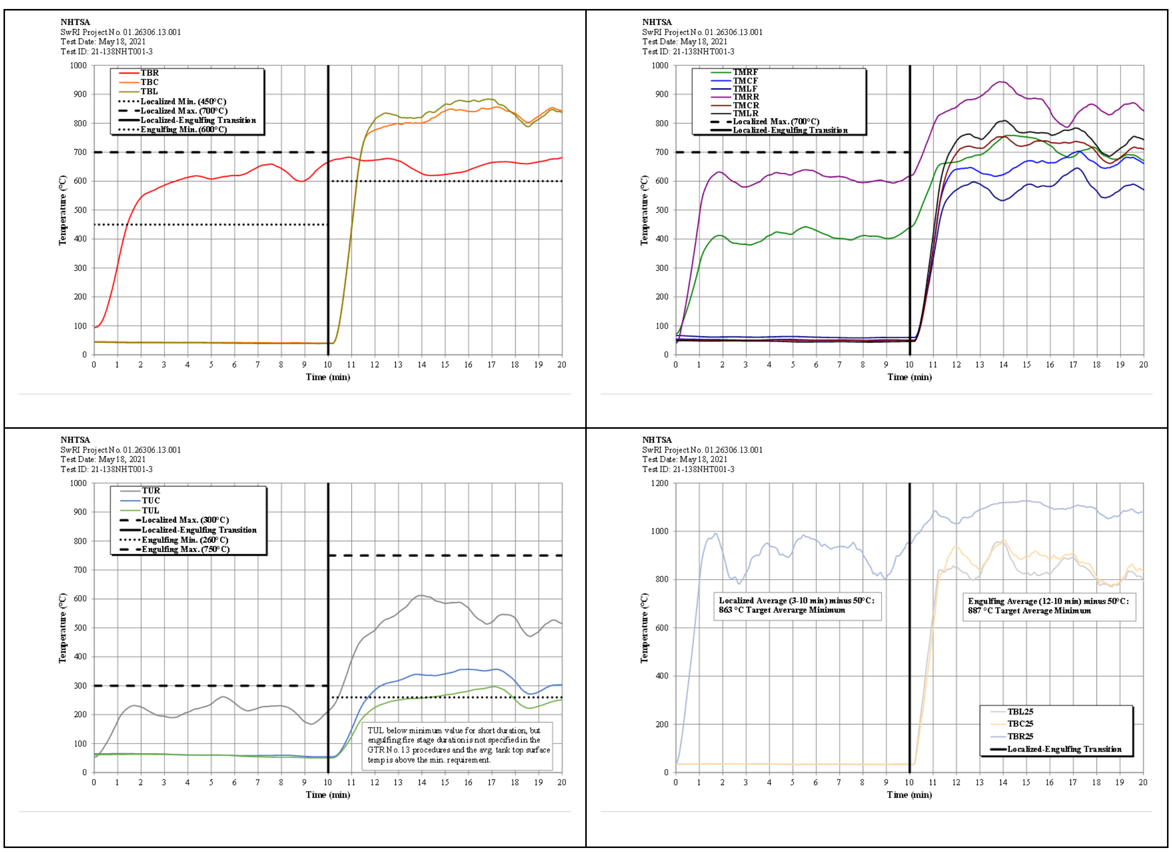

Figure 2. Base Period Pre-Test Burner Checkout Temperatures.

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5 See GTR No. 13 for a description of engulfing.

The allowable limits for the burner monitors during subsequent CHSS container fire testing was established based on the flame temperature measurements during the pre-test burner checkout:

- Localized Fire Stage minimum value for the burner monitor during the CHSS tests ($Tmin_{LOC}$) was calculated by subtracting 50 °C from the average of the 60 second rolling average of TBR_{25} from 3-10 min (stabilized conditions). **$Tmin_{LOC}$ was calculated to be 863 °C.**
- Engulfing Fire Stage minimum value for the burner monitor during the CHSS tests ($Tmin_{ENG}$) was calculated by subtracting 50 °C from the average of the 60 second rolling averages of the three flame thermocouple readings (TBL_{25}, TBC_{25}, and TBR_{25}) from 12-20 min (stabilized conditions). **$Tmin_{ENG}$ was calculated to be 887 °C**.

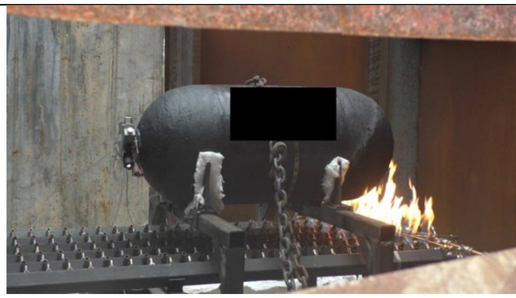
2.3 Test Performance and Results

A series of testing was performed on four (4) 70 MPa, Type IV CHSS in accordance with GTR No. 13, Section 6.2.5, on May 19 and 20, 2021, by the contractor's Fire Technology Department at a remote test site in Sabinal, Texas. Test observations and performances are summarized in Table 3.

Prior to each test, the CHSS was installed 100 mm ± 5 mm above the burner and positioned so that the main valve containing the TPRD was furthest from the localized portion of the burner per GTR No. 13 Section 6.2.5.1.1 and Figure 6a (Case 1). The pressurization tubing was connected to a high pressure port on the main valve, and the bleed device opened to allow for live monitoring of the tank pressure. A signal cable was connected to the temperature sensor on the main valve to monitor tank temperature (used to calculate State of Charge (SOC)). Three thermocouples were positioned 25 mm ± 5 mm below the bottom of the tank surface (one in the localized section, and two in the remaining burner length). Figure 3 shows the various stages of the testing for a representative tank (all four CHSS were the same make/model).

Pre-test Burner Checkout Setup

Prior to CHSS Test



CHSS Localized Fire

CHSS Engulfing Fire

CHSS TPRD Activation

CHSS Post-Test

Figure 3. Representative Two-Stage Localized/Engulfing Fire Test (Test 1).

All CHSS successfully vented pressure without tank rupture or significant jetting from areas outside of the intended TPRD (≤ 0.5 m); however, the flame temperatures were not in compliance with the requirements of GTR No. 13 which would invalidate the test results. These observations are discussed in the Lessons Learned section of this report.

Table 3. Base Period Test Observations.

Test Values	Test No.			
	1	2	3	4
Test Date	05/19/2021	05/19/2021	05/20/2021	05/20/2021
State of Charge (%)*	98.2 %	101 %	100 %	100 %
Initial Tank Pressure (MPa)†	73.41	76.10	75.15	76.61
Initial Tank Temperature (°C)	25.5	23.8	23.4	29.6
Localized Flame Average (°C) (Target = 863 °C min.) ‡	680	587	892	781
Engulfing Flame Average (°C) (Target = 887 °C min.) ‡	883	558§	865§	841
Test Events	**Time (min: s)**			
DAQ/Camera On	0:00	0:00	0:00	0:00
Ignition of Localized Fire	1:03	1:08	1:02	1:02
Ignition of Engulfing Fire	11:12	11:54#	11:11	11:13
Initial leak	12:16	17:19	13:45	13:39**
TPRD Activation	14:29	17:27	14:39	13:39**
Tank Pressure ≤ 1 MPa	15:55	18:03	16:25	15:09
Burner Off	16:04	18:15	16:29	15:25
Test Terminated	19:00	22:04	18:31	17:32

* GTR No. 13 draft used did not provide tolerance for State of Charge. The contractor assumed a +/- 2 % tolerance.
† Tank NWP is 70 MPa at 15 °C. Test tank pressure slightly higher due to higher tank temperature. State of charge calculated per GTR No. 13, Section 3.51.
‡ Flame temperatures during testing varied from those during pre-test burner checkout. Discussion in report.
§ TBL_{25} and TBC_{25} were consistently lower than TBR_{25} during the CHSS tests which decreased the average flame temperatures during the engulfing fire portion of the tests.
# Flow controller malfunctioned; engulfing fire igntion delayed.
** Initial leak and TPRD Activation occurred simultaneously.

3.0 OPTION PERIOD

The Option Period objective was to evaluate the test procedure and perform testing of four (4) 35 MPa, compressed hydrogen storage systems (CHSS) (three (3) Type III and one (1) Type IV) in accordance with the draft test method provided by NHTSA (dated/received on November 17, 2022) which references ECE Global Technical Regulation (GTR) No. 13, Section 6.2.5, *Test Procedures for Service Terminating Performance in Fire*.[6] The contractor procured the test samples and performed the pre-test checkout and CHSS tests per the NHTSA draft test method. Testing was performed December 12

[6] Note that both the NHTSA draft test procedure and GTR No. 13 were under development during testing.

– 16, 2022. The following sections summarize the test samples selected for testing, and results of the pre-test checkout and CHSS tests.

3.1 Test Samples

The contractor conducted a market survey to identify CHSS manufacturers, and request pricing and lead time for four (4) complete CHSS and associated hardware. Due to the small number of CHSS suppliers, NHTSA developed two sets of CHSS selection criteria for the contractor:

Required	**Preferred**
• Less than or equal to $10,000 USD per CHSS including On-Tank Valve (OTV) and Thermally activated Pressure Relief Device(s) (TPRD(s)) and shipping. • Delivery and testing prior to 27 months after award (12/29/2022).	• Larger than tanks selected for Base Period; preferably with length ≥ 1.65 m. • ≤35 MPa service pressure (Contractor's request due to hydrogen volume needed) • TPRD on both sides. • Manual tank valve.

The contractor contacted six manufacturers (three additional were considered but not contacted due to Base Period responses and budget/schedule constraints) and received pricing/lead time from three manufacturers. Based on the results, the contractor proposed, and NHTSA approved the selections documented in Table 4. The CHSS were received by the contractor on December 5 and 7, 2022.

Table 4. Option Period Tank Design Details.

Specification	**CHSS 1 Nominal Values**	**CHSS 2-4 Nominal Values**
Description	One (1) Type IV, Conformable Tank within aluminum enclosure and long-trigger TPRD lines with vent ports on main valve and aft.	Three (3) Type III, Standard Tanks each with TPRD on main valve and end plug.
Dimensions	2350 × 1920 × 60 mm (L×W×H)	432 mm OD × 2110 mm (valve/end plug not included in length)
Water Volume	102 L	205 L
Service Pressure	5,000 psig (~35 MPa)	5,000 psig (~35 MPa)

3.2 Pre-Test Checkout Test

The pre-test burner checkout test was performed on December 12, 2022, at the contractor's remote test site in order to determine the burner flow rate settings for each fire stage that met the temperature requirements as shown in Table 5, which are slightly different than the previous GTR NO. 13 requirements used for the base period testing due to the evolving nature of the GTR test procedures. Otherwise, the setup was the same as the Base Period.

Table 5. Pre-test Burner Checkout Temperature Requirements (NHTSA Draft Procedure, Table 6).

Fire Stage	Temperature Range on Pre-Test Container		
	Bottom	Sides	Top
Localized	450 – 700 °C	less than 700 °C	less than 300 °C
Engulfing	Average > 600 °C	N/A	100 – 750 °C

For the Localized Fire Stage, the flow rate was set to 40 SLPM to meet the temperature requirements. For the Engulfing Fire Stage, a flow rate of 325 SLPM was set to meet the temperature requirements. These were higher than the flow rates required for the Base Period testing which was likely due to the ambient temperature/time of year difference between the test series (Base Period in May 2021 and Option Period in December 2022). Figure 4 shows the flame and container surface temperatures for the pre-test burner checkout.

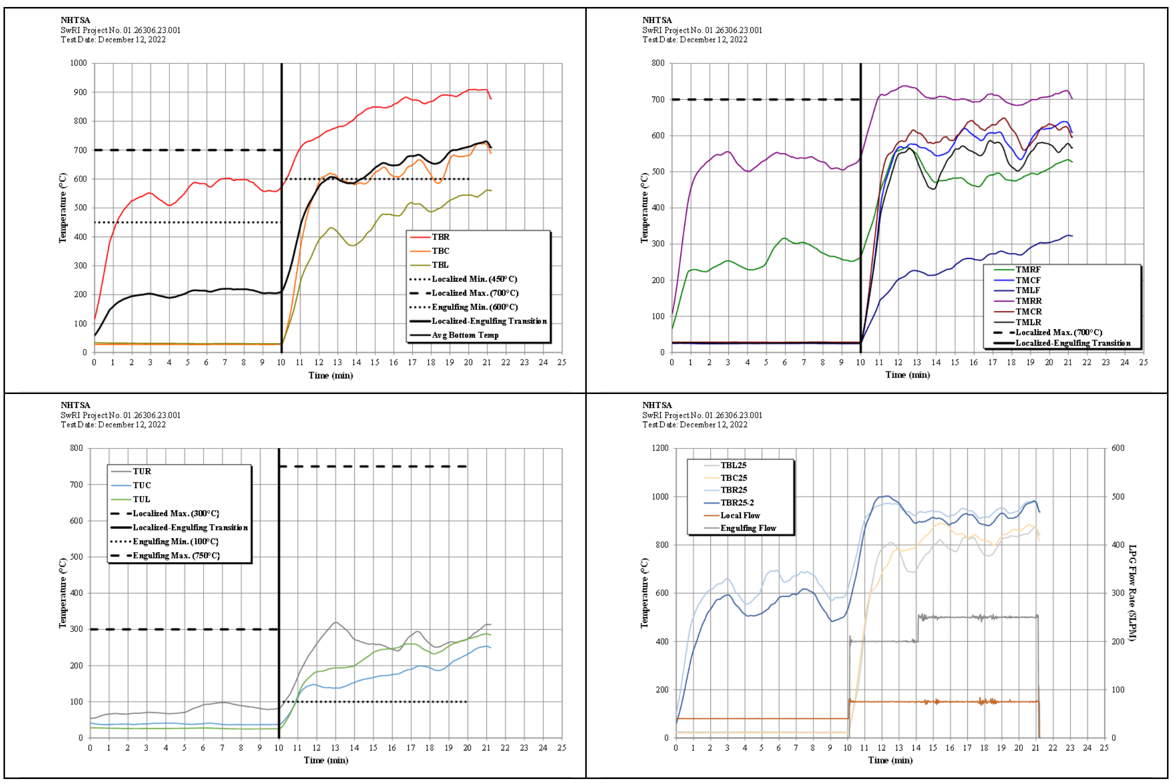


Figure 4. Option Period Pre-Test Burner Checkout Temperatures.

The allowable limits for the burner monitors during subsequent CHSS container fire testing was established based on the flame temperature measurements [7] during the pre-test burner checkout:

- Localized Fire Stage minimum value for the burner monitor during the CHSS tests ($Tmin_{LOC}$) was calculated by subtracting 50 °C from the average of the 60 s rolling average of TBR_{25} from 3-10 min (stabilized conditions). **$Tmin_{LOC}$ was calculated to be 540 °C.**

[7] See GTR NO. 13 Section 6.2.5.1.4.5 '*Pre-test Checkout Process*' for more details.

- Engulfing Fire Stage minimum value for the burner monitor during the CHSS tests ($Tmin_{ENG}$) was calculated by subtracting 50 °C from the average of the 60 s rolling averages of the three flame thermocouple readings (TBL_{25}, TBC_{25}, and TBR_{25}) from 15-21 min (stabilized conditions). **$Tmin_{ENG}$ was calculated to be 829 °C**.

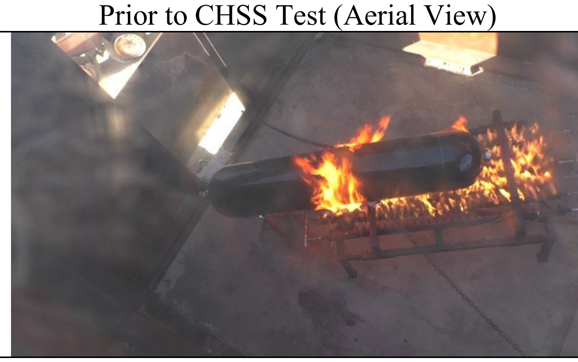
3.3 Test Performance and Results

A series of testing was performed on four (4) 35 MPa, CHSS (one Type IV and three Type III) in accordance with the draft test method provided by NHTSA which references GTR No. 13 for localized and engulfing bonfire testing of hydrogen containers (dated/received on November 17, 2022), on December 12-16, 2022, by Southwest Research Institute's Fire Technology Department at the remote test site in Sabinal, Texas. Test observations and performances are summarized in Table 6.

Prior to each test, the CHSS was installed 100 mm ± 5 mm above the burner so the TPRDs were furthest from the localized portion of the burner per the draft test method. The pressurization tubing was connected to a high-pressure port on the main valve to allow for live monitoring of the tank pressure. Three thermocouples were positioned 25 mm ± 5 mm below the bottom of the tank surface (one in the localized section, and two in the remaining burner length; two additional thermocouples were added to each zone for information purposes). Figure 5 shows the various stages of the testing for one of the standard tank tests (Test 2). Figure 6 shows the conformable tank test (Test 1). For the conformable tank test, the burner flames are not visible during the test from the camera angle, but the post-test photograph of the bottom surface shows where the burner impinged on the enclosure.

Pre-test Burner Checkout

Prior to CHSS Test (Aerial View)



CHSS Localized Fire	CHSS Engulfing Fire
	
CHSS TPRD Activation	CHSS Post-Test

Figure 5. Representative Two-Stage Localized/Engulfing Fire Test (Test 2).

 Prior to CHSS Test	 CHSS TPRD Activation (Fill Side)
 CHSS TPRD Activation (Aft Side)	 CHSS Post-Test (Bottom Surface)

Figure 6. Two-Stage Localized/Engulfing Fire Test for Conformable Tank (Test 1).

All CHSS successfully vented pressure without tank rupture or significant jetting from locations outside of the TPRDs. The localized fire stage flame temperatures were in compliance with the requirements of the draft test method, but most of the engulfing fire stage flame temperatures were slightly lower than the allowable minimum requirement. These observations are discussed in the Lessons Learned section of this report.

Table 6. Test Observations

Test Values	Test No.

	1	2	3	4
CHSS Specifications	Type IV Conformable	Type III Standard	Type III Standard	Type III Standard
Test Date	12/13/2022	12/14/2022	12/15/2022	12/16/2022
State of Charge (%)	100.5 %	100.6 %	101.5%	101.4%
Initial Tank Pressure (MPa)	37.8	36.1	36.9	37.9
Initial Tank Temperature (°C)	25.0	17.0	19.8	22.5
Localized Flame Average (°C) (Target = 540 °C min.)	696	878	795	865
Engulfing Flame Average (°C) (Target = 829 °C min.)	N/A	747	842	766
Test Events	**Time (min: s)**			
DAQ/Camera On	0:00	0:00	0:00	0:00
Ignition of Localized Fire	1:51	1:34	1:49	1:24
Ignition of Engulfing Fire	N/A	11:54	12:07	11:43
Initial leak/TPRD Activation	2:22	16:30	15:38	15:17
Tank Pressure ≤ 1 MPa	3:27	19:30	18:49	21:24
Burner Off	3:31	19:50	19:02	21:36
Test Terminated	6:20	22:17	22:46	24:55

4.0 LESSONS LEARNED

The following list summarizes the contractor's observations, comments, and findings for the fire test methods performed during the Base and Option Periods. The lessons learned are bulleted with additional details or supporting statements written in italics below each and are intended as items one may consider.

Burner

- The burner rail square tubing size is not specified. The tubing size affects the distance between the nozzle tips.
    - *The contractor utilized 2-in. square tubing.*

Pre-Test Checkout

- As stated in both test methods, the pre-test steel container must be equal to or longer than overall length of the CHSS to be tested, but no shorter than 0.80 m and no longer than 1.65 m, including closure devices and container attachments as applicable. Based on this description, a test lab could fabricate a single pre-test steel container at the maximum length to meet the requirements of the test method for any CHSS to be tested. This was the contractor's interpretation, and all pre-test checkouts were performed with a 1.65 m long container. However, during this test program it was observed that the flame temperatures were lower than the target temperatures for CHSSs that did not cover the full length of the burner (i.e., the Base Period CHSSs, and the Option Period standard Type III CHSS with TPRDs on both sides which was offset so that the CHSS was centered above the localized zone). This could be due to the difference in air flow to the burner based on the difference between the pre-test container and CHSS geometries.
    - *Considering these observations, a test lab may want to consider fabricating at least two (2) pre-test steel containers for the pre-test checkout; one that is the maximum length of the burner (1.65 m) and one that is 800 mm long and to use best judgement on which pre-test container to use based on the length of the CHSS to be tested. The contractor only fabricated a pre-test container at the maximum length that was utilized for both the Base Period and Option Period testing.*
- NHTSA draft test method states, "The pre-test container shall be mounted over the burner such that its position to the localized and engulfing zones of the burner are consistent with the positioning of the CHSS over the burner."
    - *One should be aware that the pre-test container position is varied if the CHSS to be tested has one vs. two TPRDs. S6.2.5.3 specifies, "(c) CHSS shall be positioned for the*

localized fire test by orienting the CHSS such that the distance from the center of the localized fire exposure to the TPRD(s) and TPRD sense point(s) is at or near maximum." And S6.2.5.2 says to position the pre-test container consistent with S6.2.5.3.

- The pre-test container may be similar to a standard CHSS design, but the pre-test container may not be applicable for flat conformable CHSS or larger diameter standard CHSS that cover more of the burner surface area. It is unknown how the diameter or width of the CHSS could affect air flow to the burner (e.g., larger tanks and conformable tanks with large surface areas).
    - *One may consider a flat pre-test panel that covers all or a majority of the burner area for these scenarios.*
- Thermocouple junction type is not specified and could affect test data values and response times of the sensors.
    - *One may consider using grounded junction and/or exposed junction thermocouples for the surface and flame thermocouples. Exposed junction thermocouples have the fastest response time, but as a result may provide noisier data than a grounded junction. The contractor utilized 1/8-in. diameter, Type K, grounded junction thermocouples for all locations. Since the data is processed as 1-min rolling averages, faster thermocouple response times may not have a large impact.*
- Flame thermocouple placement is discussed generically that it should be centrally located at the localized fire zone, and then spread out over the remaining length of the engulfing fire zone.
    - *One may want to consider adding a note that the flame thermocouples be placed so that they are impinged by a burner nozzle pair since temperatures will vary locally depending on whether the thermocouple is in-line with the burner nozzles or between them laterally.*
- The minimum burner monitor requirements and how these are calculated are unclear.

  Current text in S6.2.5.3(h) says:

  (1) The minimum value for the burner monitor temperature during the localized fire stage (TminLOC) shall be calculated by subtracting 50 °C from the 60-second rolling average of the burner monitor temperature in the localized fire zone of the pre-test checkout.

  (2) The minimum value for the burner monitor temperature during the engulfing fire stage (TminENG) shall be calculated by subtracting 50 °C from the 60-second rolling average of the

average of the three burner monitor temperatures during the engulfing fire stage of the pre-test checkout.

- *As currently specified, the TminLOC and TminENG could be interpreted as time dependent.*
- *Instead of calculating rolling averages, the average flame temperatures during the stabilized periods for each fire stage could be used to calculate the minimum burner monitor temperature during the CHSS test. This would require the pre-test burner checkout to be performed to the CHSS temperature profile as shown in. For example, if the burner is ignited at t = 0 min, calculate the average of the burner monitor thermocouple in the localized zone from 3-10 min, and calculate the average of all three burner monitor thermocouples from 12-20 min.*

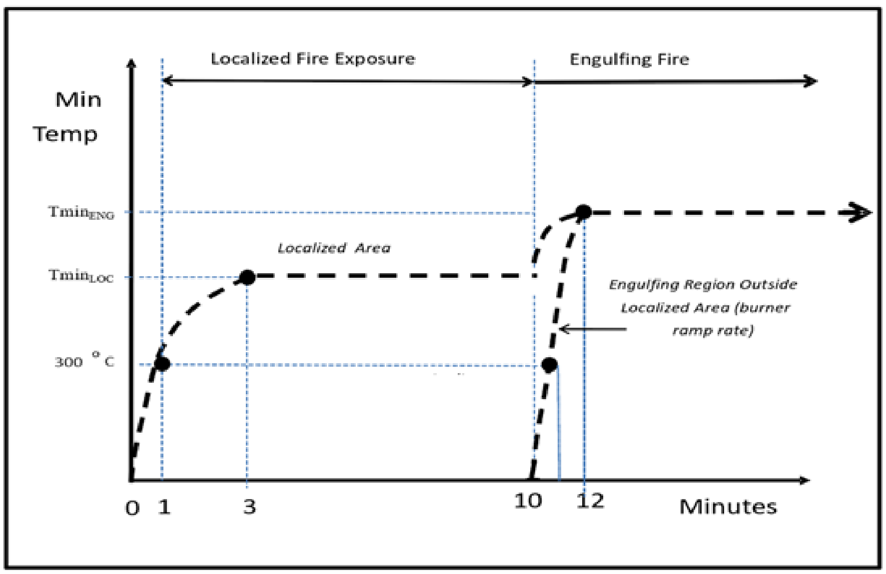


Figure 7. Temperature Profile for the Fire Test (NHTSA draft procedure, Figure 9).

<u>CHSS Test</u>

One may consider the following based on the contractor test performance observations:

- During the Base Period Test 1 the hydrogen supply line weakened due to exposure to the burner and/or vented hydrogen, and subsequently failed during Test 2. This resulted in hydrogen venting from the hole in the supply line addition to the TPRD.
    - *Subsequently, the contractor insulated the hydrogen supply line with 1-in. thick ceramic fiber insulation blanket. One may also choose to insulate/protect the hydrogen supply line to protect it from the burner and hydrogen vent gas flames.*

- The maximum allowable wind conditions, equipment, and procedures are unclear for the pre-test checkout and the CHSS specimen testing.
    - *The contractor chose to measure the wind conditions with a handheld hot-wire anemometer inside the test facility near the CHSS prior to pressurization to ensure the wind speed was below 2 m/s (requirement specified in FMVSS 304 Bonfire test for CNG tanks). Wind conditions were monitored outside the facility throughout the test to verify it was similar to pretest conditions. Wind measurement within the facility is no practical due to safety concerns and likely damage to the sensor from the burner and/or venting hydrogen.*
- The burner thermocouple locations on the horizontal plane vary relative to the specimen under study.
    - *While the contractor did not change the burner thermocouple locations, one may consider mounting the burner monitor thermocouples relative to the burner length and burner nozzle locations, not to the pre-test container/CHSS length, to maintain consistent locations between the pre-test checkout and CHSS test(s).*
- In GTR No. 13, the axis of rotation for CHSS mounting is not clear. GTR No. 13 Section 6.2.5.1.1 states, "The CHSS shall be rotated relative to the localized burner to minimize the ability to TPRDs to sense the fire and respond."
    - *The contractor assumed this statement referred to the vertical axis, but upon further review, it may also affect positioning along the longitudinal axis. In conjunction with the client, one may consider setting the rotation for ease of pressure line connection, safety/protection of equipment from TPRD vent port direction, and/or movement created by vent gases.*
    - *Note, this is not applicable for the NHTSA draft test method, which specified:*

      "CHSS shall be positioned for the localized fire test by orienting the CHSS such that the distance from the center of the localized fire exposure to the TPRD(s) and TPRD sense point(s) is at or near maximum."
- Certain conformable CHSS designs have long-trigger TPRD lines which complicate the positioning procedure, as the positioning procedure requires that the sensors be placed at the maximum distance to the localized fire zone.
    - *One may need to rely on the test lab's personnel and their engineering judgement to define the specific positioning for a particular CHSS.*

- The procedure does not specify a method to hold the CHSS in position.
    - *Due to potential risk to employee safety and equipment, the contractor mechanically secured the CHSS above the burner using heavy duty chain to prevent movement upon TPRD activation, as well as positioned the TPRD vents upward if possible.*
- For the state of charge (SOC) calculation, it may not be possible to measure internal temperature depending on the valve design and test lab's measurement capabilities. The contractor was unclear whether the SOC should be determined by internal temperature or ambient temperature.
    - *The contractor chose to fill the CHSS slowly and allowed the CHSS pressure to settle for a certain period at the conclusion of filling such that the internal and ambient temperatures would be fairly similar.*

Flow rate and temperature requirements:

- Calculation of the average or rolling average flame temperatures are unclear since there are transition periods which will affect the values.
    - *The contractor focused these calculations on the stable periods for each phase (i.e., 3-10 min for the localized fire phase, and 12 min until end of test or TPRD activation for the engulfing phase, assuming the burner is ignited at 0 min).*
- The transition period ramp rate and associated temperature requirements of the CHSS test are not captured in the pre-test checkout.
    - *The contractor determined the flow rates needed for the two-stages to meet the temperature requirements, and then ran the pre-test checkout with the CHSS test timing to verify the ramp rates shown in NHTSA draft test method Figure 9 Temperature profile of the fire test.*
- The flow rates and minimum flame temperatures for the CHSS tests are set by the pre-test checkout. However, the minimum flame temperatures are fairly tight and may not be achieved during the CHSS specimen tests due to differences in materials, dimensions, and ambient conditions from the pre-test checkout. During the time between pre-test checkout and the CHSS specimen test, thermocouples may shift slightly which could affect the temperature reading by more than 50 °C (allowable difference).
    - *One may consider locating the burner monitoring thermocouples relative to the burner, rather than the pre-test tank or CHSS specimen.*

- The flow rates are defined by the pre-test checkout, which is a separate procedure from the CHSS specimen test.
  - *In the base period, the test procedure was unclear, and the contractor varied the gas supply in the attempt to increase various temperature measurements.*
  - *In the option period, the contractor kept the gas supply nominally constant between the pre-test checkout and the CHSS specimen test, per NHTSA direction.*
  - *If the flow rates are kept constant between the pre-test checkout and the CHSS test, the test lab is not given the ability to make adjustments if the flame temperatures are lower than the minimum allowable temperature which could invalidate the test results.*
  - *One may need to decide on the prioritization prior to testing.*

Measurements:

- There was concern during the test series of sensor noise and failure.
  - *Pressure and flow rate data had some noise issues during the Option Period.*
  - *Additional thermocouples were added during the Option Period for information purposes, but a few thermocouples were non-functional, or failed during a couple tests.*
  - *One may consider performing sensor verification prior to each test in order to identify and repair/replace damaged sensors.*

5.0 TEST METHOD COST ASSESSMENT

Fire testing is an important evaluation of the CHSS's ability to withstand fire and the TPRD to activate and safely vent pressure. The test facility will need to consider costs associated with the burner and pre-test container(s), infrastructure and operation, safety planning with using hydrogen gas, and consumable material costs as discussed below.

Current material costs for the burner and pre-test container was about $10,000 - $15,000. Labor/services to fabricate both could be an additional $10,000 - $15,000. Thermocouples, pressure transducer, flow controllers, data acquisition hardware and software, wind speed sensor, and calibration services of the equipment is about $25,000. The burner is supplied by propane gas, and the cost will be dependent on the quantity of pre-test checkout runs needed to meet the temperature requirements, as well as the quantity of CHSS tests to be performed. The CHSS are filled with hydrogen gas (generally 35 or 70 MPa); gas cost and labor to fill will be dependent on the tank volume and pressure rating as well as the pressurization equipment capabilities. The pressurization equipment could be an additional $25,000.

Setup and the pre-test burner checkout test required 60-80 labor-hours. Each CHSS test required about 24 labor-hours. Cleanup, data processing, and documentation required an additional 60-80 labor hours.

Due to the nature of this testing, remote outdoor test facilities are highly recommended. The site must be permitted for outdoor burning with the local environmental committee and law enforcement, and provide sufficient stand-off distances and/or reinforced facilities to protect personnel and infrastructure. Scheduling of outdoor testing is subject to favorable weather conditions (e.g., no active precipitation, low wind speeds, no drought conditions/burn bans). Additional costs not quantified here include remote facility maintenance and/or rental fees, equipment and material transportation/shipping and travel costs to/from the remote site, and test area barriers for safety and wind shielding.

6.0 TEST METHOD VALUE ASSESSMENT

At NHTSA's request, the contractor analyzed the potential value of the test procedure. The Two-stage Localized/Engulfing Fire Test covers the important safety need of preventing container rupture due to fire exposure, which is the most severe type of CHSS failure. The localized fire stage challenges the tank integrity prior to TPRD activation, and the engulfing fire verifies the ability of the system to vent pressure safely. This test method provides an invaluable safety performance assessment.

7.0 CONCLUSIONS

At NHTSA's request, during the project the contractor identified locations in the test procedure where additional specificity would help one conduct the testing. After addressing the potential inconsistencies or incompatibilities noted in the Lessons Learned, the contractor was able to conduct the testing per procedure.

APPENDIX A

GTR NO. 13 CLOSURES TESTS

(Consisting of 60 pages)

CLOSURES TESTS EXECUTIVE SUMMARY

Southwest Research Institute under contract with the National Highway Traffic Safety Administration (NHTSA) performed testing and engineering evaluations of a compressed hydrogen storage system (CHSS) on tank valve (OTV) to the test methods prescribed in ECE Global Technical Regulation (GTR) No. 13, Section 6.2.6, *Test Procedures for performance durability of primary closures*. The objective of this program was to evaluate and provide feedback on the CHSS series of tests for primary closures (thermally activated pressure relief device [TPRD], check valve, and shutoff valve) specified in GTR NO. 13 (dated October 7, 2021, and modifications as applicable; some test methods performed later in the series utilized the version received on September 15, 2022, which included optional tolerances). The test methods were analyzed via physical test performance or engineering evaluations. The contractor furnished the necessary qualified technical personnel, facilities, materials, equipment, and services to perform the procurement, receipt, inspection, testing/evaluation, reporting, storage and disposal of the closures. This program was conducted in the Fire Technology Department in the Chemistry and Chemical Engineering Division, and the Structural Engineering Department, Fluids Engineering Department, and Materials Engineering Department in the Mechanical Engineering Division. These evaluations were performed for National Highway Traffic Safety Administration (NHTSA), with a period of performance of September 29, 2021– March 29, 2023, by Southwest Research Institute.

The contractor performed testing or engineering evaluations of the various TPRD and Check Valve/Automatic Shut-Off Valve test methods in GTR NO. 13, Sections 5.1.5/6.2.6. Several of quantity of samples tested and number of cycles performed were reduced for budgetary and schedule constraints. Engineering evaluations consisted of thorough reviews of the relevant test method by a contractor subject matter expert. Initial feedback was provided in the related closures test reports on the testing performed and results obtained.

This final report contains a summary of the test results and the contractor lessons learned from the test performance and engineering evaluations for the GTR No. 13 test procedures for primary closures. Additionally, this final report discusses the qualitative value assessments for each test method; in which the contractor reviews the difficulty/cost to perform each test method compared to the safety benefit provided and/or potential risk mitigated.

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1.0 INTRODUCTION

The objective of this program is to evaluate and provide feedback on the compressed hydrogen storage system (CHSS) series of tests for primary closures (thermally activated pressure relief device [TPRD], check valve, and shutoff valve) specified in GTR NO. 13 (dated October 7, 2021, and modifications as applicable; some test methods performed later in the series utilized the version received on September 15, 2022, including optional tolerances). The test methods were analyzed via physical test performance or engineering evaluations. The contractor furnished the necessary qualified technical personnel, facilities, materials, equipment, and services to perform the procurement, receipt, inspection, testing/evaluation, reporting, storage and disposal of the closures. This program was conducted by the Fire Technology Department in the Chemistry and Chemical Engineering Division, and the Structural Engineering Department, Fluids Engineering Department, and Materials Engineering Department in the Mechanical Engineering Division. These evaluations were performed for National Highway Traffic Safety Administration (NHTSA), with a period of performance of September 29, 2021– March 29, 2023, by Southwest Research Institute.

The following sections describe the GTR NO. 13 closures test methods, summarize the test performance or engineering evaluation, the lessons learned, and test method value assessment for each test method. The results presented in this report apply only to the materials tested, in the manner tested, and not to any similar materials or material combinations. Additional test performance details are provided in the respective test reports as referenced in each section. The intent of this project was to review the test procedures and not identify the performance of specific valve hardware. Therefore, while certain test methods required specific quantities, the contractor elected with NHTSA's concurrence to focus the procedural evaluation on a smaller quantity of physical samples.

2.0 CLOSURES TEST METHODS

The contractor performed testing or engineering evaluations of the various TPRD and Check Valve/Automatic Shut-Off Valve test methods in GTR NO. 13, Sections 5.1.5/6.2.6. Tables A-1 and Table A-2 summarize the relevant tests and sample quantities (if physical testing was performed) for TPRD and Check Valve/Shut-Off Valves, respectively. The pressurized test methods are in bold. NHTSA requested reduced quantities of some of the test replicates in the test matrix and these are highlighted if they differ from the GTR NO. 13 specified quantities.

For the version of GTR studied, Sections 6.2.6.1 and 6.2.6.2 state that all primary closures testing is to be performed with hydrogen gas with gas quality compliant with ISO 14687:2019/SAE J2719_202003, unless otherwise specified. For safety purposes, the contractor required some tests be performed with an inert gas (e.g., GTR NO. 13, Section 6.2.6.1.9 Bench Top Activation Test and Section 6.2.6.2.8 Vibration Test were performed with nitrogen). NHTSA indicated the GTR NO. 13 committee is considering allowing an inert/non-reactive gas to be used for all primary closures testing instead. The authors believe there is still merit to requiring the use of hydrogen for some of the test methods, and this will be discussed in the following individual test method sections. The contractor used hydrogen gas quality compliance ≥ 99.97% purity with low moisture and particulate (e.g., ≤ 5ppm water and ≤ 1 ppm particulate) since specialty gas suppliers do not typically analyze to the aforementioned quality standards. The ISO/SAE fuel-grade gas quality requirements are intended for fuel cell sensitivities which are unlikely to affect the performance of closure devices.

Table A-1. TPRD Performance Tests.

GTR NO. 13 Section No.*	GTR NO. 13 Section Title	Sample Quantity** and (Contractor Sample IDs)	Subsequent Tests
6.2.6.1.1	**Pressure Cycling Test**	1 Unit (T1)	Leak Test Activation Test Flow Rate Test
6.2.6.1.2	**Accelerated Life Test**	2 Units (T2 @ T_{act} and T3 @ T_L)	Leak Test (T_L only)
6.2.6.1.3	**Temperature Cycling Test**	1 Unit (T4)	Leak Test*** Activation Test Flow Rate Test
6.2.6.1.4	Salt Corrosion Resistance Test	1 Unit (T5)	Leak Test Activation Test Flow Rate Test
6.2.6.1.5	Vehicle Environment Test	1 Unit (T6)	Leak Test Activation Test Flow Rate Test
6.2.6.1.6	Stress Corrosion Cracking Test	1 Unit (T7)	N/A
6.2.6.1.7a	Drop Test	1 Unit (T8)	Vibration Test
6.2.6.1.7b	Vibration Test	1 Drop Tested Unit (T8); Plus 1 New Unit (T9)	Leak Test Activation Test Flow Rate Test
6.2.6.1.8	**Leak Test**	7 Tested Units (T1, T3, T4, T5, T6, T8, T9); Plus 1 New Unit (T10/V15)	Activation Test
6.2.6.1.9	**Bench Top Activation Test**	6 Tested Units (T1, T4, T5, T6, T8, T9); Plus 3 New Units (T11, T12, T13)	Flow Rate Test
6.2.6.1.10	**Flow Rate Test**	9 Units from Bench Top Activation Test (T1, T4, T5, T6, T8, T9, T11, T12, T13)	N/A
6.2.6.1.11	Atmospheric Exposure Test	1 Unit (T14)	N/A

Note: Test methods in bold are pressurized.
* Per NHTSA request, High Pressure Activation and Flow (6.2.6.1.12) not included.
** Highlighted cells indicate quantities vary from GTR NO. 13 specifications.
*** Leak Test is only at ≤ -40 °C.

Table A-2. Check Valve and Automatic Shut-Off Valve Performance Tests/Evaluations.

GTR NO. 13 Section No.	GTR NO. 13 Section Title	Sample Quantity* and (Contractor Sample IDs)	Subsequent Tests
6.2.6.2.1	**Hydrostatic Strength Test****	1 Unit (V18)	N/A
6.2.6.2.2	**Leak Test**	2 Tested Units (V17, V18); 1 New Unit (T10/V15)	N/A
6.2.6.2.3	**Extreme Temperature Pressure Cycling Test**	Engineering Evaluation	N/A
6.2.6.2.4	Salt Corrosion Resistance Test	Engineering Evaluation	N/A
6.2.6.2.5	Vehicle Environment Test	Engineering Evaluation	N/A
6.2.6.2.6	Atmospheric Exposure Test	Engineering Evaluation	N/A
6.2.6.2.7	Electrical Test	1 Unit (V16)	N/A
6.2.6.2.8	**Vibration Test*****	1 Unit (V17)	Leak Test
6.2.6.2.9	Stress Corrosion Cracking	Engineering Evaluation	N/A

Note: Test Methods in bold are pressurized.

* Highlighted cells indicate quantities vary from GTR NO. 13 specifications.

** Substituted performance of the hydrostatic strength test for the pre-cooled hydrogen test was removed from GTR NO. 13 by the committee.

*** Based on a structural evaluation for testing of energetics in vibration lab, this testing was performed with nitrogen.

The following sections describe the test methods in detail, the test performance and results (if applicable), lessons learned from the test performance/engineering evaluations, and qualitative value assessment for each test method.

3.0 PRESSURE CYCLING TEST (GTR NO. 13, 6.2.6.1.1)

One (1) TPRD unit, Contractor-purchased Sample ID T1, underwent internal pressure cycles with hydrogen gas at a rate of ≤ 10 cycles/min as specified in Table 7. The intent of this project was to review the test procedures and not identify the performance of specific valve hardware. Therefore, while the test method requires five (5) thermally activated pressure relief devices (TPRD) be subjected to the test, this project tested one (1) TPRD. Furthermore, the cycle quantity was reduced due to schedule and budget constraints that were estimated based on the maximum allowable cycle rate. The contractor's test stand and associated equipment was limited to a slower cycle rate, so the reduced scope was proposed by the contractor and approved by NHTSA. The sample was installed in a temperature-controlled chamber with a temperature recording device to verify the test temperature. Hydrogen gas was cycled using a dedicated pressure control system to pressurize and relieve the TPRD safely and accurately. Pressure was monitored and recorded using a calibrated pressure transducer.

Table A-3. Pressure Cycling (6.2.6.1.1) Conditions (from GTR NO. 13).

The modified pressure cycle conditions enabled timely completion of the test procedure without removing a target pressure condition or the extreme temperature conditions.

Pressure Cycle Description	Sample Temperature	No. of Cycles	
		Reduced Scope	GTR NO. 13 Full Scope
≤2 MPa to ≥150% NWP	≥85°C	First 10	First 10
≤2 MPa to ≥125% NWP	≥85°C	448	2,240
≤2 MPa to ≥125% NWP	20°C	Removed	10,000
≤2 MPa to ≥80% NWP	≤-40°C	550	2,750

Note: All cycles are at a rate of ≤ 10 cycles/min.

After the series was complete, the unit underwent the Leak Test (6.2.6.1.8), the Bench Top Activation Test (6.2.6.1.9), and the Flow Rate Test (6.2.6.1.10).

3.1 Test Performance and Results

The Pressure Cycling Test was performed in general accordance with GTR NO. 13, Section 6.2.6.1.1, and was conducted by the contractor's Fluids Engineering Department. One TPRD unit underwent pressure cycling test from September 28 – October 7, 2022. According to GTR NO. 13 Section 6.2.6.1.1, the pressure cycling consists of five TPRD units undergoing internal pressure cycles for 15,000 internal pressure cycles with hydrogen gas at a rate of ≤ 10 cycles/min, but the quantity of cycles was modified to consist of five days (i.e., more than 1,000 cycles total of testing at 85°C and -40°C with no ambient temperature testing) with only one TPRD test article.

For the testing performed, no leaks were detected. While the focus of this project is to evaluate the test procedure and not the specific test specimen, **the TPRD tested met the requirements of GTR NO. 13 Section 6.2.6.1.1, Pressure Cycling Test (reduced scope).** The subsequent Leak Test, Bench Top Activation Test, and Flow Rate Test results are provided in Sections 10.0 – 12.0. No leakage was measured by the flow meter on the outlet line during the leak testing at ambient, elevated, and cold temperature. However, the TPRD exceeded the allowable activation time, so did not meet the requirements of GTR NO. 13 Section 6.2.6.1.9. The mass flow rate during the GTR NO. 13 Section 6.2.6.1.10 Flow Rate Test was the highest for this sample at 0.448 kg/min. This flow rate was high compared to the other samples tested and resulted in the majority of the other samples not meeting the performance criteria (i.e., the lowest measured flow rate shall not be less than 90% of the highest flow rate). Since this sample did not meet the requirements of the Bench Top Activation Test, the contractor suggests the Flow Rate Test results not be used for performance testing purposes of the other samples that met the requirements for the other post-conditioning performance tests. The following subsections discuss the lessons learned, GTR No. 13 procedure evaluations, and a qualitative value assessment for this test method.

3.2 Lessons Learned

The following list summarizes the contractor's observations, comments, and findings for the Pressure Cycling Test (GTR NO. 13, Section 6.2.6.1.1). The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- The method does not include guidance for installing an integrated valve system (i.e., finished products that contain check valve, shut-off valve, and TPRD, etc.) and is written for testing the TPRD as a separate unit.
    - *For integrated valve systems, one may want to specify that the valve should be installed per manufacturer's instructions and the shut-off valve held in the closed position.*
    - *Consideration may also be given to the possibility of combining this method with the Extreme Temperature Pressure Cycling Test (6.2.6.2.3) for shut-off valves. Combination of these two test procedures is possible because the TPRD is connected to the shut-off valve outlet (i.e., tank side), so both the TPRD and shut-off valve are exposed to the same pressure.*
- There is no required hold time at each set temperature prior to conducting the pressure cycles.
    - *It is unclear if the cycling should be started as soon as the test temperature is achieved or if there should be at least a 1 h hold time before the start of the new set temperature. Alternatively, it could be assumed that a 2 h hold time is required before the start of the cycling in accordance with the 6.2.6.1.3 Temperature Cycling Test.*
- One may want to consider the possibility of integrating the Pressure Cycle Test with the Temperature Cycling Test requirements.

- *See supporting statements in Section 5.3, Temperature Cycling Test (GTR NO. 13, 6.2.6.1.3), Lessons Learned.*

- Performing the Pressure Cycle Test with an inert gas instead of hydrogen gas would apply similar mechanical stresses while reducing safety hazards.
  - *Hydrogen gas increases the cost and risk of testing and could be minimized to methods with evaluation criteria that are dependent on the gas type. Pressure cycling is an evaluation of mechanical stresses which could be applied by another gas or liquid media.*
  - *Alternatively, the Pressure Cycle Test method may still benefit from testing with hydrogen gas since there is not a method in the primary closures test series that directly evaluates hydrogen embrittlement of the metallic components. The unit could be subjected to non-destructive evaluation after this pressure cycling test to assess for hydrogen embrittlement or other damage, prior to the post-test leak, activation, and flow evaluation series.*
  - *Another option is to complete the pressure cycle test with an inert gas and add a separate test to assess hydrogen embrittlement with a simplified test procedure and coupon test sample.*
- The pressure fell below the 1 MPa threshold in-between the pressure cycles due to the response time of the pressure cycling control system and quick vent rate. A remotely actuated control valve installed on the vent line would have allowed the operator to adjust the vent rate after the pressure cycling sequence began.
  - *The valve passed the requirements of the pressure cycling test even though the pressure was vented below the 1 MPa threshold in between pressure cycles. Since venting lower than required did not appear to have an effect on the performance of the tested valve after completing 1,000 pressure cycles, one may want to consider revising the low-pressure target to be less than 2 MPa as opposed to between 1 MPa and 2 MPa. This revision would still test the TPRD at the extreme pressure conditions but would simplify the level of control required at low pressures for the test facility. Therefore, it would reduce the complexity of the test equipment while not significantly affecting the stringency of the standard.*

3.3 Test Method Cost Assessment

Pressure cycling is an important evaluation to assess a unit's ability to withstand pressure fluctuations and assess the product performance over accelerated aging conditions. The addition of extreme temperatures further challenges the test unit. This method is more strict than other pressure cycling test methods that the contractor routinely performs, because the quantity of cycles that are performed above the NWP. A test facility will need to consider costs associated with the test fixture, infrastructure and operation, safety planning if using hydrogen gas, and consumable material costs as discussed below.

To complete the pressure cycles in its entirety with the contractor's current test stand design, it is estimated that 40 days of continuous 24-h operation would be required. The cost to complete the pressure cycle testing, after the test stand is fully commissioned, is estimated to be $150,000 assuming that the system would require supervision while in operation, as was required with the current test stand design. It is possible that other test stand designs may be less costly to operate. Due to the dynamic nature of the testing, designing a system to safely operate autonomously without any personnel oversight, such as would be required for overnight operations, would be challenging and increase the setup costs. Additionally, the target thermal conditions during the testing would require autonomous thermal control systems for unmanned operation. The cost estimated above only includes the labor effort for test operation and does not consider additional material purchases that may arise. The rough cost for designing, fabricating, and commissioning a new test facility to complete this portion of the testing is estimated at approximately $300,000. It is possible that other data acquisition, automation, and test facility designs could result in lower initial capital costs, but those characteristics are unknown.

A significant part of the project set-up expenses, estimated at $50,000 for labor and materials, went towards designing, fabricating, and inspecting a test fixture to interface with the test article. Careful design and inspection of the test fixture is important to ensure that it can withstand the same integrity testing without failure. If the test article is not provided with a fixture to apply pressure to the device, then the test facility will spend a significant amount of time and resources on the test fixture design and fabrication.

In the contractor's opinion, the financial cost and duration of testing is high in comparison to the confidence gained in the test article design by testing more than one sample. Therefore, reducing the number of test articles required to undergo testing would result in significant financial savings at relatively little cost. Alternatively, the number of pressure cycles could be reduced for the additional test articles after the first test article passes the test in its entirety.

Test stand components such as tubing, fittings, valves, and instruments, are exposed to the same stresses as the test article, and consideration is needed for replacement or repair throughout the pressure cycle testing, which could also result in additional expenses and schedule delays. Given the current test stand design, it can be estimated that additional material expenses for component replacements and repairs throughout the pressure cycle test duration would total between $5,000 - $10,000. This estimate does not include the labor hours to perform the repairs, solely the material purchases; and considers routine maintenance only, not catastrophic failure of major components. To cost-effectively complete the testing, a different test stand design and method may need to be considered to expedite the pressure cycling. The contractor's test stand uses a positive displacement booster pump to achieve the desired test pressure and was designed to reduce cycling time by minimizing the system volume. To further reduce the system volume would be challenging and would likely lead to reduced pressure control and enhanced pump pulsations. Therefore, another means of pressure application may need to be considered.

Another option to reduce the testing burden would be to perform the testing with an inert gas. The contractor has existing infrastructure to safely perform this testing with an inert gas inside of a high-pressure test cell, that would facilitate unmanned operation and significantly reduce test cost. It is likely that existing infrastructure for high-pressure integrity testing with inert gases will be more readily available than systems designed for high-pressure hydrogen testing. Future standard organizations may choose to use an inert gas, should the standard organization determine that the inert gas evaluates pressure cycling performance similarly to hydrogen gas while providing a cost savings.

3.4 Test Method Value Assessment

Completing the pressure cycle test procedure in its entirety exposes the test article to very extreme conditions when compared to the other test procedures. In terms of increasing confidence in the integrity of the test article design, this test method could identify future test CHSS test specimens with a higher than appropriate risk of failure. The probability of failure when used in the field is significantly reduced, assuming the number of pressure cycles and thermal extremes are reflective of the conditions that the test article is exposed to during its life cycle. Additionally, the severity of injuries or property damage that would result due to a catastrophic failure of the test article at elevated pressures and extreme temperatures is very high. Although significant value would be added by completing this testing in its entirety for one test article, the added value for repeating the testing on additional test articles of the same design is far lower. Therefore, one may choose to reduce the number of test articles required to complete the pressure cycle test as the cost benefit may outweigh the added value for repeated tests on identical designs. Similarly, significant value would still be added by completing this testing in its entirety with an inert gas such as nitrogen gas, provided that the testing is followed up by a leak test with hydrogen gas. If a test lab uses an inert gas, the safety benefit added by completing the pressure cycle test would still be ranked as high by the contractor, when followed with the hydrogen leak test procedure.

4.0 ACCELERATED LIFE TEST (GTR NO. 13, 6.2.6.1.2)

Two (2) TPRD units were subjected to testing; one (1) at the manufacturer's specified activation temperature (T_{act}) the contractor Sample ID T2, and one (1) at an accelerated life temperature (T_L) the contractor Sample ID T3, given in °C by the following equation:

$$T_L = \left(\frac{0.502}{\beta + T_f} + \frac{0.498}{\beta + T_{ME}}\right)^{-1} - \beta$$

Where $\beta = 273.15$, $T_{ME} = 85$ °C, and T_f = manufacturer's specified activation temperature (°C). The sample quantity was decreased from eight (8) TPRD units as specified in GTR NO. 13 (three at T_{act} and five at T_L). The TPRD unit was placed in a liquid bath with the temperature held constant (±1 °C), and the unit was then pressurized with hydrogen gas on the TPRD inlet to ≥ 125 % NWP. GTR NO. 13 states that the TPRD tested at T_{act} shall activate in less than 10 h, and the TPRD tested at T_L shall not activate in less than 500 h and shall meet the requirements of the Leak Test (6.2.6.1.8). The liquid bath temperature and hydrogen pressure were monitored and recorded throughout each test.

4.1 Test Performance and Results

The Accelerated Life Temperature Test in accordance with GTR NO. 13, Section 6.2.6.1.2, was conducted by the contractor's Fluids Engineering Department. The first test evaluated the TPRD activation function at the activation temperature (T_{act}) of the device and was performed on August 2, 2022. The second test evaluated the TPRD activation function at the accelerated life temperature (T_L) and was performed August 12, 2022 – September 2, 2022. During the TPRD activation temperature test, the TPRD successfully activated and released pressure within 10 hours at an activation temperature of 102.8°C (217°F) and a pressure of 90.3 MPa (13,114 psig). This was slightly below the manufacturer's reported activation temperature range, and likely due to the contractor's temperature measurement location during the test (on the test fixture, near the center of the thermal bath; rather than near the TPRD location which was closer to the thermal bath heating element which was likely at a higher localized temperature). During the TPRD accelerated life temperature test, the TPRD did not activate in less than 500 hours at the accelerated life temperature of 94.8°C (202.6°F), and no leakage was measured during the subsequent Leak Test (GTR NO. 13 Section 6.2.6.1.8).

The TPRD tested met the requirements of GTR NO. 13 Section 6.2.6.1.2, Accelerated Life Test and the subsequent GTR NO. 13 Section 6.2.6.1.8 Leak Test. The following subsections discuss the lessons learned, GTR No. 13 procedure evaluation, and a qualitative value assessment for this test method.

4.2 Lessons Learned

The following list summarizes the contractor's observations, questions, comments, and findings for the Accelerated Life Test (GTR NO. 13, Section 6.2.6.1.2). The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- One may want to consider the possibility of performing this test method with an inert gas or liquid, instead of with hydrogen gas.
  - *Hydrogen gas increases the cost and risk of testing and should be minimized to methods with evaluation criteria that are dependent on the gas type. The contractor believes this test method could be performed with an inert gas because the TPRD activation is solely dependent on temperature. A liquid is not recommended as the test fluid since this may change the heat transfer within the sample.*
  - *Damage to the integrity of the test article would be discovered during the post-test Leak Test that is conducted with hydrogen gas.*
- The temperature measurement location is not specified in the GTR NO. 13 method.
  - *There is a potential for a temperature gradient within the liquid bath. The activation temperature measured during testing was slightly lower than the manufacturer's published range and was likely due to the location of the thermocouple. The thermocouple was located at the center of the liquid bath rather than on/near the TPRD which was closer to the heating coil within the bath and likely at a higher temperature. Directly requiring temperature measurement on the TPRD surface would improve the accuracy of the TPRD exposure temperature during testing.*
  - *One may want to consider recording multiple temperature measurements within the liquid bath to capture disparities in temperature at different locations, in addition to adding adequate mixing to reduce thermal gradients within the fluid.*
- There is a risk of high hydrogen gas concentrations around the test stand components after the TPRD is activated.
  - *If the test lab does not route the tubing from the outlet port of the TPRD away from the test stand for venting, the local hydrogen concentration could reach dangerous levels upon activation.*
  - *Routing the outlet of the TPRD away from the test stand is important, but caution should be taken to avoid closing off the vent path downstream of the TPRD outlet. The*

TPRD is not designed to hold pressure, so if any pressure builds in the outlet line, there is potential for large external leakage at the TPRD location.

  - *Purging the TPRD outlet line with nitrogen gas is prudent to prevent interaction of pressurized hydrogen gas with air. However, be aware that even a low-pressure nitrogen purge may result in external leakage at the TPRD, as was the case during this testing. A local nitrogen leak may pose a safety risk to the test operators by displacing the air in the test stand area.*
  - *In order to limit risks, the contractor monitored and controlled the testing remotely from a control room and added sensors around the test setup to monitor hydrogen concentration.*

- Deviation from the manufacturer's expected activation temperature is possible and could result in unintentional activation of the TPRD during other test procedures, such as the Temperature Cycling Test (GTR NO. 13, 6.2.6.1.3).
  - *Conducting the TPRD activation temperature testing first would determine the exact activation temperature of the TPRD using the given test stand configuration and instrumentation. This would help prevent unintentional premature TPRD activation during the other test procedures. It would also give the test operators an opportunity to observe the effects of TPRD activation and make any adjustments to the test stand, as necessary, to promote safety.*
  - *For example, after the TPRD activation temperature testing, the contractor changed the test procedure for the remaining tests to ensure that the isolation valve downstream of the TPRD outlet port was left open for the remainder of the testing, preventing the TPRD from seeing high pressures once activated.*
- There is potential for test article o-ring compatibility issues with the bath fluid.
  - *The seal provided with the test article at the location where the test article threads into the fixture was composed of Vinyl Methyl Silicone (VMQ) and was incompatible with the fluid in our high-temperature bath, Shell Diala. Although the integrity of the seal was not compromised during the activation temperature testing, the o-ring was swollen and loose when the test article was removed from the fixture.*
  - *In order to complete the test procedure safely, it is necessary to ensure o-ring chemical compatibility and stay within the temperature ratings of the seal material.*

- The test procedure does not directly state that the 500 h period during the testing at T_L may be separated into non-continuous periods that total 500 h.
    - *The contractor is not aware of a benefit to ensuring that the 500 h testing period during the testing at T_L is continuous. It is highly likely that there will be periods during which the temperature or pressure drift out of the allowable tolerance due to unforeseen leaks, test stand failures, or atmospheric weather conditions for outdoor test stands. Additionally, if a leak develops on the test stand, testing will need to be paused to locate the source of the leakage and fix the leak prior to proceeding. Directly stating that the testing period may be paused and resumed will improve clarity if/when these events occur.*

4.3 Test Method Cost Assessment

The Accelerated Life Test requires testing with high-pressure hydrogen gas at elevated temperatures. The majority of the test cost is associated with commissioning a facility to safety perform the testing. Similar to the Pressure Cycle Test, designing and commissioning a new facility for this testing is estimated to cost $300,000. Alternatively, if the facility is already commissioned, the cost for performing the Accelerated Life Test is estimated at $20,000. This test procedure is much simpler than the Pressure Cycle Test and will require less automation in the facility controls. However, due to the test duration, advanced safety controls will be important to facilitate unmanned test operation.

4.4 Test Method Value Assessment

The method is valuable from both a safety perspective (verifying the TPRD will relieve at the manufacturer's specified activation temperature), but also from a product reliability standpoint (proving the TPRD will not relieve at an elevated temperature below the manufacturer's specified activation temperature). For integrated valve samples, the sample quantity could become burdensome since a custom test fixture would be required to pressurize the TPRD. Due to the pressure requirements and hydrogen gas compatibility, this test fixture will likely be expensive to design and fabricate, and if only one test fixture is available it will require this test method to be performed in series. In order to perform the full quantity of samples, this could take up to 2,530 h test time (>105 days; three samples at T_{ACT} for up to 10 h each, and five samples at T_L for 500 h each; does not include setup/change over time). Completing 10 h of testing at T_{ACT} does not require much time, so reducing the number of samples tested at T_L would result in the largest cost savings. Additionally, the pressure cycling test procedure already includes lengthy exposure of a test article to elevated temperatures below the activation temperature.

5.0 TEMPERATURE CYCLING TEST (GTR NO. 13, 6.2.6.1.3)

One (1) TPRD, the contractor Sample ID T4, unit underwent temperature cycling in which the unpressurized unit was placed in a liquid bath maintained at ≤ -40 °C for at least 2 h, and then transferred to a liquid bath maintained at ≥ 85 °C for at least 2 h (transfer time ≤ 5 min each). This cold-hot cycle was repeated 15 times, followed by a final -40 °C exposure for at least 2 h. The internal pressure of the TPRD was then cycled with hydrogen gas between ≤ 2 MPa and ≥ 80 % NWP for 100 cycles while the liquid bath was maintained at ≤ -40 °C (assuming at a rate of ≤ 10 cycles/min though not specified in GTR NO. 13 procedures and will be added to Lessons Learned).

GTR NO. 13 Section 6.2.6.1.3 does not have specific requirements that must be met for the tested TPRD. However, after the thermal and pressure cycling, the TPRD shall comply with the Leak Test (6.2.6.1.8) but conducted only at the -40 °C temperature. Then, the TPRD shall comply with the Bench Top Activation Test (6.2.6.1.9) and then the Flow Rate Test (6.2.6.1.10). Liquid bath temperature and hydrogen pressure were monitored and recorded throughout the testing.

5.1 Test Performance and Results

The temperature cycling test was performed from March 28 – April 7, 2022. During this testing, the test article underwent 15 temperature cycles at two different temperature extremes: ≥ 85°C and ≤ -40°C. During the unpressurized thermal cycling, the TPRD was activated prematurely. Consequently, the pressure cycles at ≤ -40°C, the leak test, and the benchtop activation test could not be performed; however, the flow rate test was performed.

The TPRD test per GTR NO. 13 Section 6.2.6.1.3, Temperature Cycling Test, is discussed in Section 5.0. Due to the premature activation, it is not possible to deduce the pass/fail performance of the tested unit. The results of the subsequent Flow Rate Test was 0.4 kg/min, which was the average of all samples tested. The following subsections discuss the lessons learned, GTR No. 13 procedure evaluations, and a qualitative value assessment for this test method.

5.2 Lessons Learned

The following list summarizes the contractor's observations, questions, comments, and findings for the Temperature Cycling Test (GTR NO. 13, Section 6.2.6.1.3). The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- The TPRD's thermal mass affects the liquid bath temperature when inserted. One may want to specify an allowable duration of time to re-stabilize the liquid bath temperature, and/or may want to suggest that the liquid bath temperature may be lower/higher initially to compensate for the effect of the cooled/heated TPRD sample (within certain limits).

  - *The contractor assumes the intent of this method is to thermally shock the test unit. Transferring the test unit from either extreme temperature will affect the localized bath temperature at the unit. To maintain compliance with the standard, test facilities will need to design large thermal baths and thermal systems with enough power to overcome this local temperature gradient. A 5 °C band on the bath temperature is challenging and will likely result in nonconformances with the testing standard due to the thermal mass from the TPRD when inserted. The cost and effort associated with commissioning the hot and cold thermal systems will increase to successfully stay within the current 5 °C temperature band. A larger allowable temperature band (e.g., 15 °C) could be allowed while keeping the extremes of the thermal shock the same (i.e., >85 °C and <-40 °C) and would be easier for test facilities to achieve.*
  - *Alternatively, one may consider revising the procedure to suggest preheating or precooling of the temperature bath to remain within tolerance after the test unit is inserted. However, this increases the likelihood of premature TPRD activation during the high-temperature cycles which would prevent completion of the full GTR NO. 13 test requirements for that unit. If one decides to preheat or precool the temperature bath, then one may consider adding an additional test bath temperature maximum/minimum tolerance, which would identify a potential test anomaly should test bath exceed the tolerance, because a preheated or precooled temperature bath could result in premature activation or damage to the test unit.*
  - *Retesting due to nonconformance can be expensive. Consequently, one may also consider adding guidance on how to proceed if a nonconformance occurs and the temperature of the unit falls outside of the allowable temperature band, such as repeating the entire test program on a new unit or adding thermal cycles on the same unit.*
  - *The contractor assumed that the exposure duration (≥ 2 h) was initiated when the thermal bath reached the target temperature. one may want to directly state that the exposure duration starts once the bath has recovered to the target temperature range.*
- The main o-ring and back-up o-rings may be damaged prior to the pressurized leak, activation, and flow rate tests.
  - *The contractor chose to remove the main o-ring and back-up rings from the valve body prior to performing the temperature cycles, because the contractor staff didn't want to subject the seal material to the extreme temperatures and risk damaging it prior to the pressurized leak, activation, and flow rate tests.*

- *This was not specified in the test method; possibly because the method is written for component testing rather than the integrated valve system that was tested. One may want to consider providing guidance for the testing of integrated systems and/or a note to alert test labs to protect portions of the valve that are internal to the tank in normal operation.*

- This method involves temperature and pressure cycling similar to those of GTR NO. 13, Section 6.2.6.1.1, Pressure Cycling Test, and it seems like the two methods could be combined to fully cover evaluation of the unit's performance.
    - *The thermal shock aspect is unique to the temperature cycling test, but one may want to consider how likely the sudden thermal shock is during the life cycle of the TPRD when in service. If sudden thermal shock is unlikely in service, then there is potential to combine this testing with the pressure cycling test or to remove the requirement to change the TPRD temperature to the extremes within 5 min.*
    - *One option would be to perform the unpressurized thermal cycling (15 cycles) per 6.2.6.1.3 on the 5 units undergoing pressure cycle testing prior to performing the pressure cycling conditions specified in 6.2.6.1.1. Then, all units would be subjected to the full leak test (6.2.6.1.8), Bench Top Activation (6.2.6.1.9), and Flow Rate Test (6.2.6.1.10). This would only reduce the test units by one (1), but since the initial temperature cycles are unpressurized it wouldn't add much cost to pre-condition test units this way before performing the pressure cycles.*
- The contractor noted that the pressure cycle testing is only required at -40°C.
    - *If the -40°C condition is the most likely failure scenario, then there is potential to avoid duplicating effort and resources. One may recommend conducting this test prior to the Pressure Cycling Test (GTR NO. 13, Section 6.2.6.1.1) to save testing time and resources if the test article fails at cold temperatures.*
- The use of high-pressure hydrogen gas increases test complexity and cost.
    - *Performing the pressure cycle portion of the Temperature Cycle Test with an inert gas instead of hydrogen gas would apply similar mechanical stresses while reducing the safety hazards. A liquid is not recommended as the test fluid since this may change the heat transfer within the sample.*
    - *The contractor believes that the post-test Leak Test should still be performed with hydrogen gas, because small hydrogen molecules may leak more easily from small holes.*

5.3 Test Method Cost Assessment

Compared to the testing that requires application of high-pressure hydrogen gas, the cost to setup for the Temperature Cycle Test is relatively low. The cost for setting up and operating this testing could range from $15,000 to $60,000, depending on the test lab's existing temperature control equipment and instrumentation. Since the test procedure does not involve hydrogen gas, it is less likely the test labs will not run into difficulties finding a safe place to conduct the testing in their lab environment.

5.4 Test Method Value Assessment

Temperature and pressure cycling tests are important to evaluate the performance of a pressurized system under extreme conditions. However, both the Pressure Cycling Test (6.2.6.1.1) and Temperature Cycling Test (6.2.6.1.3) involve temperature and pressure cycling, which could possibly be integrated to lessen the testing burden while maintaining the important performance demonstration. One may want to consider the likelihood of rapid thermal shock in service when determining the time requirement for switching between the hot and cold extremes to prevent putting unnecessary burden on the test facility and test article. If the Pressure Cycle Test and Temperature Cycling Test are combined, the quick temperature cycling requirement should not be extended to the pressurized tests because it will put additional stresses on the test article and facility that would not be seen if these tests are performed sequentially (i.e., quick unpressurized thermal cycles followed by slower pressurized thermal cycles).

6.0 SALT CORROSION RESISTANCE TESTS (GTR NO. 13, 6.2.6.1.4 AND 6.2.6.2.4)

One (1) TPRD unit, the contractor Sample ID T5, was subjected to the accelerated cyclic corrosion test as shown in Figure 8 (The contractor provided this revised flow diagram for NHTSA consideration), for a total of 100 cycles. The sample quantity has been decreased from three (3) TPRD units specified in GTR NO. 13, per NHTSA request. A total of four salt mist applications were applied during the ambient stage (first at the beginning, and each subsequent applied approximately 90 min after the previous application). The salt solutions (% by mass) were 0.9 % Sodium Chloride (reagent or food grade), 0.1 % Calcium Chloride (reagent grade), and 0.075% Sodium Bicarbonate (reagent grade) and balance water (ASTM D1193 Type IV). Only the exterior surfaces of the TPRD was exposed to the test environment. Chamber temperature and humidity were monitored and recorded. Salt solution mixing was documented.

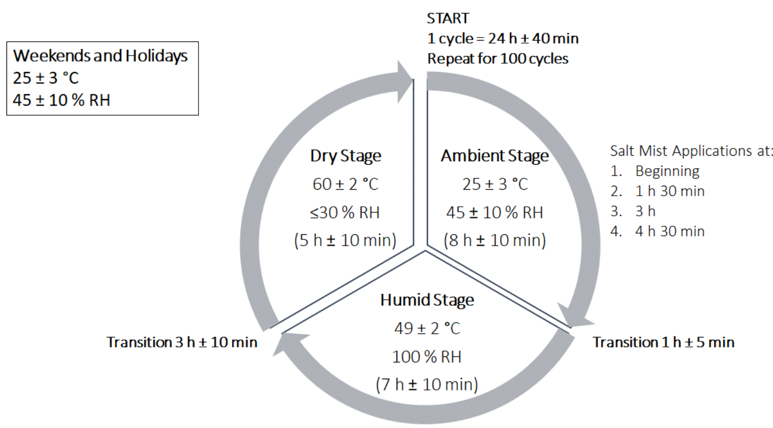

Figure A-1. Accelerated Cyclic Corrosion Flow Diagram (from GTR NO. 13 adapted by the contractor).

After the series was complete, the unit underwent the Leak Test (6.2.6.1.8), the Bench Top Activation Test (6.2.6.1.9), and the Flow Rate Test (6.2.6.1.10).

An engineering evaluation of the check valve/shut-off valve test method (6.2.6.2.4) was performed. The valve testing consists of three (3) component samples with post-test rinse, visual inspection for physical degradation, leak test (6.2.6.2.2), and hydrostatic strength test (6.2.6.2.1); otherwise, the two methods are identical.

6.1 Test Performance, Results, and Engineering Evaluation

After exposure, salt deposits were noted inside the bleed device and manual valve locations as well as along the joint between the specimen body and the TPRD vent block. Minor corrosion rust was noted in the bolt on block. No other corrosion areas were affected. **The TPRD tested met the requirements of GTR NO. 13, Section 6.2.6.1.4, Salt Corrosion Resistance Test**. The subsequent Leak Test, Bench Top Activation Test, and Flow Rate Test results are provided in Sections 10.0 – 12.0.

In reviewing GTR NO. 13, Section 6.2.6.2.4, for engineering evaluation it was noted that the salt corrosion exposure conditions are identical for the TPRD and Shut-off/Check Valve test methods. Both require the component samples to be installed in accordance with the manufacturer's recommended procedure and exposed to the cyclic corrosion test method illustrated in the flow diagram shown in Figure A-1. In order to accomplish this for an integrated valve system, the portion of the valve that is installed within the tank and the I/O port is protected from the test environment. The contractor is unsure how the shut-off/check valve would be affected by this exposure since the majority of these components are internal to the valve. The external shut-off valve components include the solenoid and manual valve control. The solenoid is an enclosed assembly that uses an electromagnetic force to move an encased armature, and for this sample is a normally closed valve. The manual valve external control may have the potential to corrode and effect the ability to operate the valve. The check valves are all internal and would not be affected by this exposure.

For integrated valve systems, the TPRD and shut-off valve external components could be exposed to the salt chamber environment simultaneously. However, the post-test performance tests are slightly different for these components so would still require three (3) samples per test method. The TPRD must comply with the requirements of the Leak Test (6.2.6.1.8), Bench Top Activation Test (6.2.6.1.9), and Flow Rate Test (6.2.6.1.10). Whereas the shut-off valve must comply with the requirements of the Leak Test (6.2.6.2.2) and the Hydrostatic Strength Test (6.2.6.2.1). The Leak Tests (6.2.6.1.8 and 6.2.6.2.2) are slightly different but could possibly be integrated to evaluate both components on the same test sample. However, the Bench Top Activation Test (6.2.6.1.9) and Hydrostatic Strength Test (6.2.6.2.1) could compromise the ability for the test sample to contain pressure and/or be destructive to the sample. For this reason, with the current test methods it is recommended to evaluate the components on separate test samples. The salt chamber is sufficiently sized to allow simultaneous exposure of six (6) integrated valve systems (three (3) samples per test method), which streamlines the overall test schedule since this exposure is 100 days long.

The following subsections discuss the lessons learned, GTR No. 13 procedure evaluations, and a qualitative value assessment for this test method.

6.2 Lessons Learned

The following list summarizes the contractor's observations, questions, comments, and findings for the Salt Corrosion Resistance Tests (GTR NO. 13, Sections 6.2.6.1.4 and 6.2.6.2.4). The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- Regarding the solution type used in the salt application, the chemical grade specifications are subjective.

  - *One may want to suggest that the test protocol include a minimum purity requirement for each chemical compound for clarity.*
- The salt solution concentration tolerances are not provided.
  - *Generally, the salt solution is mixed in large batches (The contractor mixed the solution in about 160 L batches), and if the mass of each component is measured with a balance with sufficient resolution (±0.1 mg) the solution concentrations should be reasonably accurate. ASTM B117, Standard Practice for Operating Salt Spray (Fog) Apparatus, recommends a post-mixing verification of the concentration using a salometer hydrometer or specific gravity hydrometer; however, this method suggests a 5±1% by mass salt solution, whereas the GTR NO. 13 method has a 3-part salt solution that is just over 1% by mass total. One may want to require documentation of the mixing procedure to ensure consistent salt solution concentration.*
- During the ambient stage, salt is fogged inside the chamber to facilitate the corrosive conditions. The current test protocol does not have provisions for the amount of salt that is required to be deposited on the sample surface.
  - *Based on previous work by the contractor, at least 20 min of salt mist application time is needed to ensure sufficient salt is applied on the part surface and that the part has dried prior to the start of the next salt application.*
- The current test protocol establishes the interruption of the test cycle during weekend/holidays.
  - *Normally, the exposure chambers commercially available are automated and do not require personnel intervention unless there is a power shortage or other source of power interruption of the chamber. As such, the chamber can be programmed so that 100 cycles of exposure can be executed throughout the 7-day week duration. Periodic observation of the solution level inside the companion compartment is performed throughout the exposure test. For non-automated chambers, the test exposure will be interrupted during weekend/holiday periods. For clarity, one may want to specify that this time period does not count towards the cycle count.*
- The test method requirements are the same for the Salt Corrosion Resistance Tests in GTR NO. 13, Section 6.2.6.1.4 and 6.2.6.2.4, for the TPRD and Shut-off/Check Valve, respectively.
  - *For integrated valve systems, the TPRD and shut-off valve external components could be exposed to the salt chamber environment simultaneously. Assuming the salt chamber is sufficiently sized to allow simultaneous exposure of six (6) integrated valve systems (three (3) samples per test method), this would streamline the overall test schedule since this exposure is 100 days long.*
- Orientation of samples is not specified in the current test method.

- *The contractor oriented the part within the chamber so that the integrated valve system was mounted horizontally, which is likely how it would be installed in the field. Rotationally, the TPRD or valve location could be oriented so that an area of interest is pointed upward, so it is exposed to a worst-case condition where sizable amount of salt could accumulate on the surface.*

6.3 Test Method Cost Assessment

Salt corrosion test is an important evaluation to assess a unit's ability to withstand the effect of aggressive salts on the test fixture and assess the product performance over accelerated aging conditions. The contractor routinely performs this test as part of environmental testing of bare metals. The test facility will need to consider costs associated with the test fixture, infrastructure and operation, and consumable material costs. To complete the test in their entirety with current test design, it is estimated that 100 days of continuous 24-h operation would be required. The cost to complete the test is estimated to be $60,000, if the system would require supervision while in operation, as was required with the current test design. The cost estimated above only includes the labor effort and does not consider equipment purchases. Typically, purchase of a single autonomous environmental chamber can range from $50,000 to $90,000. Additional purchase of salt for the entire test period increases the cost of the test. Maintenance of the environmental test chamber (tubing, fittings, valves, pumps, and instruments) is periodically required.

6.4 Test Method Value Assessment

This accelerated aging method is valuable for both safety and product reliability, by which providing verification that salt corrosion will not affect the function and performance of the TPRD or Shut-off/Check Valve. The exposure duration is long, and consideration may be given to increase the salt solution concentration in order to reduce the period of exposure. However, this test method does not require pressurizing the sample and the test chamber is likely to be automated, so it would be possible to perform the testing in parallel with other test methods. For integrated valve systems, there is the additional potential to condition the samples simultaneously to further reduce the overall project timeline. Completing the salt corrosion test procedure in its entirety exposes the test article to the most extreme condition when compared to the other test procedures. In terms of increasing confidence in the integrity of the test article design, the contractor would rank this test method with a high safety benefit. The probability of corrosion failure due to salt exposure when used in the field may be significantly reduced. Overall, the performance benefit outweighs the cost burden, especially if parallel/simultaneous testing can be utilized.

7.0 VEHICLE ENVIRONMENT TESTS (GTR NO. 13, 6.2.6.1.5 AND 6.2.6.2.5)

One (1) TPRD unit, the contractor Sample ID T6, was exposed to common automotive fluids. Only the exterior surfaces of the TPRD are exposed for 24 h at 20 (±5 °C) to each of the following fluids:

1. Sulfuric acid – 19 vol. % in water
2. Methanol/gasoline – 5 % / 95 % of M5 fuel (ASTM D4814 compliant)
3. Windshield washer fluid – 50 vol. % methyl alcohol in water

The unit is exposed to all of the fluids in sequence and is wiped off and rinsed with water after exposure to each fluid. According to GTR No. 13, the unit shall not show signs of physical degradation that could impair the function (e.g., cracking, softening, or swelling). Cosmetic changes such as pitting or staining are acceptable. After the series is complete, the TPRD unit underwent the Leak Test (6.2.6.1.8), the Bench Top Activation Test (6.2.6.1.9), and the Flow Rate Test (6.2.6.1.10).

In addition to the TPRD testing, the contractor staff conducted an engineering evaluation of the check valve/shut-off valve test method (6.2.6.2.5). The valve testing consists of one (1) sample with post-test rinse, visual inspection for physical degradation, and then subjected to the Leak Test (6.2.6.2.2) and Hydrostatic Strength Test (6.2.6.2.1); otherwise, the exposure conditions of the two methods are identical.

7.1 Test Performance, Results, and Engineering Evaluation

The Vehicle Environment Test was performed on March 15-22, 2022, by the contractor's Materials Engineering Department. After exposure to 19 vol.% sulfuric acid, the TPRD vent port body showed a noticeable color change (black) over the entire surface. A closer examination revealed that the discoloration was restricted to the outer surface of the TRPD vent port body. The exposure to ethanol/gasoline E10 fuel did not promote further damage on the TPRD specimen. Same visual observations of the TPRD specimen were noted after exposure to 50% windshield washer solution but with the presence of traces of pink-colored formation due to the color of the solution.

While the focus of the test was to evaluate the test procedure and not the specific test specimen, **the TPRD tested met the visual inspection requirements of GTR NO. 13, Section 6.2.6.1.5, Vehicle Environment Test, and further details are discussed in Section 7.0,** and includes the engineering evaluation of the valve test procedure. The subsequent Leak Test, Bench Top Activation Test, and Flow Rate Test results are provided in Sections 10.0 – 12.0. No leakage was measured by the flow meter on the outlet line during the leak testing at ambient, elevated, and cold temperature. The TPRD activated with 2 min of the new valves during the bench top activation test.

For the engineering evaluation on the valve components, the vehicle environment test conditions are identical for these two test methods (GTR NO. 13, Section 6.2.6.1.5 and 6.2.6.2.5). Both test methods are evaluations of the external surfaces and require the inlet/outlet connections of the component to be connected or capped in accordance with the manufacturer's installation instructions. In order to accomplish this for an integrated valve system, the portion of the valve that is installed within the tank and the I/O port is protected from the test environment. The contractor is unsure how the shut-off/check valve would be affected by this exposure since the majority of these components are internal to the valve. The external shut-off valve components include the solenoid and manual valve control. The solenoid is an enclosed assembly that uses an electromagnetic force to move an encased armature, and for this sample is a normally closed valve. The manual valve external control may have the potential to corrode and effect the ability to operate the valve. The check valves are all internal and would not be affected by this exposure.

Each method, including both the TPRD and check valve / shut-off valve test procedure, requires only one sample to be subjected to the environment. Both samples could be conditioned together, assuming the exposure bath was large enough. However, the post-conditioning performance tests are slightly different for these components so would still require one (1) sample per test method. The TPRD must comply with the requirements of the Leak Test (6.2.6.1.8), Bench Top Activation Test (6.2.6.1.9), and Flow Rate Test (6.2.6.1.10). Whereas the shut-off/check valve must comply with the requirements of the Leak Test (6.2.6.2.2) and the Hydrostatic Strength Test (6.2.6.2.1). The Leak Tests (6.2.6.1.8 and 6.2.6.2.2) are slightly different but could possibly be integrated to evaluate both components on the same test sample. However, the Bench Top Activation Test (6.2.6.1.9) and Hydrostatic Strength Test (6.2.6.2.1) could compromise the ability for the test sample to contain pressure and or be destructive to the sample. For this reason, with the current test methods it is recommended to perform the post-conditioning performance tests of the components on separate test samples.

The following subsections discuss the lessons learned, GTR No. 13 procedure evaluations, and a qualitative value assessment for this test method.

7.2 Lessons Learned

The following list summarizes the contractor's observations, questions, comments, and findings for the Vehicle Environment Tests (GTR NO. 13, Sections 6.2.6.1.5 and 6.2.6.2.5). The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- The current test methods mention the word "exposed" without indication of the type of exposure, such as full immersion, surface wetting (e.g., with a sorbent media placed on the surface of the sample.

- *Material compatibility may need to be considered for the various solutions if this method is employed) or any other form of exposure.*
    - *The contractor performed TPRD testing with full immersion of the tested part.*
- The test methods the contractor referenced denotes at least 24 h of exposure time without advising on the maximum number of hours of exposure. Corrosion degradation is time dependent, so it is critical to set boundaries to the exposure time.
    - *After evaluation, NHTSA shared a newer draft of the procedure that included a table with optional tolerances for the test methods. The contractor agrees with the tolerance NHTSA proposed, +2/-0 h.*
- Paragraph (a) states, "A distinct test is performed with each of the fluids. One component may be used for exposure to all of the fluids in sequence." One may want to suggest requiring an inspection after each exposure to observe visual degradation attributed to the individual solutions.
    - *The contractor opted to test one component exposed to each of the fluids in sequence, rather than testing a separate sample for each fluid. The current test protocol allows the use of a single sample exposed to all three solutions sequentially. If it is important to identify the degradation from individual solutions, using the same sample for all the solution exposures could impair visualization. However, it may be a more challenging scenario if the overall result is the main interest. Additionally, the effect of solution sequence order may have an unknown effect on certain materials and may affect the results. The contractor included an inspection and photograph of the sample after each exposure to determine the level of degradation to each solution during the TPRD testing since a single sample was used.*
- Paragraph (c) states, "Cosmetic changes such as pitting or staining are not failures" in the current test method, but pitting corrosion can ultimately lead to cracking and part failure.
    - *Pitting corrosion is an aggressive mode of metal degradation that can lead to stress corrosion cracking if there is enough salt and internal stresses in the part. As such, one may want to consider pitting as more than a cosmetic form of corrosion or specifying the pitting characteristics that would be allowable.*

7.3 Test Method Cost Assessment

This method is in line with what the contractor performs for other clients to assess corrosion and stress corrosion cracking of metallic components. Any test facility in charge of performing this test will need to consider the costs associated with the test fixture, infrastructure and operation, safety planning, and consumable material costs. To complete the vehicle in their entirety with the contractor's current test stand design, it is estimated that 7 days of exposure plus examination would be required, at an estimated cost of about $15,000. This estimated cost includes the labor effort as well as material purchases. In the contractor's opinion, the financial cost of implementing this test is low in comparison to the confidence gained in understanding the effects of these solutions on the test fixture. The test apparatus does not involve moving parts or extreme temperatures and pressures. As such, no consideration is needed for replacement or repair of items associated with the testing. Thus, there is no additional cost to be incurred during the execution of the test.

7.4 Test Method Value Assessment

These accelerated aging methods are valuable for both safety and product reliability, by which providing verification that exposure to common automotive fluids will not affect the function and performance of the TPRD or Shut-off/Check Valve. The test method can determine the rate of corrosion, in the form of pits, if there are provisions to perform cross section analysis of the specimens at the corrosion sites. Pitting corrosion rates can range from a few microns per year to millimeters per year, depending on the component material. More importantly, pitting corrosion can trigger stress corrosion cracking if there are sufficient residual stresses accumulated on the specimen in the presence of moisture and chlorides. Stress corrosion cracking is a more aggressive form of corrosion, which could lead to component failure in relatively short times. The samples are tested unpressurized, so these methods can be performed in parallel with other test method in the series. The exposure duration is relatively short, and there is a potential to condition the samples simultaneously to further reduce the overall project timeline. Overall, the improved confidence in the test article gained through this test procedure appears to outweigh the cost burden of performing the testing, especially if parallel/simultaneous testing can be utilized.

8.0 STRESS CORROSION CRACKING TESTS (GTR NO. 13, 6.2.6.1.6 AND 6.2.6.2.9)

One (1) TPRD unit, the contractor Sample ID T7, will be exposed for at least 10 days to a moist ammonia-air mixture maintained in a glass chamber having a glass cover (test method will be evaluated regardless of whether the component contains copper alloy). The aqueous ammonia (specific gravity of 0.94) is maintained at the bottom of a glass chamber below the sample at a concentration of at least 20 mL/L of chamber volume. The sample is positioned 35 (±5) mm above the solution and supported by an inert tray. The chamber is maintained at atmospheric pressure at 35 (±5) °C. Copper alloy components shall not exhibit cracking or delaminating due to the exposure. Copper and copper alloys are susceptible to ammonia stress corrosion cracking, which can be transgranular or intergranular, depending on the environment and stress levels. The required conditions for ammonia stress corrosion to develop in copper and copper alloys are the presence of liquid ammonia (water content no more than 0.2%) in the presence of oxygen or carbon dioxide and when the operating temperature is greater than -5 °C. Chamber temperature will be recorded. Solution mixing and volume versus chamber volume was documented.

8.1 Test Performance, Results, and Engineering Evaluation

The Stress Corrosion Cracking Test was performed on March 4-14, 2022, by the contractor's Materials Engineering Department. After exposure, solution droplets were seen over the entire specimen surface. Traces of surface discoloration were noted at the three hexagonal screws located at the bottom of the specimen body, referred to as "(OTV) M5 Aging DOWEL" on the supplier drawing. The remaining parts of the specimen remained free of visible corrosion. The lack of surface corrosion suggests that the likelihood of developing stress corrosion cracking is negligible. No additional post-condition testing is required for this test specimen.

While the focus of the test was to evaluate the test procedure and not the specific test specimen, **the TPRD tested met the requirements of GTR NO. 13 Section 6.2.6.1.6, Stress Corrosion Cracking.** The methods described in GTR No. 13, Sections 6.2.6.1.6 (TPRD) and 6.2.6.2.9 (valve) describe the same conditions, and there are no post-conditioning performance requirements; so, the TPRD and check/shut-off valve could be evaluated on the same test sample.

8.2 Lessons Learned

The following list summarizes the contractor's observations, questions, comments, and findings for the Stress Corrosion Cracking Tests (GTR NO. 13, Sections 6.2.6.1.6 and 6.2.6.2.9). The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- Given that without input from a manufacturer or significant disassembly of the component, it is not possible for a test lab, such as the contractor, to determine which components contain copper/copper alloys.

- *As the test method states, this assessment can be performed whether or not is know if the unit contains copper/copper alloys. These materials would be most susceptible to this exposure test, and so corrosion cracking would be noted in the post-exposure inspection.*

- Sample positioning for the exposure is ambiguous (e.g., the sample is positioned 35 (±5) mm above the aqueous ammonia solution) and may be difficult to accomplish equivalent exposures for the TPRD and valve for integrated valve systems depending on the sample geometry.
    - *The contractor positioned the sample so that the closest surface was within the required position, but the TPRD and valve were slightly further away. This was partially for convenience since this allowed the sample to rest on a flat edge and fit within the test chamber.*
- The sample position above the solution is very specific in the current test method, but there is no mention about the orientation of the sample.
    - *The contractor is unsure if this is important or should be left vague to allow flexibility. In the proposed test method, a statement reflecting the correct orientation of the part would help clarify the procedure. As stated above, the contractor positioned the sample so that the closest surface was within the required position, but the TPRD and valve were slightly further away. At this time, the contractor does not know if the orientation affects the results.*
- These evaluations are intended for the external surface only (more clearly stated in 6.2.6.2.9); however, 6.2.6.2.9 mentions disassembly, degreasing, and reassembly prior to exposure testing.
    - *This would be very difficult for testing of fully assembled integrated valve systems and degreasing of the external surfaces should be sufficient. One may consider stating in both methods that the internal components and inlet/outlet (I/O) port should be protected/installed in accordance with manufacturer's installation instructions.*
- The test methods the contractor referenced denotes at least 10 days of exposure time without advising on the maximum number of hours of exposure. Corrosion degradation is time dependent, so it is critical to set boundaries to the exposure time.
    - *After evaluation, NHTSA shared a newer draft that included a table with optional tolerances for the test methods. The contractor agrees with the tolerance NHTSA proposed, +2/-0 h.*

- The aqueous ammonia mixture description in the current test protocol is vague. For instance, specific gravity is a function of temperature, and it is not clear what temperature value the specific gravity is determined. This makes the specification very inconvenient (and incomplete without a corresponding temperature). Additionally, aqueous ammonia can be generated in several ways.
    - *Based on the specific gravity information, it is assumed that the aqueous ammonia has the composition of ammonium hydroxide. The specific gravity of 0.94 ammonia in water (given in the current test protocol) is around 15-20 wt% of ammonium hydroxide, depending on the temperature. Therefore, 16.7 vol.% ammonium hydroxide (reagent grade) is the solution the contractor used for the stress corrosion test.*
- The ammonia solution volume to chamber volume is described as a "concentration" in the current test protocol.
    - *To improve clarity, the concentration could be referred as a "volume ratio" instead.*
- The glass cover used in the current test method can result in solution evaporation through gaps between the glass cover the glass beaker.
    - *As such, the contractor used Parafilm® placed around the opening of the beaker and affixed with a tape. The contractor notes the test method may be modified to specify the chamber should be sealed in order to mitigate the potential for solution evaporation.*

8.3 Test Method Cost Assessment

Corrosion is an important evaluation to assess a unit's ability to resist changes in the alloy properties and assess the product performance over accelerated aging conditions. This test is in line with other tests that the contractor has routinely performed for numerous clients. The test itself is fairly straightforward and does not require specialty equipment. To complete the test in their entirety with the contractor's current test stand design, it is estimated that 10 days of continuous 24-h operation would be required. The cost to complete the stress corrosion cracking testing, after the test stand is fully commissioned, is estimated to be $35,000 assuming that the test would require limited supervision. Careful design of the test setup is needed to minimize the effect of solution evaporation, which would lead to a more efficient test with minimal personnel oversight, thus, minimizing the cost of the test. The cost estimated above includes the labor effort and material purchases. No additional costs are required to perform the test.

8.4 Test Method Value Assessment

This accelerated aging method is valuable for both safety and product reliability to ensure copper alloy components do not degrade when exposed to a common vehicle fluid. The testing is relatively simple and easy to perform; however, it is difficult to determine if the sample contains copper alloy components and/or evaluate if they exhibit deterioration. This method does not require any post-conditioning performance test requirements which would help evaluate the function of the components post-conditioning. Overall, this is an inexpensive and beneficial material performance method, but modifications to the post-conditioning evaluation could enhance the utility. In terms of increasing confidence in the integrity of the test article design, the contractor would rank this test method with a high safety benefit to understand accelerated corrosion of the fixture due to ammonia. Over time, accelerated corrosion could lead to catastrophic failure of the test article that may promote severity of injuries or property damage.

9.0 DROP AND VIBRATION TESTS (GTR NO. 13, 6.2.6.1.7 AND 6.2.6.2.8)

Per GTR NO. 13, Section 6.2.6.1.7, one (1) TPRD unit, the contractor Sample ID T8, was dropped from a height of ≥2 m without restricting its motion as a result of gravity, at ambient temperature (20 ± 5 °C) onto a smooth concrete surface and allowed to bounce after the initial impact. This was repeated for a total of 6 drops of the same unit to cover the six major axes. After each drop, the sample was examined for visible damage that indicates the part was unsuitable for use (e.g., threads damaged sufficiently that part is rendered unusable). If acceptable, the drop tested unit, plus one (1) new unit, the contractor Sample ID T9, underwent vibration testing. Vibration testing consisted of 30 min along each of the three orthogonal axes (vertical, lateral, and longitudinal) at the most severe resonant frequency for each axis. This was determined by using an acceleration of 1.5 g and sweeping through a sinusoidal frequency range of 10 to 500 Hz with a sweep time of 10 min. The most severe resonance frequency was identified by a large increase in vibration amplitude measured on the test specimen). Note that when not specified by a test procedure or its supporting documentation, it is typical to define a resonance as a peak acceleration greater than two times the input excitation. Per the test procedure, if resonance is not found in this range, the test shall be conducted at 40 Hz. This testing was performed on unpressurized test units. After the series is complete, the two units will undergo the Leak Test (6.2.6.1.8), the Bench Top Activation Test (6.2.6.1.9), and the Flow Rate Test (6.2.6.1.10).

Per GTR NO. 13 Section 6.2.6.2.8, one (1) shut-off valve, the contractor Sample ID V17, underwent the vibration test. The valve was pressurized to ≥100 % NWP, sealed at both ends, and vibrated for 30 min along each of the 3 orthogonal axes (vertical, lateral, and longitudinal) at the most severe resonant frequencies (determined by acceleration of 1.5g with a sweep time of 10 min within a sinusoidal frequency range of 10-40 Hz; if resonance frequency not found within this range the test will be conducted at 40 Hz). Following this test, the sample shall not show visible exterior damage that indicates the performance of the part is compromised, and then the unit shall comply with the ambient temperature leak test (6.2.6.2.2).

GTR NO. 13 specifies the testing should be performed with hydrogen gas; however, the contractor believes that there is a strong justification for choosing to use helium or another inert gas to mitigate risk potential to personnel and facilities. This is unlikely to affect the results since the subsequent leak test will be performed with hydrogen.

9.1 Test Performance and Results

Drop testing was performed on September 19, 2022. The valve sustained minor scuffing after each of the six (6) drops. Visual inspection of the TPRD revealed no apparent damage that would affect functionality. Through a brief visual examination, the drop test unit appeared to be in an operable condition following the drop test and was therefore subjected for subsequent vibration testing. Two (2)

TPRD units (contractor IDs T8 and T9) were tested in accordance with GTR NO. 13 Section 6.2.6.1.7(b) Vibration Test on September 20 and 21, 2022. The T8 unit was subjected to drop testing prior to the vibration testing. Each test article was subjected to a resonance search for 10 min (1.5g Sine Sweep, 10-500 Hz), followed by a resonant frequency dwell for 30 min (1.5g at determined resonant frequency) for each axis. For both units, the Z Axis was tested first, followed by X Axis, and Y Axis. At the conclusion of vibration testing, no visible damage due to vibration testing was observed on either of the units tested. While the focus of the test was to evaluate the test procedure and not the specific test specimen, **the TPRD tested met the requirements of GTR NO. 13 Section 6.2.6.1.7, Drop and Vibration Test, as discussed in Section 9.0.** Following the Drop and Vibration Test, the samples were evaluated against the requirements of the Leak Test (para. 6.2.6.1.8), Bench Top Activation Test (para. 6.2.6.1.9), and Flow Rate Test (para. 6.2.6.1.10). **The samples met the requirements of these post-test evaluations, which are discussed in Sections 10.0 – 12.0.**

One (1) unit (contractor ID V17) was tested in accordance with GTR NO. 13 Section 6.2.6.2.8 Vibration Test on September 22, 2022. Before vibration was conducted in each axis, the unit was pressurized (internal to the fixture) with Nitrogen to ≥ 70 MPa. The test article was then subjected to a resonance search for 10 min (1.5g Sine Sweep, 10-500 Hz), followed by a resonant frequency dwell for 30 min (1.5g at determined resonant frequency) for each axis both in accordance with the GTR NO. 13 test method. The Z axis was tested first, followed by X axis and Y axis, respectively. At the conclusion of vibration testing, no visible damage was observed on the unit, and the valve maintained pressure during the vibration tests without leaks. While the focus of the test was to evaluate the test procedure and not the specific test specimen, **the TPRD tested met the requirements of GTR NO. 13 Section 6.2.6.2.8, Vibration Test, as discussed in Section 9.0. Following the Vibration Test, the sample was evaluated and met the requirements of the Leak Test (GTR NO. 13, Section 6.2.6.2.2)., which are discussed in Sections 10.0 – 12.0.**

9.2 Lessons Learned

The following list summarizes the contractor's observations, questions, comments, and findings for the Drop and Vibration Tests (GTR NO. 13, Sections 6.2.6.1.7 and 6.2.6.2.8). The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- Axes definitions were not specified by the GTR NO. 13 test methods nor the valve manufacturer.
    - *The contractor defined the axes for testing and reporting. It is unclear how these decisions would affect the results.*
- The type of acceleration measurements is not specified.

- *The contractor utilized single-axis accelerometers which had to be repositioned for each test axis. A pair of tri-axial accelerometers could decrease setup time and potential for human error but may increase the inertial mass of the assembly plus sensor system.*

- Paragraph (b) states, "The resonance frequency is identified by a pronounced increase in vibration amplitude."
  - *While not defined in the GTR NO. 13 test procedure, a resonant frequency is typically designated by the contractor as a frequency where the amplitude of the response of the test article is at least 2x the input energy as measured by response accelerometers.*
- The acceleration level was not defined for the resonant dwells.
  - *The contractor assumed 1.5g acceleration for the resonant dwells to match the acceleration used during the resonance sweeps.*
- Hydrogen gas increases the cost and risk of testing and should be minimized to methods with evaluation criteria that could be affected by hydrogen.
  - *The contractor believes this test method could be performed with an inert gas. A liquid is not recommended as the test fluid since this would affect the sample mass. However, it is recommended to perform post-test leak tests using hydrogen gas to more accurately evaluate the sealing integrity due to hydrogen's small molecular size.*
- Following the drop and pressurized vibration test portions, the samples are to be inspected for visible exterior damage that indicates that the performance of the part is compromised. This may not be sufficiently objective.
  - *One may consider including examples of damage that could impair functionality of the components. However, both test methods currently require post-conditioning performance tests that would indicate if the functionality of the components has been compromised by the drop/vibration testing.*

9.3 Test Method Cost Assessment

The cost to perform the drop and vibration testing is low in comparison to the test procedures that require application of high-pressure hydrogen gas. The drop and vibration tests do not require temperature control which greatly simplifies the test setup. Additionally, the test procedure is relatively simple and possible to complete within a couple of days. However, the pressurization with hydrogen gas and even an inert gas requires additional safety considerations. The estimated cost to setup and conduct this testing is $20,000. This estimate assumes that the test labs already have the existing equipment to apply the resonance frequencies to the test article.

9.4 Test Method Value Assessment

Drop and vibration testing are important abuse tests to evaluate the damage potential and reliability of components when exposed to common physical stresses. Pressurized testing adds safety concerns, and thus increases costs. The safety concerns can be partially mitigated by testing with an inert gas, which should not drastically affect the severity of the evaluation; especially if the samples are subjected to post-conditioning leak tests with hydrogen to verify the drop/vibration testing has not affected the component performance. These test methods are within the capabilities of standard environmental test lab equipment and requires minimal additional setup if the test fixture for pressurized testing has already been developed for the other tests in the series.

10.0 LEAK TESTS (GTR NO. 13, 6.2.6.1.8 AND 6.2.6.2.2)

Seven (7) tested TPRD units (contractor Sample IDs T1, T3, T4, T5, T6, T8, T9) underwent the leak test per GTR NO. 13 Section 6.2.6.1.8. One (1) out of two potential Check/Automatic Shut-Off Valve samples underwent the subsequent leak test per GTR NO. 13 Section 6.2.6.2.2. (Contractor Sample ID V17 was leak tested. V18 was not leak tested, because it failed the hydrostatic strength test.) One (1) new TPRD/Check/Automatic Shut-off Valve unit (contractor Sample ID T10/V15) underwent the leak test per GTR NO. 13, Sections 6.2.6.1.8/6.2.6.2.2. The tests were performed at ambient, high, and low temperatures, with at least a 1 h hold prior to pressurizing with hydrogen to ensure thermal stability before testing. The test procedure requires conditioning at each of the specified test temperatures at a pressure greater than or equal to 2 MPa (-0 MPa / +3.5 MPa), followed by leakage observation while immersed in a temperature-controlled fluid for a minimum period of a least 1 min at each of the test pressures. If no bubbles are observed, the sample passes the test. If bubbles are observed, the leak rate is measured and must be less than 10 NmL/hr. The test settings are as follows:

a. Ambient temperature: condition at 20 (±5) °C; test at 2 (±0.5) MPa and ≥ 125 % NWP.
b. High temperature: condition ≥ 85 °C; test at 2 (±0.5) MPa and ≥ 125 % NWP
c. Low temperature: condition at ≤ -40 °C; test at 2 (±0.5) MPa and ≥ 100 % NWP.

10.1 Test Performance and Results

The contractor's Fluids Engineering Department performed the GTR NO. 13 Section 6.2.6.1.8 Leak Test on eight (8) TPRD samples and Section 6.2.6.2.2 Leak Test on two (2) Check/Automatic Shut-Off Valve samples. The testing was conducted from October 11–21, 2022. For all of the TPRD test articles, no leakage was measured by the flow meter on the outlet line during the leak testing at ambient, elevated, and cold temperature. Likewise, for the two shut-off valve test articles, no leakage was measured by the flow meter on the outlet line during leak testing at ambient, elevated, and cold testing. **The test articles met the requirements of GTR NO. 13, Section 6.2.6.1.8 and Section 6.2.6.2.2.**

10.2 Lessons Learned

The following list summarizes the contractor's observations, questions, comments, and findings for the Leak Tests. The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- The temperature control methods and test stand design may not easily and safely permit visual observation of leakage, so measuring leakage from the TPRD and/or Shut-Off Valve outlet ports may be used as the sole leakage indicator.

- *For the low-temperature testing, the test articles were not submerged in a fluid bath but rather were sprayed by liquid nitrogen in air. This cooling method proved to be quick and controllable but would not facilitate observation of bubbles as a leakage indicator. Additionally, to improve thermal stability, the test articles were enclosed in insulation for the elevated and cold temperature testing which blocked the camera view of the test article. In the event that significant leakage occurs, test facilities may consider assembling their test stands to avoid a rise in hydrogen concentration near the test stand which is accomplished by plumbing the outlet of the TPRD or Check/Automatic Shut-Off Valve to a vent. This test stand configuration would also prevent the use of visual bubble observation as a leakage measurement technique, because the valve would not be submerged in a fluid.*

- The allowable leakage criterion is expressed in units of "NmL" which is not defined in GTR NO. 13.
    - *After initial review of GTR NO. 13, Section 6.2.6.1.8 and Section 6.2.6.2.2, there was confusion from multiple members of the project team on the meaning of this unit. It was initially assumed that "NmL" stood for nano-milliliters. Confusion could be avoided if this unit is defined in GTR NO. 13.*
- Regarding the Check/Automatic Shut-Off Valve leak testing (6.2.6.2.2), the connection setup described in this method does not seem appropriate for the shut-off valve nor the check valve. It is not the same as the TPRD test method, but seems more appropriate for that setup, "The outlet opening is plugged with the appropriate mating connection and pressurized hydrogen is applied to the inlet."
    - *If one are concerned with the shut-off valve setup description, then they may choose to reword as follows, "The valve unit is installed in a test fixture corresponding to the manufacturer's installation specifications. The unit is pressurized at the outlet (i.e., tank side), and leakage is observed or measured at the inlet. The shut-off valve should be tested in the closed position."*
    - *It is unclear whether the leak test is concerned with leaks out of the tank, or leaks into the tank, or both. If both, then one may want to require that the shut off valve be tested in the reverse as well where the inlet side is pressurized, and the outlet (i.e., tank side) is open to the thermal bath or the pressure is monitored within the test fixture.*

10.3 Test Method Cost Assessment

The majority of the cost associated with performing the Leak Tests is associated with designing, fabricating, and fully commissioning a test facility that can safely apply high-pressure hydrogen gas and control the test article temperature. Similar to the Pressure Cycle Test, the cost for setting up the facility to perform Leak Tests is estimated at $300,000. If the test facility is already commissioned, performing the Leak Testing will be a much quicker procedure to follow, estimated at 1-2 days per test article. This testing does not require as advanced facility controls since it is short in duration and would likely not require unmanned operation of the test facility. The cost to perform the Leak Tests is estimate at $10,000 per test article.

10.4 Test Method Value Assessment

This test offers valuable insight on the integrity of the TPRD and Check/Automatic Shut-Off Valve after exposure to the various test conditions. It is critical that this testing is conducted with hydrogen, or possibly helium as an inert gas substitute; substitution with other inert gases would not provide an adequate evaluation due to hydrogen's small molecular size. The simplicity of this test procedure makes it a fairly quick test that should not put too much burden on the test facility when using hydrogen gas. The idea of requiring leakage measurement at both low and high pressures is valuable and is common for leak testing. Some valves have a hard time sealing at low pressures since the high pressure helps to push the sealing mechanism closed. Additionally, the low-pressure test helps to catch failure before testing at high pressure, which would result in higher hydrogen concentrations and more significant safety risks if leakage occurs. If a new TPRD or Check/Automatic Shut-Off Valve fails the leakage testing, then it is reasonable to assume that the other units would fail after undergoing the preliminary testing according to the other sections of GTR NO. 13. Therefore, performing the hydrogen leak testing on the new test articles prior to all of the other GTR NO. 13 testing would lower the risk of investing time and resources into testing that will result in a failure, allowing the manufacturer to improve the test article design after a smaller initial testing investment. This testing sequence could be suggested in GTR NO. 13.

GTR NO. 13 as currently written only requires leakage measurement if bubbles are observed. Thus, a test facility has to be prepared to measure leakage, should it occur. If a limit on leakage is desired, a suitable unit of measurement is bubbles per minute or pressure decay over time. Alternatively, the requirement could be changed so that any visible external leakage is considered a failure. Requiring measurement of the leak rate will require the test facility to purchase and calibrate an accurate low-range flow meter that is classified for use with flammables. Additionally, there will be labor and materials required to plumb the outlet ports to the flow meter

11.0 BENCH TOP ACTIVATION TEST (GTR NO. 13, 6.2.6.1.9)

Six (6) units from earlier tests (contractor Sample IDs T1, T4, T5, T6, T8, T9), and three (3) new units (contractor Sample IDs T11, T12, T13) underwent the bench top activation test. An oven or chimney is preheated to 600 (±10) °C (no flame impingement on TPRD) and the temperature must be within the acceptable range for 2 min prior to inserting the TPRD unit. The TPRD unit is pressurized to 25 % NWP or 2 MPa (whichever is less), and then inserted into the oven or chimney while connected to the pressure monitoring line, and activation time is recorded. Activation is determined by sudden loss of pressure. The three new units must all activate within ±2 min from each other, and the tested units must activate within a period no more than 2 min longer than the baseline activation time set by the new units. Oven/chimney temperature, TRPD unit pressure, and activation time will be recorded.

11.1 Test Performance and Results

The contractor's FTD performed Bench Top Activation Test (GTR NO. 13, 6.2.6.1.9) for eight (8) samples (reduced to five (5) tested units due to premature activation of the unit that underwent the Temperature Cycling Test) on October 26, 2022. All new units activated within 18 s of each other, which meets the requirements of GTR NO. 13 Section 6.2.6.1.9 (±2 min). All but one of the tested units activated within 44 s of the average activation time of the new units, which meets the requirement of GTR NO. 13 Section 6.2.6.1.9 (±2 minutes). The contractor Sample ID T1, which was conditioned per the Pressure Cycling Test, GTR NO. 13 Section 6.2.6.1.1, activated 3 min 26 s longer than the average activation time of the new units, which exceeds the requirements. **In summary, all new TPRDs and all but one of the tested TPRDs met the requirements of GTR NO. 13, Section 6.2.6.1.9, Bench Top Activation Test.**

11.2 Lessons Learned

The following list summarizes the contractor's observations, questions, comments, and findings for the Bench Top Activation Test (GTR NO. 13, Section 6.2.6.1.9). The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- The use of hydrogen gas increases the cost and risk of testing and should be minimized to methods with evaluation criteria that could be affected by hydrogen.
    - *The contractor believes this test method could be performed with an inert gas. A liquid is not recommended as the test fluid since this would affect the heat transfer of the valve system.*
- The temperature measurement location and sensor type are not specified and may affect the results based on response time and localized temperature fluctuations.

- *The contractor used two different sensor types during testing, a 1/8-inch diameter, grounded-junction, Type K thermocouple (TC) was used for the oven temperature throughout the test series. This seemed sufficient since the response time was not critical for this location. The contractor used either the same sensor type or a 0.04-in. diameter, ungrounded-junction, Type K TC near the TPRD to indicate when the test unit was inserted to the oven. The larger sensor had a slower response time, so the contractor preferred use of the smaller sensor for this testing to improve the response time.*

- The chimney temperature is required to be at 600 ±10°C for 2 min prior to exposure, but the temperature after insertion is not specified.
    - *The contractor measured the chimney temperature and the temperature near the TPRD to indicate when the unit was inserted. The chimney temperature was maintained within the acceptable range for at least 2 min prior to insertion, but depending on how the unit was placed within the flow, and the resulting change in the flow, the localized chimney temperature could fall below the range after insertion.*

11.3 Test Method Cost Assessment

The Bench Top Activation test is relatively simple compared to the other tests that require application of hydrogen gas. A test fixture will be necessary to conduct the testing, but the fixture does not need to be rated for elevated pressures; although, it simplifies the setup cost if a fixture is already fabricated for other pressurized tests in the series. The setup cost for the Bench Top Activation test is estimated at $7,500, and the cost per test article is around $500. The test cost is fairly inexpensive because only 30 minutes is required to swap out and activate each test article.

11.4 Test Method Value Assessment

This test method is important to ensure the performance of TPRD samples pre-conditioned by other testing has not been degraded in comparison to new units. The test setup is straight-forward and utilizes the test fixture for pressurized testing which may already been developed for the other tests in the series; however, does require customization based on the unit under test (e.g., the integrated valve system tested for this project is bulky and the test fixture was cumbersome to move in/out of place to a chimney).

12.0 FLOW RATE TEST (GTR NO. 13, 6.2.6.1.10)

After the bench top activation test, the activated test units (contractor Sample IDs T1, T4, T5, T6, T8, T9, T11, T12, T13) underwent the flow rate test (units naturally cooled, but no cleaning, removal of parts, or reconditioning). The flow rate test is performed with a gas inlet pressure of 2 (±0.5) MPa, and the outlet at ambient pressure. The inlet temperature and pressure are recorded. The contractor's set-up measures the flow rate within an accuracy of ±2 % of the reading, while GTR No. 13 requires that lowest measured flow value of all TPRD units shall not be less than 90 % of the highest flow value.

12.1 Test Performance and Results

After the bench top activation test, the same six (6) tested units, and three (3) new units underwent the nitrogen flow rate test performed by the contractor's Fluids Engineering Department (contractor Sample ID T4 did not undergo the activation test since it prematurely activated during the Temperature Cycling Test). Prior to the nitrogen flow rate test, the test articles were naturally cooled but did not undergo any cleaning, removal of parts, or reconditioning. The flow rate test was performed with a gas inlet pressure of 2 (±0.5) MPa and ambient outlet pressure. The inlet temperature and pressure were recorded. This testing was conducted on October 27, 2022. The flow rate was measured with a Coriolis flow meter, and the average flow rate for each test article was approximately 0.4 kg/min of nitrogen gas.

GTR NO. 13, Section 6.2.6.1.10 states that the minimum flow rate measured during the flow tests should not be less than 90% of the maximum flow rate measured when comparing the flow performance of each test article. These requirements were not met. The maximum average flow rate was measured for test article T1 at 0.45 kg/min, while the minimum average flow rate measured was 0.39 kg/min for test article T13. Based on the GTR NO. 13, Section 6.2.6.1.10, the flow rate for all of the test articles should have been above 0.4 kg/min, 90% of the maximum average rate of 0.45 kg/min. However, looking at all of the test articles and excluding test article T1, the requirements were met with the second highest average flow rate being 0.42 kg/min for test article T8. The upstream pressure was maintained within the allowable tolerance throughout the duration of the 1-minute flow period, and the temperature of the nitrogen gas flow stream varied from approximately 9°C to 13°C. GTR NO. 13, Section 6.1.6.1.10 did not include an allowable tolerance for the nitrogen gas or test article temperature during the flow testing. **In summary, the requirements of GTR NO. 13, Section 6.2.6.1.10, Flow Rate Test, were not met.**

12.2 Lessons Learned

The following list summarizes the contractor's observations, questions, comments, and findings for the Flow Rate Test (GTR NO. 13 Section 6.2.6.1.10). The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- Planning for the flow test would be simplified if a range of expected flow rates through the activated TPRD was provided by the test article manufacturer.
  - *It is challenging to plan for a flow test without knowing the approximate flow rate. For this testing project, the contractor relied on previous data that we captured during another project to approximate the flow rate which allowed us to size an appropriate flow meter and ensure our nitrogen source could keep up with the flow while maintaining the target inlet pressure. Without an initial idea of the flow rate, the risk of needing to redesign the test stand to meet the flow accuracy and target pressure requirements increases. If the TPRD outlet flow rate is not expected to vary much for different TPRD designs, a flow rate range could be provided in GTR NO. 13 for guidance. If the sizes and target flow rates through the TPRD will vary for each design, a note could be added that encourages the test article manufacturer to provide an expected flow rate to the test facility.*
- The flow rate requirement appears (i.e., lowest flow rate must not be lower than 90% of the highest flow rate) to be inconsistent with other industry standards, like ANSI ISA 75.02.01.
  - *For standardized flow coefficient testing according to ANSI ISA 75.02.01, the measured flow coefficient must be within 5% of the average measured flow coefficient for applicable valve designs. This requirement makes it clearer on which of the test articles is the outlier if the requirement is not met. With the performance criteria as written, it is unclear if the entire suite of test articles is not in compliance or if just the test articles that measured at the highest and/or lowest flow rate is not in compliance. Comparing the measured flow rate through each test article with the average flow rate through all of the test articles would be a suitable and more common performance metric, resulting in consistency between the test articles.*
- The procedure does not list the mass flow rate significant digits.
  - *For the testing performed by the contractor, if only one significant digit was used to display the results, then the measured flow rate through the activated TPRD would have been the same for all test samples. However, the data was provided using three significant figures, so the requirement that the lowest flow rate must not be lower than 90% of the highest flow rate was not met.*

12.3 Test Method Cost Assessment

Due to the inert gas test fluid and low target test pressures, the setup for this testing is relatively simple and does not introduce significant safety hazards. Additionally, the low flow rates through the activated TPRD do not necessitate the need for a large nitrogen source. This testing could likely be conducted using standard nitrogen gas cylinders as the gas source with a pressure regulator to adjust to the target inlet pressure. The test procedure itself is also very simple, and several test articles can undergo the Flow Rate Test within one working day. The largest cost associated with this testing is the purchase and calibration of an accurate flow meter for measuring the flow rate through the activated TPRD. If the test facility already has a flow meter, then the cost for setting up and operating the Leak Tests on multiple test articles is estimated at $10,000 to $20,000.

12.4 Test Method Value Assessment

This test method is very simple in comparison to the other test procedures and provides cost-effective and valuable insight into the flow performance behavior of the TPRD once activated. It is not necessary to perform this testing with hydrogen gas since the purpose is to characterize the flow performance behavior. However, the requirements may need revising to ensure the objectives of the test are met. To better correlate the flow performance through the TPRD with nitrogen gas to flow performance with hydrogen gas, the test facility could determine the flow coefficient instead of simply measuring the flow rate. This value is dependent on the upstream pressure, upstream temperature, differential pressure across the valve, specific gravity of the gas, and flow rate through the valve. The measured flow coefficient value could then be used to understand the predicted flow rate at different service conditions with hydrogen gas.

13.0 ATMOSPHERIC EXPOSURE TESTS (GTR NO. 13, 6.2.6.1.11 AND 6.2.6.2.6)

One (1) TPRD unit, the contractor Sample ID T14, underwent the atmospheric exposure test. This test evaluates the non-metallic materials exposed to the atmosphere during normal operating conditions or that provide a fuel containing seal. These parts shall not crack or show visible evidence of deterioration after exposure to oxygen for 96 h at 70 °C at 2 MPa in accordance with ASTM D572; as well as exposure to ozone when tested in accordance with ASTM D1149.

13.1 Test Performance, Results, and Engineering Evaluation

The Atmospheric Exposure Test was performed on August 1–7, 2022, by Akron Rubber Development Laboratory, Inc. (ARDL), in Akron, Ohio. The contractor did not have the capability to perform the testing required, and ARDL is an A2LA accredited laboratory for these methods. The results were analyzed and are reported by Southwest Research Institute's Fire Technology Department, located in San Antonio, Texas. Based on the testing and results obtained, the contractor performed the engineering evaluation of the check/shut-off valve test method. No visible signs of deterioration nor cracking were observed on the three samples tested. **The non-metallic components tested met the requirements of GTR NO. 13 Section 6.2.6.1.11, Atmospheric Exposure Test as interpreted by the contractor.**

13.2 Lessons Learned

The following list summarizes the contractor's observations, questions, comments, and findings for the Atmospheric Exposure Tests (GTR NO. 13, Sections 6.2.6.1.11 and 6.2.6.2.6). The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- This test evaluates the non-metallic materials exposed to the atmosphere during normal operating conditions or that provide a fuel-containing seal. Accessing the non-metallic materials on assembled components may require component disassembly which could affect the test methods and results.
    - *These types of tests are generally meant to be performed on slabs of the rubber/non-metallic compounds that are used for sealing within the components (e.g., o-rings, gaskets, etc.). Manufacturers seeking to test to GTR NO. 13 should order slabs of the sealing component material(s), and the lab should cut these into appropriate test strip size/shape as designated by the ISO/ASTM requirements. This also allows the mechanical stress/strain evaluations to be performed. If the intent of this test is to evaluate final products for compliance, this is more difficult, and may require disassembly of the valve to locate and remove the rubber/non-metallic sealing components and modification of the test performance and results criteria.*

- Both methods state that the tests are to be performed on non-metallic materials exposed to the atmosphere during normal operating conditions and/or provide a fuel-containing seal, but it may be challenging to correctly determine which materials fall into these categories.
    - *Determining which non-metallic materials are exposed to the atmosphere during normal operation or provide a fuel-containing seal may be difficult or impossible to determine without manufacturer input. Alternatively, one may consider evaluating all non-metallic materials within the valve system to ensure acceptable performance.*
- Compliance testing of integrated valve systems pulled from service and/or commercially procured would require disassembly and removal of the non-metallic components. Some of these components may require a special tool or removal instructions to avoid damaging the components during the removal process.
    - *Residual lubricant may affect results. One may want to consider removing residual lubricant prior to atmospheric exposure testing. The contractor does not have a recommended best practice for this removal.*
    - *The exposure specifications from standard test methods referenced may not apply to o-rings and other seals removed from these systems. If the intent is to require the atmospheric exposure tests on o-rings, one may want to consider including guidance and specifications that are relevant to these form factors. The methods described in this test report could be considered, or there may be o-ring specific test methods that could apply if mechanical testing is preferred over visual observation after aging (e.g., ASTM D1414).*
    - *Determination of acceptable visible degradation is subjective and relies on the best judgement of the test laboratory. Mechanical testing (e.g., tensile strength, ultimate elongation, hardness, etc.) to compare unaged and aged samples could provide more definitive evaluation criteria but would require more samples. This would be easier to perform on sheets of raw material to create the standard test specimen according to each referenced ASTM/ISO standard; however, relies on manufacturers to provide information on the materials and/or raw material to test.*
- It is unclear whether the same samples should be exposed to oxygen and then ozone sequentially in that order, or separate samples exposed to each.
    - *Sequential exposure is more challenging, but it is unknown if the order would affect performance results. Studying the order was out of scope for this contract.*

- This test is intended to ensure the rubber/non-metallic sealing components will not degrade when exposed to potential atmospheric gases (i.e., oxygen and ozone). However, there is not a test method to evaluate the metallic components for hydrogen embrittlement.
    - *Non-destructive evaluation (NDE) of the metallic components may be performed after the thermal/pressure cycling evaluations to check for embrittlement.*

13.3 Test Method Cost Assessment

Environmental test laboratories that routinely perform these standardized test methods are efficient in the performance. The test methods involve extended exposure to oxygen and ozone environments and do not require constant oversight. The test cost is relatively low (estimated at <$5,000) and they can be turned around quickly.

The form factor that is specified for these standardized test methods is what is expected by these laboratories, so compliance testing of non-metallic seals that are removed from production level systems may be considered custom testing. If these methods would be considered for compliance testing, some additional consideration would need to be given for the test sample preparation and selection, exposure conditions, and post-test performance criteria.

13.4 Test Method Value Assessment

Material compatibility testing is an important component of system performance. The standardized test methods (e.g., ASTM, ISO, etc.) are proven to evaluate materials, but often require specific form factors which may not be conducive to compliance testing in which production level systems are pulled from inventory/service and disassembled for evaluation. These methods may be more valuable to industry as a pre-production evaluation and/or assist in the selection and qualification of non-metallic seal materials.

14.0 HYDROSTATIC STRENGTH TESTS (GTR NO. 13, 6.2.6.2.1)

One (1) new valve unit, the contractor Sample ID V18, was subjected to the hydrostatic strength test. The valve internal portion is sealed, and the valve seat/internal blocks are fixed in the open position. A hydrostatic pressure of at least 250% NWP is applied to the inlet of the component for at least 3 min, and then the component is examined to ensure that rupture has not occurred. Then, the hydrostatic pressure is increased at a rate of less than 1.4 MPa/s until component failure. The hydrostatic pressure at failure is recorded. The failure pressure of previously tested units must be at least 80% of the failure pressure of the new valve unless the hydrostatic pressure is greater than 400% NWP.

Originally, the contractor planned to perform an engineering evaluation of this test method, but physical performance of this method was substituted for the pre-cooled hydrogen exposure test (formally GTR NO. 13, Section 6.2.6.2.10) which was removed from GTR NO. 13. Only the baseline evaluations were performed, because the test fixture used to pressurize the test valve was not designed to withstand the potentially full range of pressures of the failure pressure analysis. The 400% NWP test method would have been identical to the baseline 250% NWP, with the exception of the higher pressure. Therefore, the authors believe that the baseline 250% NWP is an adequate surrogate for the 400% NWP test, especially since the purpose of the study is to evaluate the method and not specific valve hardware.

14.1 Test Performance and Results

The valve unit tested did not meet the requirements of GTR NO. 13 Section 6.2.6.2.1, Hydrostatic Strength Tests, due to a leak that developed between the inlet outlet port (I/O port) and the valve body.

14.2 Lessons Learned

The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- There is some ambiguity in the determination of failure when a leak, rather than a catastrophic failure occurs.
    - *When a leak occurs, the flow of water into the test article must be increased in order to offset the leak. However due to the small volumes inherent to valves and because of the pressurization rate restrictions, a low flow pump would typically be used during this test. Low flow pumps will likely not be able to overcome any leaks at high pressures. This could create a situation where there is a leak in the specimen such that the test stand equipment can not continue to build pressure. One may want to consider revising the acceptance criteria, so any observable leakage is considered a failure, as this is typically*

the standard criteria in other industries where hazardous fluids are contained at high pressures.

- Only 1 new valve unit is subjected to the hydrostatic strength test, but variance may be likely between different valves, so subjecting multiple test articles to this procedure may be beneficial.
  - *The contractor ended up performing hydrostatic strength tests on 3 new valve units and observed some variance in the peak pressure achieved. One may want to consider increasing the number of new units tested to a total of 3 units.*
- The procedure requires holding pressure at 250% NWP for 3 min, then examining the component to ensure that rupture has not occurred. However, the occurrence of rupture would be obvious form the pressure response without physical examination.
  - *An examination is likely not necessary to determine if rupture occurred. If rupture occurred, the pressure would not be maintained at the target pressure condition, as evident by an abrupt pressure loss. Other industries typically require a hydrostatic test with a pre-determined time requirement (typically 1 h, 4 h, or 8 h). After the time period has elapsed, the test article is inspected for leakage or damage. Then, a separate test is performed, typically referred to as a burst test, in which pressure is increased to failure.*
- The test is performed with the valve in the open position, but confidence in the valve integrity could also be gained by testing with the valve in the closed position.
  - *One may want to consider requiring testing with the valve in the closed position in addition to the testing with the valve in the open position.*

14.3 Test Method Cost Assessment

The Hydrostatic Strength Test is a simple procedure with a much safer working fluid than hydrogen. Although using water as the test fluid is much safer than a compressible medium, safety precautions must be taken to protect the test operators since the purpose of this test is to take the test article to failure. The engineering controls put in place to minimize safety hazards can be a significant upfront cost, such as an enclosure to contain the energy if an explosive failure occurred and remotely operated controls. However, hydrostatic strength testing is a common test required in industry, so it is likely that most test facilities will already have labs commissioning to perform this testing. With a fully commissioned hydrostatic test lab, the cost to perform this testing is estimated at $10,000 to $20,000.

14.4 Test Method Value Assessment

Hydrostatic and burst testing is performed on virtually every type of pressure-containing vessel or device in all major industries. The primary function is to verify the design requirements and ensure that no material or manufacturing flaws exist before a product is brought to market. Hydrostatic testing is relatively easy and inexpensive to perform and is substantially safer than performing pressure testing with hydrogen or inert gas, due to the compressible nature of gas that increases the stored energy when pressurized.

15.0 Extreme Temperature Pressure Cycling Test (GTR NO. 13, 6.2.6.2.3)

This method provides different operational cycle testing requirements for the check valve and the shut-off valve.

For the check valve, a total of 11,000 cycles are to be performed. An operational cycle consists of applying ≥100% NWP in 6-step pulses to the check valve inlet with the outlet closed. The pressure is vented from the check valve inlet, and the pressure of the outlet is lowered to ≤60% NWP prior to the next cycle. The first 90% of cycles are performed at ambient temperature (20 °C) and ≥125% NWP, followed by an ambient leak test per GTR NO. 13 Section 6.2.6.2.2. The next 5% of cycles are performed at high temperature (85 °C) and ≥125% NWP, followed by a high temperature leak test per GTR NO. 13 Section 6.2.6.2.2. The last 5% of cycles are performed at low temperature (-40 °C) and ≥100% NWP, followed by a low temperature leak test per GTR NO. 13 Section 6.2.6.2.2. After the 11,000 cycles are complete, the check valve is subjected to a chatter flow test, leak tests per GTR NO. 13, Section 6.2.6.2.2, and finally the hydrostatic strength test per GTR NO. 13, Section 6.2.6.2.1. This test series is summarized in Table A-4.

Table A-4. Extreme Temperature Pressure Cycling Series – Check Valve.

Cycling Phase and Temperature Setting	Cycle Nos.	Pressure High And Low Settings	Post-Phase Checkout
Ambient Temp Cycling 20 °C	1 to 9,900	≥ 125% NWP ≤ 2 MPa inlet / 60% NWP outlet	Ambient Leak Test (20 °C) per GTR NO. 13, Section 6.2.6.2.2
High Temp Cycling 85 °C	9,901 to 10,450	≥ 125% NWP ≤ 2 MPa inlet / 60% NWP outlet	High Temp Leak Test (85 °C) per GTR NO. 13, Section 6.2.6.2.2
Low Temp Cycling -40 °C	10,451 to 11,000	≥ 100% NWP ≤ 2 MPa inlet / 60% NWP outlet	Low Temp Leak Test (-40 °C) per GTR NO. 13, Section 6.2.6.2.2
Post-Test Checkouts			
1. Chatter Flow Test – 24 h chatter flow at a flow rate that causes the most chatter (valve flutter) 2. Leak Tests per GTR NO. 13, Section 6.2.6.2.2 3. Hydrostatic Strength Test per GTR NO. 13, Section 6.2.6.2.1			

For the shut-off valve, a total of 50,000 cycles are to be performed. An operational cycle consists of applying/relieving pressure to both the inlet and outlet sides. The first 90% of cycles are performed at ambient temperature (20 °C) and ≥125% NWP, followed by an ambient leak test per GTR NO. 13, Section 6.2.6.2.2. The next 5% of cycles are performed at high temperature (85 °C) and ≥125% NWP, followed by a high temperature leak test per GTR NO. 13, Section 6.2.6.2.2. The last 5% of cycles are performed at low temperature (-40 °C) and ≥100% NWP, followed by a low temperature leak test per GTR NO. 13, Section 6.2.6.2.2. No additional post-test checkouts are specified for the shut-off valve. This test series is summarized in Table A-5.

Table A-5. Extreme Temperature Pressure Cycling Series – Shut-off Valve.

Cycling Phase and Temperature Setting	Cycle Nos.	Pressure High And Low Settings	Post-Phase Checkout
Ambient Temp Cycling 20 °C	1 to 45,000	≥ 125% NWP ≤ 2 MPa	Ambient Leak Test (20 °C) per GTR NO. 13, Section 6.2.6.2.2
High Temp Cycling 85 °C	45,001 to 47,500	≥ 125% NWP ≤ 2 MPa	High Temp Leak Test (85 °C) per GTR NO. 13, Section 6.2.6.2.2
Low Temp Cycling -40 °C	47,501 to 50,000	≥ 100% NWP ≤ 2 MPa	Low Temp Leak Test (-40 °C) per GTR NO. 13, Section 6.2.6.2.2

15.1 Engineering Evaluation and Lessons Learned

Although the physical testing required in Section 6.2.6.2.3 was not conducted at the contractor, this section's language has been reviewed for the purpose of providing an engineering evaluation on the test requirements. This evaluation was conducted in large part based on the findings from conducting the Section 6.2.6.1.1 TPRD pressure cycling test, as well as the lessons learned as summarized below.

- The title of Section 6.2.6.2.3 could be misinterpreted to mean pressure cycling as opposed to operational cycling. In regard to the check valve, the operational cycling procedure is very similar to the pressure cycling test procedure since the check valve cannot be actuated. However, the operational cycling procedure for the shut-off valve is different from the pressure cycle test procedure since the valve is being actuated between the open and closed positions. Titling Section 6.2.6.2.3 as "Extreme Temperature Operational Cycling Test" may eliminate some confusion.
- Section 6.2.6.2.3 states that hydrogen or a non-reactive gas can be used. If it is acceptable to use substitute gases for Section 6.2.6.2.3, then substitute gases would likely be acceptable for the Section 6.2.6.1.1 TPRD pressure cycling test as well. Requiring the same gases would ensure uniformity in the test methods and improve the likelihood of being able to combine this testing with the TPRD pressure cycle test.
- Requiring helium as the substitute gas would increase confidence in the valve testing due to helium's small molecular size. Additionally, it would significantly reduce the safety risk associated with conducting this test.

- Section 6.2.6.2.3 requires a flow test on the shut-off valve and check valve at chatter flow rate for at least 24 hours following the pressure cycles. The contractor agrees with the note that states chatter flow testing is only required on the shut-off valve if it is functioning as a check valve because chatter flow only applies to check valves and relief valves. GTR NO. 13 does not currently provide guidance on how to determine the chatter flow rate, but a potential method would be to slowly lower the flow through the valve until the noise of the closure member switching from the open to closed position is audible. The noise could be measured to determine the flow rate corresponding to the highest noise level, establishing this as the chatter flow rate. the contractor is unaware of a published standardized test procedure that currently exists for valve flow testing at the chatter flow rate. Prior to designing the test stand, it would help test operators if GTR NO. 13 included a range for the estimated chatter flow rate through the check valve and shut-off valve or recommends that an estimated range is provided by the valve manufacturer to the test lab based on the minimum design velocity for proper check valve operation. One may choose to incorporate pass or fail criteria for the chatter flow rate, if deemed necessary. For example, a requirement could be added that establishes a maximum acceptable chatter flow rate. Alternatively, each test article could be required to have a chatter flowrate within 5% of the average chatter flow rate for all tested units, to observe for consistency.
- The 24-h flow duration for the chatter test could be accommodated fairly easily using advanced software controls to allow autonomous test facility operation or by manning the system overnight. The contractor agrees with the optional tolerance in Table 10 that allows an additional hour beyond the 24-h minimum.
- Similar to Section 6.2.6.1.1, the contractor agrees that the tolerance allowed on the target temperatures is appropriate at +5°C for the high-temperature target and -5°C for the low-temperature target. Although Table 10 is currently labeled as optional tolerances for test parameters, test operators might ask the manufacturer of the check valve and shut-off valve for a maximum allowable temperature to avoid damage to the test article.
- Similar to Section 6.2.6.1.1, the contractor agrees that the tolerance allowed on the target test pressure is appropriate at +5% of the nominal working pressure of the valve. Although Table 10 is currently labeled as optional tolerances for test parameters, determination of the absolute pressure limit is critical to avoid damaging the test article or causing a safety incident.
- A summary table of the cycling conditions, similar to that provided in Section 6.2.6.1, could improve clarity.
- Conducting 50,000 pressure cycles on the shut-off valve, as is currently required in Section 6.2.6.2.3, would require a considerable amount of time and put a toll on the test facility components. Likely, there would be significant delays throughout the testing process to replace

valve or pump seals and perform other preventative maintenance activities. The average cycle time when pressurizing to 125% of the working pressure was about four minutes per cycle. It is estimated that using our existing test stand to cycle to 100% of the working pressure would require approximately 3.5 minutes per cycle. Assuming the cycling time is cut in half since the procedure only requires venting down to 50% of the test pressure, an estimated 61 days of continuous 24-h operation would be required to complete this testing.

- Since the purpose of the Section 6.2.6.2.3 shut-off valve testing is to ensure proper functioning of the shut-off valve after a significant number of operational cycles, one method to expedite the testing process would be to lock in pressure downstream of the shut-off valve and not require venting of the inlet pressure to 50% of the test pressure in between operational cycles. Therefore, the valve will be cycled against zero differential pressure. This would make the cycles purely operational and not pressure cycles as currently stated. The same method is used when completing mechanical cycles on rising stem valves to complete fugitive emissions qualification testing according to American Petroleum Institute (API) Standard 624. Without waiting to increase and vent pressure, the test duration would solely be dependent on the cycling speed of the valve which is likely much faster. For example, an actuation rate of 1 Hz would require less than 14 h to complete all 50,000 operational cycles. Additionally, this would allow the operator to more easily conduct this testing simultaneously with the Section 6.2.6.2.3 check valve testing and Section 6.2.6.1.1 TPRD testing by simply completing several operational cycles of the shut-off valve in between each pressure cycle.

15.2 Test Method Cost Assessment

The contractor conducted an evaluation of the test language and did not conduct physical testing. Therefore, the contractor is unable to evaluate the physical test costs.

15.3 Test Method Value Assessment

Material compatibility testing is an important component of system performance. The standardized test methods (e.g., ASTM, ISO, etc.) are proven to evaluate materials, but often require specific form factors which may not be conducive to compliance testing in which production level systems are pulled from inventory/service and disassembled for evaluation.

16.0 ELECTRICAL TESTS (GTR NO. 13, 6.2.6.2.7)

One (1) shut-off valve, the contractor Sample ID V16, was subjected to the electrical test. The test procedure is not applicable to check valves. The electrical test consists of two parts, the abnormal voltage test and the insulation resistance test. For the abnormal voltage test, the solenoid valve is connected to a variable DC voltage source and operated as follows:

a. Steady state temperature is established for 1 h at 1.5x the rated voltage.
b. Then the voltage is increased to 2x the rated voltage or 60 Vdc, whichever is less, and held for 1 min.
c. Any failure shall not result in external leakage, open valve or unsafe conditions (e.g., smoke, fire, or melting).

For the insulation resistance test, 1,000 Vdc is applied between the power conductor and the component casing for at least 2 s. The minimum allowable resistance for that component is 240 kΩ.

16.1 Test Performance and Results

The abnormal voltage test was performed on April 21, 2022. Initially, 20.7 Vdc was applied at 10 kHz and 50% duty cycle (1.5 times the rated voltage), until the steady state temperature of 126.5 °C was achieved after approximately 1 h 6 min. Then, the voltage was increased to 27.6 Vdc (same frequency/duty cycle) for at least 1 min. No failure or unsafe conditions were observed, and the valve successfully closed once power was shut-off.

The insulation resistance test was performed on April 22, 2022, after the solenoid coil was allowed to cool down. First the insulation resistance test was performed between the solenoid coil power conductor and the set nut. Next the insulation resistance test was performed between the solenoid coil power conductor and the valve I/O port. The resistance measured at both locations was greater than 11 Giga-ohms, which meets the requirement of greater than 240 kiloohms-ohms.

The valve tested met the requirements of GTR NO. 13 Section 6.2.6.2.7, Electrical Tests. The following subsections discuss the lessons learned, GTR No. 13 procedure evaluations, and a qualitative value assessment for this test method.

16.2 Lessons Learned

The following list summarizes the contractor's observations, questions, comments, and findings for the Electrical Tests. The lessons learned are bulleted with additional details or supporting statements written in italics below each.

- The test order is not specified, nor is it specified if the tests both have to be performed on the same unit. The unit might be more likely to pass the insulation resistance test if performed first. Heating during the abnormal voltage test could degrade the insulation.

- *The contractor performed the abnormal voltage test followed by the insulation resistance test (same order as currently listed in GTR NO. 13) on the same unit in order to represent the worst-case scenario.*

- Use of the manufacturer wire harness is not mentioned in the test method.
  - *The contractor used the manufacturer wire harness. One may consider a note to use the harness, if available, since it is the intended setup and would be considered part of the electrical system under evaluation. However, if it is not available, connecting directly to the pins could also be acceptable as long as caution is taken to avoid shorting within the connector.*

- The voltage specifications in the test method do not provide guidance when manufacturers specify peak and hold Pulse Width Modulation (PWM) requirements. It is unclear from the test method if the intent was to double voltage, double current, or both.
  - The contractor contacted *Graham Meadows for guidance on the intent of the voltage specifications in this test method. Mr. Meadows has twenty years of work experience testing and validating H_2 vehicle fuel systems and components. He is heavily involved in the development of H_2 codes and standards (e.g., CSA's HGV 3.1 and HPRD 1 and ISO equivalent documents). Additionally, Mr. Meadows was specifically involved on the Phase 2 development of GTR NO. 13, serving on Task Force 3 and contributing towards the component test procedures. He provided the following guidance:*

    "Need to consider valve manufacturer's peak and hold or PWM requirements. I have talked about this with a Technical Service for Europe previously and he was in agreement. For example, if PWM requirement would be 12V with a 50% PWM duty cycle then the 1.5x should be 18V with 50% duty cycle. Similarly, for a peak and hold setup then I think you can increase voltage to 1.5x and 2x but limit the current to the manufacturer's requirements. Peak and hold or PWM values are not intended for 1.5x or 2x current and the manufacturer's information should include information about required fusing which would prevent an over-current situation."

  From the contractor testing experience, it was possible to apply 1.5x and 2x voltage with PWM. The manufacturer specified frequency but not duty cycle, so the contractor assumed 50% PWM duty cycle. The power supply current should be set to the peak allowable, because when setting the voltage above the rated voltage the current will also increase.

- The equilibrium hold is unclear as written, "An equilibrium (steady state temperature) is established for at least one hour at ≥ 1.5 times the rated voltage." Is steady state temperature established and then maintained for at least 1 h? Or is 1.5x voltage applied until steady state temperature is met, which should be at least 1 h? The temperature measurement location is not specified.
  - *One may want to consider rewording the test method to remove ambiguity. A temperature sensor may be placed on the solenoid coil surface to monitor temperature rise as the valve heats up when energized. From the contractor testing experience, it took just over an hour for the solenoid coil surface temperature to reach steady state. If one may choose to update the language as follows:*

    "Apply ≥ 1.5 times the rated voltage until an equilibrium (steady state temperature of solenoid coil surface) is established or for at least one hour, whichever is longer."
- The upper bound for the voltages applied and the durations they are to be applied are not specified (tolerances).
  - *After evaluation, NHTSA shared a newer draft that included a table with optional tolerances for the test methods. The contractor agrees the suggested +0.5 V is sufficient.*
- Method of verification for external leakage/open valve failure criteria is not specified.
  - *The contractor suggests that after the test is complete, the valve could be pressurized from the tank side to TBD% NWP to confirm that gas isn't flowing through the I/O port, which would confirm that the valve is not open and there is no external leakage. Alternatively, this test unit could undergo the Leak Test per GTR NO. 13, Section 6.2.6.2.2.*
- Use of the manufacturer wire harness is not mentioned in the test method.
  - *The contractor used the manufacturer wire harness. One may consider a note to use the manufacturer's wire harness, if available, since it is the intended setup and would be considered part of the electrical system under evaluation. However, if it is not available, connecting directly to the pins could also be acceptable as long as caution is taken to avoid shorting within the connector.*
- Position of the lead attachment to the component casing is vague, but understandably so since it is valve design dependent. The solenoid valve that was tested had a plastic coil cover, which would not provide a good electrical contact.

- *One may consider location examples and/or guidance be provided, for example:*

  "Insulation resistance test. 1,000 V D.C. is applied between the power conductor and the component casing (i.e., solenoid coil casing, if metal, otherwise the set nut or valve I/O port are acceptable connection points) for at least two seconds."

16.3 Test Method Cost Assessment

The Electrical Test consists of two parts, the abnormal voltage test and the insulation resistance test. Both tests involve standard laboratory equipment, such as a DC power supply and an insulation resistance tester. Assuming the laboratory has these pieces of equipment and the ability to measure and log temperature, these tests should be fairly quick to setup and perform. The estimated test cost would be <$5,000 per test unit.

16.4 Test Method Value Assessment

The Electrical Test is simple to setup and perform and provides valuable performance demonstration of the solenoid valve for system integrity. The suggestions in the Lessons Learned section should further improve this method by removing ambiguities and standardizing the evaluation.