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Hydrogen Field Test Standard Design, Operating Instructions, & Specifications

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Hydrogen Field Test Standard Design, Operating Instructions, & Specifications

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August 26, 2015

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Abstract

The National Institute of Standards and Technology (NIST) developed a prototype field test standard (FTS) that incorporates three test methods that could be used by state weights and measures inspectors to periodically test the accuracy of fuel delivery at retail hydrogen gas vehicle refueling dispensers, much as gasoline dispensers are tested today. The three field test methods are: 1) gravimetric, 2) Pressure, Volume, Temperature (*PVT*), and 3) master meter. The FTS was tested in NIST's Transient Flow Facility with helium gas and in the field at a hydrogen dispenser location. This document describes the design and construction of the test standard, necessary safety instrumentation for working with *high* pressure hydrogen gas, how to execute tests with the FTS, and the operating specifications of the FTS components. This document is intended to aid others attempting to design and construct similar apparatuses.

Key Words: Hydrogen, Gaseous Refueling, Test Standard

1. Introduction to the Standard

1.1 The H₂ field test standard

The H₂ field test standard (FTS) consists of a 35 MPa (15 °C), 1 kg H₂ capacity storage tank equipped with pressure and temperature sensors that is mounted into a frame made from 2.5 cm² T-slotted aluminum with wheels for mobility. The storage tank is a type 3 cylinder, which is a seamless aluminum liner fully wrapped with a continuous filament made of carbon fiber in an epoxy reinforcement laminate [1]. The empty weight of the FTS is approximately 80 kg. Figure 1 shows the H₂ FTS in the horizontal position; Fig. 2 is a plumbing and instrumentation diagram (P&ID) of the FTS. For density measurements, the FTS is equipped with two, 46 cm long type K thermocouples (TCs) inserted at each end (to reduce stem conduction errors, TCs with a long insertion depth were chosen) and two analog pressure sensors; one with a 35 MPa (5000 psi) range and one with a 1.4 MPa (200 psi) range.

Accompanying the FTS are 1) a 150 kg capacity weigh scale with 1 g resolution for gravimetric measurements, 2) a 3.7 m x 3.7 m tent with ventilated holes to protect the weigh scale from the environment, 3) a portable data acquisition (DAQ) box and laptop with acquisition software, 4) a 0.95 cm diameter, 3.05 m tall stainless steel vent stack with support stand for venting H_2 gas in the field, and 5) a hand truck with a tie down strap for moving the FTS securely while in the vertical position; the FTS must be in the vertical position during testing due to the position of pressure relief valves. The DAQ box supplies power to the master meter and acquires data from the temperature and pressure sensors. All wires can be easily plugged into or unplugged from the DAQ box and coiled on the FTS frame during weighing. Figure 3 shows the H_2 FTS positioned on the scale with the accompanying DAQ components in the laboratory. All electrical components on and accompanying the H_2 FTS are intrinsically safe.

Elastomer materials are subject to H_2 embrittlement just as metals are [2,3,4], therefore all valves have H_2 compatible seating material. All plumbing on the FTS and the diaphragm in the pressure sensors is 316-stainless steel. The TC sheath material is Inconel.

1.2 Valves

The valves on the FTS are shown in Fig. 2. There are two check valves and one ball valve (Valve #1) at the FTS tank inlet. One of the check valves is incorporated into the H_2 dispenser receptacle. The ball valve can be used to control the inlet flow if necessary. There is one check and one ball valve (Valve #2) at the inlet for purge gas. The FTS tank must be purged of all H_2 gas before vehicular transport. The tank outlet has one ball valve (Valve #3) upstream from a non-venting regulator that is used to control the flow through the master meter when blowing the tank down. Valve #4, a ball valve, protects the low ranged pressure sensor when the tank is pressurized above 1.4 MPa.

The handles for the four ball values are on the front plate of the H_2 FTS (Fig. 4). Each value opens away from its labeling; *i.e.* when the FTS is in the vertical position, the value is open when the handle is perpendicular to the floor and it is closed when the handle is parallel to the floor.



Figure 1. H₂ FTS in horizontal position.



Figure 2. Plumbing and instrumentation diagram of the H₂ FTS.



Figure 3. H₂ FTS in the vertical position on the scale with DAQ components.





Valve #1 Open position





Valve #3 & #4 Closed positions Valve #3 & #4 Open positions



Valve #2 Closed position; rotate upward to open Figure 4. Valve numbering and open/closed positions.

1.3 Over pressurization features

The H₂ FTS tank was hydrostatically tested by NIST up to 45 MPa (6500 psi) in June of 2013. The FTS is equipped with a pressure relief valve that will open if the pressure reaches 37.9 MPa (5500 psi) \pm 5 %. The seating and seal material in this pressure relief valve is ethylene propylene rubber (EPR). It has a temperature

range of -57 °C to 135 °C. The pressure relief valve was tested three times at NIST for proper functionality before being installed in the FTS. The valve re-sealed when the pressure decreased to approximately 88 % of the pressure at which it opened. Therefore, <u>if this valve opens during field tests</u>, the tank pressure should be relieved to the starting pressure and the test draft re-started.

A pressure relief valve is also installed upstream of the low range pressure sensor but downstream of Valve #4 that isolates it from the FTS tank. This relief valve will protect the sensor in the event the isolation valve leaks when the FTS tank is under "high" pressure and protect persons and property by preventing the sensor from mechanically failing resulting in uncontrolled H_2 gas release. The seating and seal materials are fluorocarbon FKM, which has a temperature range of -4 °C to 65 °C. The pressure relief valve was tested six times at NIST for proper functionality before being installed in the FTS. The valve opens at pressures ranging from 1.73 MPa (251 psi) to 1.85 MPa (268 psi). The valve re-sealed when the pressure decreased to approximately 60 % of the pressure at which it opened. If this valve opens during field tests, it is indicative that Valve #4 has failed. See Section 4.7 for how to instructions for this situation.

1.4 The DAQ box

Figure 5 shows the portable DAQ box. The box contains a 24 VDC/3 A power supply and a National Instrument (NI) DAQ chassis. Both the power supply and the DAQ chassis have power cords at the back of the DAQ box that require 115 VAC power. A 14 AWG earth ground wire for grounding the electronics and an Ethernet cable for computer connection also protrudes from the back of the DAQ box. **The DAQ box should never be opened while powered up.**

The power supply powers the pressure sensors, master meter, H_2 gas sensor alarms, and the infrared/ultraviolet (IR/UV) flame detector (Section 1.5). The NI DAQ chassis holds 4 DAQ modules. Two modules are installed: 1) a 32 channel analogue input (AI) card and 2) a 4 channel TC card. The AI card reads the pressure sensors and the master meter analogue output. It is not necessary to read the master meter analogue output, it is for diagnostic purposes if needed. The TC card reads the 4 TCs; the two installed and the two spare. When a TC channel is not in use, plastic inserts protect the plug from collecting debris (Figure 5).

The front of the DAQ box has plugs for each TC, TC0 through TC3. The TCs are labeled appropriately. <u>Each</u> <u>TC must be plugged into its respective plug</u> because the calibration coefficients in the DAQ program are specific to these channels. The front of the box also has a plug for the higher ranged (34.5 MPa) pressure sensor and for the lower ranged pressure sensor. <u>The pressure sensors must be plugged into their respective plug</u> for the proper calibration coefficient to be applied to the correct sensor.

1.5 Safety instrumentation

The H_2 FTS has three safety technologies that travel with it: 1) Four H_2 sensors with two alarms (two sensors per one alarm), 2) an IR/UV flame detector with alarm, and 3) a handheld combustible gas detector.

The H₂ alarms are attached to the side of the DAQ box (Figure 6). The top of each alarm has two plugs for connection to the H₂ sensors. There is 7.6 m (25 ft) connector cable for each sensor. The sensor, cable and plug on the alarm are labeled so the calibrated sensor can be plugged into the port it was calibrated in. The H₂ sensors are a safety feature that sounds an audible alarm in the event of a H₂ leak leading to a combustible limit (2 %) within the weighing area during testing. An orange light emitting diode (LED) on the sensors flashes when the sensors are plugged into an alarm properly. When < 1 % H₂ gas is present, a green LED is illuminated continuously on the alarm, indicating the sensors are functioning properly. When the H₂ concentration reaches 1 %, a yellow LED on the alarm comes on to warn prior to reaching the 2 % limit. These sensors should be tested for functionality and calibrated if needed prior to field use. Refer to user manual for more detailed information on how to calibrate and validate the sensors [5].

The IR/UV flame detector is a safety feature that detects hydrocarbon flames from a distance of 25 m (82 ft) and was designed for outdoor use. Because H_2 gas is flammable at a concentration of 4 % in air, it is necessary to monitor the FTS during testing to assure no un-detected leak gives rise to a fire. The flame detector is powered by the DAQ box. There is approximately 30 m (100 ft) of cable for supplying power to the flame detector. To power the flame detector, the banana plug can be "piggy-backed" into the "meter power" plug on the front of the DAQ box (Fig. 7). A blue light on the detector face illuminates when it is powered. Both the UV and IR LED indicators blink alternating three times approximately every 25 seconds to 40 seconds when the device is working properly and no fire is present. The flame detector should be positioned within 25 m (82 ft) of and aimed at the FTS. Both of the two LEDs on the front panel will light up and stay lit continuously and an 80 dB (at 1 meter distance) audible alarm will sound if fire is present. Refer to user manual for more detailed information [6]. The presence of fire at the FTS outlet during blow-down (*i.e.* gas release) is not a hazard because the gas will be released at a location designed for such, *i.e.*, away from people and property.



Figure 5. The DAQ box.

The handheld combustible gas detector is used to check all valve stems and joints during the initial introduction of H_2 gas. The detector will detect small leaks that could be a safety hazard if unnoticed. The detector can detect 5 x 10⁻⁴ % (500 parts per million) H_2 gas and is for use in Class I, Division I hazardous locations.



Cable labels Plug labels Figure 6. H₂ sensors and alarms.



Figure 7. IR/UV flame detector plugged into DAQ box. Alarm is located at rear of face plate.

2. On-site setup

2.1 The test area

The test area consists of: 1) the DAQ area, 2) the filling area, 3) the weighing area, and 4) the gas blow-down area (Fig. 8). Prior to arriving at a test site, the site layout will be known and a rough footprint will be generated so the placement of equipment, site hazards, and safe "escape" routes will be known. During testing, this area is isolated via pennants that meet OSHA regulations for marking above ground construction areas. All

electronics requiring 115 V power will plug into an uninterruptable power supply with 45 minutes of battery backup time.

The DAQ area (DAQ box and laptop) must be setup a minimum distance of 1.5 m (5 ft) from the H₂ FTS during measurements to assure the electrical components will not ignite a fire if there is a H₂ gas leak [7]. <u>Warning tape should be placed around the DAQ area during tests so tripping hazard is minimized due to wires being strung from the FTS to the DAQ area</u>. The pressure sensor wires are the shortest of the sensors and therefore, limit the distance the DAQ box can be from the H₂ FTS to 3.7 m (12 ft). The IR/UV flame detector (Section 1.5) should be positioned no more than 25 m (82 ft) from the H₂ FTS during filling and blow-down to assure no fire is present due to an un-detected leak from the FTS. <u>The grounding cables: 1) extruding from the DAQ box and 2) attached to the IR/UV flame detector</u>, must be grounded at a suitable location at the dispenser site.

The filling area is the area where the dispenser is located. The H_2 FTS can be filled from the dispenser just as a vehicle would be. The dispensers tested with the FTS must have a standard 35 MPa hydrogen gas nozzle. The FTS inlet has the standard receptacle for the nozzle and it CANNOT accept a 70 MPa nozzle. Only sites with standard equipment can be tested. The FTS must be grounded using the grounding cable with clamps that accompany it during filling. The FTS collection tank internal temperature MUST be continuously monitored during filling to prevent overheating of the collection tank. This is particularly important if the dispenser does not pre-cool the H_2 gas.

The weighing area is typically located in a 3.7 m x 3.7 m (12 ft x 12 ft) canvas tent with ventilated holes in the top that travels with the FTS unless an appropriate structure is already at the test site. The electrical connections that need to be made in the weighing area are: 1) the scale weighing pan, 2) its display, and 3) the H₂ sensors (Fig. 8 and Fig. 9). The H₂ sensors should be placed inside the weighing area, preferably at *high* locations and around the FTS. The scale display should be kept outside of the weighing area for fire safety reasons. However, if this is not possible due to the length of the cable connecting the scale weigh pan to the scale display, the display can be brought into the weighing area as long as the digital communications port is plugged (see Section 2.3 for further information). The scale has a grounding lug that must be used to ground the scale during testing. An environmental monitor is used to make buoyancy corrections to scale measurements. The environmental monitor must be kept outside of the weighing area because it is not intrinsically safe.

The gas blow-down area can be any distance greater than 3 m (10 ft) from the DAQ area. A 3 m (10 ft) high 3/8" diameter vent stack that accompanies the FTS is used unless the test site already has such a vent stack in place. This will assure the gas escapes away from people or property. The plumbing that connects the FTS to the vent stack is 0.64 cm (0.25 in) stainless steel and can be plumbed on-site. The FTS must be grounded using the grounding cable with clamps that accompany it during the blow-down. It is not necessary to take real time temperature and pressure measurements in the FTS tank during blow-down.

2.2 H₂ FTS DAQ box and laptop

Figure 10 shows the laptop with locations for DAQ component connections. The USB to RS-232 converter plugs into the USB port labeled "COM 4, 5". The USB to RS-232 converter has two plugs, one for the environmental monitor and one for the scale digital output. The plugs and the cables are labeled accordingly. The DAQ box Ethernet cable plugs into the Ethernet connection at the rear of the laptop. Power (115 VAC) must be supplied to the DAQ box and the laptop. <u>The DAQ box must be grounded during testing.</u>



Figure 9. Scale setup. The port for digital communications must be plugged if the scale display is inside the weighing enclosure.

2.3 The scale

The scale has digital communications with the laptop. However, this makes the scale display not intrinsically safe. Therefore, if the scale display (Fig. 9) cannot be located outside of the weighing enclosure, the cable can be removed and the port plugged so it is intrinsically safe again. Refer to the scale user manual for more

detailed information [8]. The scale readout is stable if there are no air currents or vibrations around the scale and hence, averaging the scale measurements is not necessary, the readout can be read directly from the display and the value manually entered into the data reduction spreadsheet (see Section 3.5). <u>The scale must be grounded during testing.</u>



Figure 10. Laptop with locations for DAQ component connections.

3. Acquiring data

3.1 The laptop

The laptop that runs the DAQ software is NIST owned. The laptop cannot be connected to the internet due to security reasons. The laptop has a functional account that allows multiple authorized users to use the same user name and password for MacAfee software and Windows. The username and password will only be given to trained personnel authorized to operate the FTS.

3.2 The software

The software that runs the DAQ program is LabView 10. To open the program, open LabView and open the program "Hydrogen Field Test". Before starting:

- 1. On the main page of the LabView program, make sure the toggle switch under the heading "cal on/off?" is put to the *on* position (Fig. 11). This applies the calibration coefficients to the sensor readings.
- 2. Put the toggle switch under the heading "Read Scale" to the *yes* position if the scale digital output is used, put it to the *no* position if the scale measurements are manually read from the scale display (Fig. 11).

The calibration coefficients are fixed for all sensors and should not be changed. The array containing all of the calibration coefficients are labeled for each sensor (Fig. 11). The first column is the " a_0 " coefficient; the second column is the " a_1 " coefficient and so on. If the coefficients are accidently changed, they will return to the starting values if the program is closed and reopened again.



Use calibration coefficients function

Figure 11. LabView DAQ program interface. Toggle buttons are used to turn on or off the functions: averaging, read scale, and apply calibration coefficients to sensor readings.

3.3 Running the Software

Before running the LabView program, perform the following so the DAQ chassis will communicate with the laptop:

- 1. Open the program "NI MAX", a shortcut is on the desktop (Fig. 12).
- 2. In the left hand column, expand "Devices and Interfaces", "Network Devices", "NI cDAQ-9184".
- 3. Right click on "NI cDAQ-9184".
- 4. In the menu click "reset chassis".

This will reset the DAQ chassis. Close "NI MAX" and open the DAQ program "Hydrogen Field Test" and click the start arrow (Fig. 11). Data will be continuously acquired from all sensors that are plugged into the DAQ box at a rate of 1 Hz until the "Stop" button is pressed (Fig. 11). This data is automatically saved to a text file located on the computer's hard drive. The data is saved with file name "file number_1" if it is the only file in the folder; if there are other files preceding the current file, the file will be given the name "file number_x"; where x is the next sequential number.

This data is useful for evaluating the FTS tank temperature and pressure profiles during filling and blow-down of the gas through the master meter. However, for all necessary calculations, average readings from each instrument are most useful.



Figure 12. NI MAX used to reset DAQ Chassis if errors appear.

3.4 Taking average data

To start averaging sensor readings, move the toggle switch shown in Fig. 11 that is labeled "averaging" to the *on* position. The switch can be switched to the *on* position while the DAQ program is running and will average the sensor readings until the button is put back to the *off* position. The switch can be switched to the *on* position and back to the *off* position numerous times while acquiring data; this is useful when acquiring scale measurements because the zero readings and the FTS readings will be in the same file that can be easily pasted into the data reduction workbook (Section 3.5).

The average data is saved to a text file that is located on the computer's hard drive. The data is saved with file name "Average Data" if it is the only file in the folder; if there are other files preceding the current file, the file will be given the name "Average Data_x"; where x is the next sequential number.

3.5 Inserting data into the reduction workbook

The reduction workbook is located on the computer's hard drive. The workbook has a separate sheet for entry of data from each test draft and a single output sheet for all the test drafts. There are nine worksheets for data input; therefore, nine test drafts can be performed without having to open a new workbook. The calculations performed in the workbook are explained in detail in Section 7.

Figure 13 shows the Input Worksheet for the first test draft. Each Input Worksheet (1 - 9) looks identical. There are 4 "questions" that need to be answered in each Input Worksheet used: 1) was the scale manually read,

2) how many times was the FTS weighed before filling, 3) how many times was the FTS weighed after filling, and 4) which TCs were used. Type "y" for yes and "n" for no to tell the software if the scale was manually read and which TCs were used. The number of times the FTS can be weighed ranges from 1 to 3; any number outside of this range will return an error.



Figure 13. Data Input Worksheet from the data reduction workbook.

The "average data" files from (Section 3.4) that are collected during test drafts (Section 4) and the manual readings from the master meter transmitter are used in the reduction workbook. Only cells that are meant for data entry can be manipulated in the Input Worksheets. To insert data into an Input Worksheet:

- 1. Copy only the data from the text file generated by LabView.
- 2. Paste the data under the appropriate header.
 - a. *I.e.*, If you are weighing the FTS before taking a test draft, insert the data in the green field shown in Figure 13 next to the label "Mass pre-fill".
 - b. Only pertinent data will show up in black, however, placeholders are present for data that is not relevant to the calculation being performed.
- 3. Input the master meter transmitter readings before and after blowing the gas down.
- 4. On subsequent Input Worksheets, the data that is inserted next to the heading "*PVT* pre-fill" is automatically filled in from the previous sheets "post blow-down" data input.
 - a. <u>Therefore, it is important to use the Input Worksheets in their numbered order</u>.

The Results Worksheet (Fig. 14) has a column for entering the dispenser readings (in kilogram) for each test draft performed. <u>This is the only data that needs to be entered in this worksheet</u>. The data entered in the Input Worksheets is used to calculate the mass dispensed for each test draft, as determined by each of the three methods, and displayed in the Results Worksheet for comparison with the dispenser. The agreement is displayed in the red column in the Results Worksheet.

gas species Hydrogen	V H2 FTA	(A)	expansion (α)												
	39529.92525	2.43E-10	3.00E-06												
		Prefill Tank Conditio	ins	p	ost-fill Tank Condi	itoins	Post-E	3low-Down Tank (Conditons		Tank Volume			Tank Gas Densit	у
Test Dan B	Tang	D bish	D low	Taur	Dhiet	D low	Т	Phiet	D low	Dre 611	Beet 60	Post-Blow-	Dro 611	Dest 61	Post-Blow-
[#]	[K]	[kPa]	[kPa]	[K]	[kPa]	[kPa]	[K]	[kPa]	[kPa]	[cm3]	[cm3]	[cm3]	[kg/cm3]	[kg/cm3]	[kg/cm3]
1	273.15	0	0	273.15	0	0	273.15	0	0	39521.1	39521.1	39521.1	0	0.0	0.0
2	273.15	0	0	273.15	0	0	273.15	0	0	39521.1	39521.1	39521.1	0	0.0	0.0
3	273.15	0	0	273.15	0	0	273.15	U	0	39521.1	39521.1	39521.1	U	0.0	0.0
*	273.15	0	0	273.15	0	0	273.15	0	0	39521.1	39521.1	39521.1	0	0.0	0.0
6	273.15	0	ñ	273.15	ñ	ñ	273.15	ñ	ñ	39521.1	39521.1	39521.1	ñ	0.0	0.0
7	273.15	0	0	273.15	0	0	273.15	0	0	39521.1	39521.1	39521.1	0	0.0	0.0
8	273.15	0	0	273.15	0	0	273.15	0	0	39521.1	39521.1	39521.1	0	0.0	0.0
9	273.15	0	0	273.15	0	0	273.15	0	0	39521.1	39521.1	39521.1	0	0.0	0.0
									Scale mas	s"pre-fill"	Scale mass	s "post fill"			
		Pre-fill Atmosphe	aric Conditions			Post-fill Atmosphe	ric Conditions	\$	Apparent	TRUE	Apparent	TRUE	Gravimetric	PVT	Master Meter
Test Draft	Patm	T atm	RH	ρ air	Patm	T atm	RH	p air	Scale	mass	Scale	mass	Mass to Fill	Mass to Fill	Mass to Fill
[#]	[Pa]	[K]	[%]	[kg/cm3]	[Pa]	[K]	[%]	[kg/cm3]	[kg]	kg	[kg]	[kg]	[kg	[kg]	[kg]
1	#DIV/01	#DIV/01	#DIV/01	#DIV/01	#DIV/01	#DIV/01	#DIV/01	#DIV/01	0.000000	#DIV/01	0.000000	#DIV/01	#DIV/01	0.000000	0.000000
2	#DIV/01	#DIV/01	#DIV/01	#D17/01	#DIV/01	#DIV/01	#DIV/01	#D1V/01	0.000000	#D17/01	0.000000	#DIV/01	#D17/01	0.000000	0.000000
4	#DIV/01	#DI37/01	#D17/01	#D17/01	#DIV/01	#DIV/01	MC/14/01	wL/14/01	0.000000	DI3//01	0.000000	#DI37/01	#D137/01	0.000000	0.000000
5	#DIV/01	#DIV/01	#DIV/01	#DIV/01	#DIV/01	#DIV/01		-		DIV/01	0.000000	#DIV/01	#DIV/01	0.000000	0.000000
6	#DIV/01	#DIV/0!	#DIV/01	#DIV/01	#DIV/01	#DIV/01		Inn	nt	DIV/01	0.000000	#DIV/01	#DIV/01	0.000000	0.000000
7	#DIV/81	#DIV/01	#DIV/01	#DIV/01	#DIV/01	#DIV/01		- mp	ui	DIV/01	0.000000	#DIV/01	#DIV/01	0.000000	0.000000
8	#DIV/01	#DIV/01	#DIV/01	#DIV/01	#DIV/01	#DIV/01				DIV/01	0.000000	#DIV/01	#DIV/01	0.000000	0.000000
9	#DIV/01	#DIV/0!	#DIV/01	#DIV/01	#DIV/0!	#DIV/0!	1	disne	nser	DIV/01	0.000000	#DIV/01	#DIV/01	0.000000	0.000000
			Die			-		uispe	nsei						
		DISPENSER	Di	Herence from A	rerage		1				Dicn	ensei	r		
Test Draft	Average	MEASUREMENT	Gravimetric	PVT	Master Meter	DISPENSER		read	Ings		Dish	CHSCI	L		
[#]	[kg]	[kg]	[%]	[%]	%	[%]			8				•		
1	#DIV/01		#DIV/01	#DIV/01	#DIV/01	#DIV/ui				6	mna	red t	n 3		
2	#DIV/01		#DIV/01	10/01	#DIV/01	#DIV/0!					Jinpa	i cu t	05		
3	#DIV/01		#DIV/01	#DIV/01	#DIV/01	#DIV/0!									
4	#DIV/01		#DIV/01	#D17/01	#DIV/01	#DIV/0					met	hode			
5	#D17/01		#D17/01	#D17/01	#DIV/0	#DTV/0:					met	nous			
7	#DIV/01	N /	#DIV/01	#DIV/01	#DIV/01	#DIV/0									
8	#DIV/01		#DIV/01	#DIV/01	#DIV/01	#DIV/0!					ave	rage			
9	#DIV/01		#DIV/01	#DIV/01	#DIV/01	#DIV/0!						- "SC			
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Figure 14. Results Worksheet.

4. Test draft protocol

WARNING: Only authorized personnel are to operate the FTS.

WARNING: H₂ gas sensors need to be in place in the weighing area during testing.

WARNING: Flame detector needs to be in the filling area before filling the H₂ FTS.

WARNING: The FTS must be kept in the vertical position during testing. Use the aluminum hand truck with a 2" ratchet strap that travels with the FTS to secure it in the vertical position.

4.1 Personal protective equipment

During field tests, steel toed shoes and safety glasses are required.

4.2 Environmental conditions

During field tests, the ambient temperature should be between 7.5 $^{\circ}$ C (45 $^{\circ}$ F) and 34 $^{\circ}$ C (93 $^{\circ}$ F), the range the measurement uncertainty is applicable over. Because the DAQ box and laptop are not water proof, if rain, snow or ice is present, testing cannot be performed unless there is a sufficiently covered area at the test site. If wind speeds are high enough the weighing tent cannot be stabilized, testing cannot be performed unless a suitable weighing area is available on site.

4.3 Pre-test check list

Before any person begins a field test, the following checklist must be followed:

- \Box Section 1 of this document must be fully read and understood for safe operation of the FTS.
 - $\hfill\square$ Make sure the location of valves and what they control are known.
 - □ Understand how the over pressurization safety valves work and know where they vent to.
- □ Section 2 of this document must be fully read, understood and abided by for the safe set up of test and DAQ equipment.
 - □ Make sure the DAQ box and laptop is at least 1.5 m (5 ft) away from the FTS filling and weighing areas.
 - □ Determine if the scale readout can be located outside of the weighing area, and if not, be sure to install the plug into the back of the readout that makes it safe for use in explosive environments.

- \Box Make sure the H₂ sensors are installed in the weighing area and functioning.
- □ Make sure the IR/UV flame detector is functioning and positioned a minimum distance of 25 m (82 ft) from the FTS and aimed towards it during filling and weighing.
- □ Section 3 of this document must be fully read and understood for the proper acquisition of data and to assure the quality of the calculated results.
 - □ Make sure the toggle switches in the DAQ software are in the proper positions (Section 3.2).
- □ Section 4 of this document must be fully read and understood to assure the operator knows what to do and anticipate during testing.
- \Box The FTS tank should be under a slight pressure of inert (He or N₂) gas and all valves in the closed positions during travel and therefore should be in this state upon arrival.
 - □ If the FTS is found to not be under pressure, it is assumed a leak occurred during transport and the cause must be resolved before testing can begin.
 - \square <u>H₂ can only be introduced into the FTS if an inert gas is present</u>.
- □ The master meter has been zeroed according to the manufacturer's instructions [9].
- \Box All test equipment must be grounded to earth.

4.4 Initial FTS tank preparation

The FTS tank is filled with approximately 344 kPa (50 psi) of inert (He or N_2) gas that needs to be purged before the start of testing. Gas purity needs to be established so no more than 0.02 % error occurs in the mass measurement in the FTS tank for the master meter and *PVT* methods. For the initial tank preparation, the tank pressure can be read from the gauge on the FTS tank. To prepare the FTS for test drafts:

- 1. Open Valve #3 and open the regulator to less than 1.4 MPa (200 psi) so the gas is slowly released.
- 2. Open Valve #1 (inlet) and connect the plumbing from the facility dispenser to the FTS inlet.
- 3. Fill the FTS to approximately 20.7 MPa (3000 psi) with H_2 gas from the dispenser.
 - a. Close Valve #1 and disconnect the inlet plumbing if necessary to move the H₂ FTS to the blowdown area, otherwise it can be left plumbed.
 - b. The handheld combustible gas detector can be used at this time to check for H_2 leaks at the valve stems and joints.
- 4. Connect the outlet plumbing on the H₂ FTS tank to the facility plumbing for directing the gas into the blow-down area.
- 5. Relieve tank pressure by opening Valve #3 and opening the regulator to less than 1.4 MPa (200 psi) (the maximum the regulator should be set to) so the gas is slowly released.
 - a. Do not completely void the tank; leave it under approximately 1.4 MPa (200 psi).
- 6. Close Valve #3
 - a. Disconnect outlet plumbing if necessary to move the FTS to the filling area, otherwise it can be left plumbed.
- 7. Repeat steps 1 through 5 two more times if the inert gas is He and three more times if it is N_2 before the start of a test draft.

4.5 Taking a test draft

Before the start of the initial test draft, the FTS tank should be under approximately 1.4 MPa (200 psi) of H₂ gas as outlined in Section 4.4, all DAQ components connected to the DAQ box properly, and the DAQ box, laptop and scale powered. To start taking test drafts follow the procedure:

- 1. Open the LabView program and click the start arrow (Fig. 11) to start data acquisition.
- 2. Open valve # 4 (low range pressure sensor)
- 3. Record the pressure and temperature in the FTS to get the initial density (ρ_1) and therefore the FTS tank mass pre-fill. Quasi-thermal equilibrium should be reached prior to measurements. After the initial tank preparation (Section 4.4), a minimum wait time of 20 minutes should elapse before taking measurements.

- a. This is done by using the "Average Data" function (Section 3.4). A minimum of 60 average readings should be acquired.
- 4. Stop the software by clicking the "Stop' button (Fig. 11).
- 5. Open the "Average Data" file generated to and rename it "PVT1_Test_Draft 1 mm_dd_yyyy", where the "1" following "PVT" denotes the pre-fill state and the "1" following "Test_Draft" denotes the test draft number.
 - a. Copy only the data and paste it into the Excel data reduction workbook as instructed in Section 3.5.
- 6. Close Valve # 4 (low range pressure sensor).
- 7. Disconnect the pressure and temperature sensors from their DAQ box plugs and neatly coil the wires and attach them to the FTS frame via Velcro wraps so nothing is hanging off the FTS.
- 8. If using the laptop to acquire data from the scale, start the software again by clicking the forward arrow (Fig. 11) to start data acquisition.
- 9. Weigh the "empty" FTS to get the apparent mass measurement, m_{a1} . Weigh the FTS a minimum of two times, taking it on and off the scale between each weighing. Wait a minimum of 30 seconds after placing the FTS on or taking it off the scale before taking measurements.
 - a. A minimum of two people should be used to lift the FTS on and off the weighing platform.
 - b. Nothing should be touching or hanging from the FTS during weighing.
 - c. Before the FTS is initially weighed, a zero must be read from the scale. A zero must be read between each weighing of the FTS.
 - d. If the scale is being read via the DAQ program, then use the "Average Data" function repeatedly (Section 3.4) to record the scale readings, otherwise, manually record the zero and the FTS readings. A minimum of 60 individual readings should be acquired to calculate the average during each weighing.
 - e. If the scale is being manually read, record the readings from the atmospheric monitor to enter into the data reduction workbook.
- 10. If the laptop was used to acquire data from the scale, stop the acquisition program.
- 11. Open and rename the "Average Data" file generated to: "M1_Test_Draft 1 mm_dd_yyyy", where the "1" following "M" denotes the pre-fill state and the "1" following "Test_Draft" denotes the test draft number.
 - a. Copy only the data and paste it into the Excel data reduction workbook as instructed in Section 3.5.
- 12. Connect the inlet plumbing from the dispenser to the FTS and reconnect the temperature sensors and the high range pressure sensor to their respective DAQ box plugs.
 - a. This is so the pressure and temperature can be monitored in real time during filling.
- 13. Provide the master meter with power.
 - a. This is so the meter electronics will be warmed up before measurements are made with it.
- 14. Re-start the data acquisition software.
- 15. Open Valve #1 (inlet) and connect the inlet plumbing.
- 16. Fill the FTS to either the specified mass or pressure from Table 1 with H₂ gas.
 - a. SAE J2601, 2010 <u>must</u> be abided by for the appropriate filling pressure ramp rate (PRR). This will vary by dispenser type and ambient temperature.
 - b. The tank will warm as it is filled, but not uncomfortably hot to touch if SAE J2601 is abided by.
 - c. <u>If any *hissing* noises are heard it may indicate a leak and the filling should be stopped until the noise can be investigated</u>. If it is not clear where the leak is, the hand held combustible gas detector can be used to identify the leak. In the event of a slow leak, open Valve #3 and open the regulator to less than 1.4 MPa (200 psi) and allow the tank to depressurize. if the leak cannot be repaired, the tank should be purged of residual H₂ for transport back to NIST (Section 5)
- 17. Close Valve # 1 after filling.

- 18. Wait for quasi-thermal equilibrium and record the pressure and temperature in the FTS to get ρ_2 and therefore the mass in the FTS tank after filling. Quasi-thermal equilibrium should be reached prior to measurements. After filling the FTS tank, a minimum wait time of 20 minutes should elapse before taking measurements.
 - a. This is done by using the "Average Data" function (Section 3.4). A minimum of 60 average readings should be acquired
- 19. Stop the software by clicking the "Stop' button (Fig. 11).
- 20. Open and rename the "Average Data" file generated to: "PVT2_Test_Draft 1 mm_dd_yyyy", where the "2" following "PVT" denotes the post-fill state and the "1" following "Test_Draft" denotes the test draft number.
 - a. Copy only the data and paste it into the Excel data reduction workbook as instructed in Section 3.5.
- 21. Open and rename the "file_number x" data file generated to: "Test_Draft 1 Fill mm_dd_yyyy".
 - a. This file is for filling temperature and pressure information only and does not go into the data reduction workbook.
- 22. Disconnect the inlet plumbing.
- 23. Disconnect the temperature and pressure sensors, and power to the master meter. Neatly coil the wires and attach them to the FTS frame via Velcro wraps so nothing is hanging off the FTS.
- 24. If using the laptop to acquire data from the scale, start the software again by clicking the forward arrow (Fig. 11) to start data acquisition.
- 25. Weigh the "full" FTS to get the apparent mass measurement, m_{a2} . Weigh the FTS a minimum of two times, taking it on and off the scale between each weighing. Wait a minimum of 30 seconds after placing the FTS on or taking it off the scale before taking measurements.
 - a. A minimum of two people should be used to lift the FTS on and off the weighing platform.
 - b. Nothing should be touching or hanging from the FTS during weighing.
 - c. Before the FTS is initially weighed, a zero must be read from the scale. A zero must be read between each weighing of the FTS.
 - d. If the scale is being read via the DAQ program, then use the "Average Data" function repeatedly (Section 3.4) to record the scale readings, otherwise, manually record the zero and the FTS readings. A minimum of 60 individual readings should be acquired to calculate the average during each weighing.
 - e. If the scale is being manually read, record the readings from the atmospheric monitor to enter into the data reduction workbook.
- 26. If the laptop was used to acquire data from the scale, stop the acquisition program.
- 27. Open and rename the "Average Data" file generated to: "M2_Test_Draft 1 mm_dd_yyyy", where the "2" following "M" denotes the post-fill state and the "1" following "Test_Draft" denotes the test draft number.
 - a. Copy only the data and paste it into the Excel data reduction workbook as instructed in Section 3.5.
- 28. Connect the temperature and pressure sensors to the DAQ box and provide power to the master meter.
- 29. Connect the outlet plumbing from the master meter to the blow-down area.
- 30. Start the acquisition program.
- 31. Make sure the regulator is completely backed off so no flow will occur when Valve #3 is opened.
- 32. Open Valve # 3 (valve to master meter).
- 33. Manually record the totalized mass reading from the master meter transmitter.
 - a. The units should be kg, if they are not, tap the "scroll" button on the meter transmitter until that unit is displayed.
- 34. Increase the regulator to no more than 1.4 MPa (200 psi) which will initiate the blow-down of the FTS gas through the master meter.

- a. <u>1.4 MPa (200 psi) is the maximum the regulator should be set to</u>. This is to prevent bubbles of absorbed H_2 from forming in the BUNA-N regulator seating material that will ruin the regulator.
- b. The gas exiting the FTS will become very cold (< -30 °C) and therefore caution should be taken to not touch the outlet plumbing during the blow-down. Frost will be present to indicate it is cold.
- c. The FTS outlet has a check valve with a stainless steel ball that may *rattle* during the blow-down, this is normal.
- d. The gas should be blown down until the FTS tank pressure is within 172 kPa (25 psi) of the starting pressure before filling the tank. <u>This pressure must always be below the range of the installed low range *P* sensor.</u>
- 35. When done blowing the gas down, dial back the regulator and close Valve # 3.
- 36. Manually record the totalized mass reading from the master meter transmitter.
- 37. Open Valve # 4 (low range pressure sensor)
- 38. Wait for quasi-thermal equilibrium and record the temperature and pressure (via the low ranged pressure sensor) in the FTS to get the mass in the FTS tank post-blow down. After blowing the FTS tank down, a minimum wait time of 20 minutes should elapse before taking measurements.
 - a. This is done by using the "Average Data" function (Section 3.4). A minimum of 60 average readings should be acquired.
 - b. This mass is used in the master meter calculations and is the pre-fill mass (Step #3) for the next test draft.
- 39. Stop the software by clicking the "Stop' button (Fig. 11).
- 40. Open and rename the "Average Data" file generated to: "BD_Test_Draft 1 mm_dd_yyyy", where "BD" stands for blow-down and the "1" following "Test_Draft" denotes the test draft number.
 - a. Copy only the data and paste it into the Excel data reduction workbook as instructed in Section 3.5.
 - b. The subsequent Input Worksheet uses this data for the pre-fill mass calculation for the next test draft.
- 41. Open and rename the "file_number x" data file generated to: "Test_Draft 1 blow-down mm_dd_yyyy".
 - a. This file is for filling temperature and pressure information only and does not go into the data reduction workbook.
- 42. Enter the transmitter readings into the Excel data reduction spreadsheet as instructed in Section 3.5.
- 43. Repeat steps 6 42 for subsequent test drafts. Name the data files accordingly.

Table 1 gives the desired mass (*m*) of H₂ gas and the corresponding pressures (*P*(*m*)) at 20 °C that are to be collected during field tests. The expected expanded uncertainties are included (k = 2).

<i>m</i> [kg]	<i>P(m)</i> [MPa]	Gravimetric U(k=2)	<i>PVT U</i> (<i>k</i> = 2)	master meter <i>U</i> (<i>k</i> = 2)
0.5	17	0.57	1.8	0.45
1	35	0.29	0.9	0.46

Table 1. Desired mass and corresponding tank pressure of H₂ gas collected during test drafts.

4.6 Emergency stop procedure

The reasons a test would need to be abruptly stopped is: 1) in the event of a fire or 2) other incident that causes the H_2 FTS tank to become over pressurized.

If a fire is in direct contact with the H₂ FTS tank, the type 3 cylinder wrap insulation properties will prevent the internal gas temperature (and hence pressure) from increasing significantly above the pre-fire value. Therefore, the over pressurization features will not relieve the gas before the tank will rupture due to material failure. The IR/UV flame detector will alert to fire presence. If a fire occurs in any part of the testing area that <u>can</u> be quickly extinguished, the fire should be immediately extinguished by following the protocol specific to the dispenser facility. If the H₂ FTS tank is under pressure, open Valve #3 and open the regulator to less than 1.4 MPa (200 psi) so the gas is slowly released away from the present fire. Tests of Type 3 H₂ cylinders show that if exposed directly to fire, it takes approximately 12 minutes for the tank to soften and rupture [10].

If a fire occurs in any part of the testing area that <u>cannot</u> be quickly extinguished:

- 1. The area should be immediately vacated to a distance of more than 110 m (361 ft) [10].
- 2. The H₂ FTS should be abandoned and the proper authorities notified so the fire can be extinguished.

In the event the tank becomes over pressurized without the presence of a fire:

- 1. Slowly open valve #3 and the regulator to less than 1.4 MPa (200 psi) so the gas is slowly released.
- 2. If the pressure relief valve opens before the gas can be manually relieved, allow the gas to release until the relief valve reseals. If the valve does not completely re-seal in a timely manner, open Valve #3 and the regulator to less than 1.4 MPa (200 psi) so the gas is slowly released and the pressure relief valve will re-seal.

4.7 Assuring proper operations

There are multiple scenarios that can happen to cause testing to be halted or that invalidates the results. None of these scenarios will cause a hazard; however, if such a scenario occurs it needs to be known.

The scenarios that will <u>halt testing</u> are: 1) complete facility power failure that cannot be restored in a timely manner and 2) leaks from the FTS tank that cannot be repaired on site.

A complete facility power failure will most likely prevent the dispenser equipment from operating properly. If this is not the case, it will however, prevent measurements to be taken from the scale and temperature and pressure sensors. Furthermore, the safety equipment (H_2 sensors with alarms and the flame detector) and the master meter will not have power. If power failures are intermittent, depending on the frequency, it may be best to halt testing. The decision to continue testing with intermittent power failures will be determined by the engineer performing the test drafts and the dispenser owner/manager on site.

Leaks in the FTS plumbing may or may not require a halt in testing. Leaks that are identified and can be repaired on site will not require testing to be stopped. <u>The following leaks **WILL** halt testing</u>:

- 1. Leaks past a valve stem.
 - a. These leaks may be hard to detect as they will likely be very *small*. The handheld combustible gas detector should be used to check for small leaks at the first pressurization of the FTS with H_2 . If a leak develops after this initial check, then the scale will indicate there is a leak. When the FTS is weighed in the *full* state, the scale mass measurement will slowly decrease. Therefore, it is important to leave the FTS on the weighing pan until the reading is stable for more than 30 seconds.
 - b. If the leak is *large*, a hissing noise will be heard and the leak can be quickly identified.
- 2. If the pressure regulator seating fails.
 - a. A failure in the regulator will halt testing because the gas cannot be blown-down in a controlled manner. The lack of controlled release may damage elastomer materials in the FTS tank.
- 3. If pressure relief valve #1 on the FTS tank fails to stay closed because the FTS tank will not be able to hold gas.
 - a. This is the relief valve that cannot be isolated from the FTS tank.

The following leaks and scenarios will **NOT** cause testing to be stopped:

- 1. If the valve seating material in Valves #1, #2 and #3 fails or the operator cannot close one of these valves.
 - a. Valve #1 has a check valve upstream from it and will not allow gas to escape from the FTS tank.
 - b. Valve #2 has a check valve upstream from it and will not allow gas to escape from the FTS tank.
 - c. Valve #3 is upstream of the pressure regulator that will not allow gas to escape from the FTS tank.
- 2. If the valve seating in Valve #4 fails or the operator cannot close this valve.
 - a. The low range pressure sensor can be removed and the port capped. The pressure relief valve #2 can also be capped.
 - b. The consequence of this is the pressure measurements made during the "empty" state will have larger uncertainty than desired.
- 3. If the pressure relief valve that protects the low range pressure sensor develops leaks during testing, Valve #4 can be kept closed and testing resumed.
 - a. The consequence of this is the pressure measurements made during the "empty" state will have larger uncertainty than desired.
- 4. If the laptop or DAQ components fail.
 - a. Only the gravimetric method can be performed due to lack of temperature and pressure data that is needed for the *PVT* and master meter methods.
 - b. To calculate the tank volume for buoyancy corrections, the tank pressure gauge can be read and the atmospheric temperature used.
 - i. This will introduce extra uncertainty in this measurement; however, the magnitude will have to be calculated off site after the fact.
 - c. The environmental monitor can be manually read from its display for buoyancy corrections.
- 5. If the pressure sensors develop leaks past their diaphragms.
 - a. Gas may or may not leak out of the FTS tank. <u>Leaking gas from the high range pressure sensor</u> will cause the testing to halt. This is because it cannot be isolated from the FTS tank.
 - b. Diaphragm leaks will damage the sensors and their measurements will be invalid.
 - c. When these pressure sensors become damaged, they typically output a single voltage; therefore, if the software does not show the sensors responding during filling the tank, they are damaged.
 - d. If the high range pressure sensor becomes damaged, only the gravimetric method can be used.
 - e. If the low range pressure sensor becomes damaged, it can be valved off and the high range sensor used.
- 6. If the temperature sensors become damaged in the field.
 - a. Damaged TCs output a large value (>10000); which is easily seen in the data acquisition software.
 - b. If one of these sensors is damaged, testing can be resumed without the second temperature measurement. If both sensors are damaged, they may be able to be replaced in the field, see Section 6. If they cannot be replaced in the field, only the gravimetric method can be performed.

The H₂ FTS is rugged so operator errors will lead to little or no damage. If the operator accidently opens Valve #4 while the FTS is pressurized above 1.4 MPa (200 psi), the pressure relief valve #2 (Fig. 2) will open and gas will vent from the top of the FTS, which is approximately 2 m (7 ft) high. This situation will not cause testing to be halted.

5. On-site tear down

When testing is complete, the H_2 FTS tank should be completely voided of H_2 gas by blowing it down in the blow-down area following the final gas density measurement in the tank. The check values at the inlet and

outlet of the FTS tank will prevent atmospheric air from entering the tank and hence atmospheric pressure of H_2 gas will remain in the tank. Therefore, the "inlet to purge" port (Fig. 2) can be used to purge the FTS tank using nitrogen for transport back to NIST. A small compressed gas bottle of nitrogen, a regulator for it, and 0.635 cm (0.25 in) diameter copper tubing with compression fittings for plumbing into the FTS tank will travel with the FTS. To purge the tank for travel:

- 1. Blow-down all H_2 gas until the FTS tank has only atmospheric pressure H_2 left.
- 2. Connect the nitrogen bottle to the purge port (Fig. 2).
- 3. Open Valve #2 and fill the FTS tank to approximately 700 kPa (100 psi).

a. The round pressure gauge on the FTS tank can be used to aid in this.

- 4. Repeat steps 1 through 3 one more time.
- 5. Blow-down the nitrogen so atmospheric pressure is left in the FTS tank.

All DAQ components and scale components have their own respective carrying cases and will be repacked in the same manner they were unpacked upon arrival for transport back to NIST. There is no specific order that needs to be followed for disassembling the test area.

6. Changing T sensors

In the event one or both of the installed TCs quit working while in the field, they cannot be replaced in the field unless there is enough inert gas to replace the air in the tank prior to introducing H_2 into the tank and to be able to purge the FTS tank for transport back to NIST after testing.

- 1. Connect the outlet plumbing on the H₂ FTS tank to blow-down area.
- 2. Relieve tank pressure by opening Valve #3 and opening the regulator to less than 1.4 MPa (200 psi) so the gas is slowly released.
 - a. Void the tank as much as possible.
- 3. Remove the damaged TC(s) along with the fittings, *i.e.*, remove the large fitting holding the entire TC assembly, not the small nut directly on the TC.
- 4. Install the traveling spare(s). Plumbing is already attached to the spares.
- 5. Be sure to plug in the spare TC(s) into the proper place on the DAQ box front (Fig. 5) when making measurements.
- 6. Follow instructions in Section 5 to get N_2 into the FTS.
- 7. Follow instructions in Section 4.4 to assure pure H_2 is in the FTS before taking a test draft.

7. Calculations

All calculations are included in the Excel file "H₂ FTS Laptop Reduction Workbook".

7.1 PVT method

The mass dispensed (Δm_{PVT}) into the H₂ FTS is calculated by:

$$\Delta m_{PVT} = \rho_2 V_2 - \rho_1 V_1 , \qquad (1)$$

where ρ_1 and ρ_2 are the initial and final gas densities, and V_1 and V_2 are the initial and final volume of the H₂ FTS tank, respectively. The gas density is defined by:

$$\rho = \frac{PM}{zRT},\tag{2}$$

where P is the gas pressure, M is the gas molar mass, R is the universal gas constant, T is the gas temperature, and z is the compressibility factor. Refprop 23 [11] is used for density calculations. The FTS tank volume is a function of T and P and is given by:

$$V = V_{\rm ref} [1 + \lambda \Delta P] [1 + 3\alpha \Delta T], \qquad (3)$$

where V_{ref} is the FTS tank volume at 20 °C and 101 kPa, λ is the pressure expansion coefficient, α is the linear thermal expansion coefficient, and ΔT and ΔP are the difference of the temperature and pressure from the

reference values, respectively. Inserting Eqn. 2 and 3 into Eqn. 1 gives the governing equation for the mass dispensed via the *PVT* method:

$$\Delta m_{PVT} = \frac{V_{\text{ref}}M}{R} \left[\frac{P_2}{z_2 T_2} [1 + \lambda \Delta P_2] [1 + 3\alpha \Delta T_2] - \frac{P_1}{z_1 T_1} [1 + \lambda \Delta P_1] [1 + 3\alpha \Delta T_1] \right].$$
(4)

7.2 Gravimetric Method

The mass dispensed ($\Delta m_{\rm grv}$) into the H₂ FTS is calculated by:

$$\Delta m_{\rm grv} = m_2 - m_1,\tag{5}$$

where m is the true mass and the subscripts 1 and 2 denote the mass of the FTS pre-filling and post-filling, respectively.

The scale indicated mass (m_a) is buoyancy corrected to determine the true mass via the equation: $m = m_a + \rho_{air}V$; where ρ_{air} is the density of the air that is a function of atmospheric *P*, *T*, *RH* and the molecular weight of air. *V* is the volume of the H₂ FTS tank. Therefore:

$$\Delta m_{\rm grv} = m_{a2} + \rho_{\rm air2} V_2 - [m_{a1} + \rho_{\rm air1} V_1]. \tag{6}$$

The volume of the FTS tank is given by Eqn. 3. Inserting Eqn. 3 for the volume leads to the governing equation for mass determination for the gravimetric method:

$$\Delta m_{\rm grv} = m_{a2} - m_{a1} + V_{\rm ref} [\rho_{\rm air2} [1 + \lambda \Delta P_2] [1 + 3\alpha \Delta T_2] - \rho_{\rm air1} [1 + \lambda \Delta P_1] [1 + 3\alpha \Delta T_1]].$$
(7)

7.3 Master meter method

Following the fill of the FTS tank, the gas is blown-down through a coriolis master meter. The mass that filled the FTS tank (Δm_{MM}) is calculated by:

$$m_{\rm MM} = \Delta m_{\rm CM} + \Delta m_{\rm FTS},$$

where Δm_{CM} is the difference in totalized mass indicated on the meter transmitter before and after the blowdown, Δm_{FTS} is the change in mass in the FTS tank following the blow-down through the master meter from before the tank was filled (mass post blow-down – mass pre-filling).

8. Operating specifications for H₂ FTS components and instrumentation

<u>Plumbing, fittings and adaptors:</u> All plumbing, fittings and adaptors on the FTS tank are made of 316 stainless steel. The minimum working pressure rating is 52 MPa (7500 psi).

<u>H₂ filling dispenser receptacle</u>: The dispenser receptacle is from the WEH Company, part # C1-31316-X1. This is a standard H₂ service receptacle that will only fit 35 MPa dispenser nozzles.

<u>FTS H₂ tank</u>: The FTS tank is a 1 kg H₂ capacity storage tank; Dynetek model #M039H350G5N8N. The tank is a type 3 cylinder, which is a seamless aluminum liner fully wrapped with a continuous filament made of carbon fiber in an epoxy reinforcement laminate [1]. The maximum pressure is 35 MPa (15 °C or 59 °F). The maximum operating temperature is 80 °C (176 °F). The tank must be decommissioned in the year 2022 as instructed by the manufacturer or after 11,200 pressure cycles, whichever comes first.

<u>Ball Valves:</u> The ball valves are High Pressure Equipment Company part #10-74AF6HYD. They have fluorocarbon FKM (Viton-A) O-rings and 30 % glass filled PEEK ball seating material. The operating temperature range is -73 $^{\circ}$ C (-100 $^{\circ}$ F) to 177 $^{\circ}$ C (350 $^{\circ}$ F). The pressure rating is 69 MPa (10000 psi).

<u>Check Valves</u>: The FTS has three independent check valves and one incorporated into the nozzle receptacle. The valve at the FTS tank inlet is from High Pressure Equipment Company, part #10-41AF6 and has a pressure rating of 69 MPa (10000 psi). The other check valves located at the inlet for the purge gas and at the FTS outlet are from the WEH Company, part # C1-18485/4. These check valves are for H₂ service.

(8)

<u>Regulator</u>: The regulator is a Tescom model #26-1062-66045 non-venting pressure reducing regulator. The main valve seating material is $\text{Vespel}^{\text{(B)}}$ and the seals are Buna-N with Teflon backup O-rings. The operating temperature range is -40 °C (-40 °F) to 74 °C (165 °F). The maximum inlet pressure is 41.4 MPa (6000 psi).

<u>Thermocouples</u>: The temperature sensors are type K rugged heavy duty transition joint thermocouple probes: Omega Engineering model # TJ-CAIN-18U-18-SB. The sheath material is Inconel and has diameter of 0.5 cm (0.20 in). The junction is not exposed and it is ungrounded. The operating temperature is -200 °C (-328 °F) to 1250 °C (2282 °F). However, the probes were calibrated over the range 14 °C to 32 °C (57 °F to 90 °F). Therefore, this is the range over which their calibration is valid.

<u>Pressure sensors</u>: The pressure sensors are GP:50 model # 311Z intrinsically safe transducers. Their operating temperature range is -40 $^{\circ}$ C (-40 $^{\circ}$ F) to 80 $^{\circ}$ C (176 $^{\circ}$ F).

<u>Pressure relief valve for the low range pressure sensor:</u> The pressure relief valve is Swagelok model #SS-4R3A. The seating material and seals are fluorocarbon FKM (Viton). The operating temperature range is -4 °C (24.8 °F) to 65 °C (149 °F). The valve will open if the FTS tank pressure reaches 1.7 MPa (250 psi) \pm 5 %. The valve will reseal when the FTS tank pressure is approximately 88 % of the pressure at which it opened.

<u>Pressure relief valve for the FTS tank</u>: The pressure relief valve is Parker model #HPRVS-4A-EPR-K8-5500. The seating material and seals are ethylene propylene rubber (EPR). The operating temperature range is -57 °C (-70 °F) to 135 °C (275 °F). The valve will open if the FTS tank pressure reaches 38 MPa (5500 psi) \pm 5 %. The valve will reseal when the FTS tank pressure is approximately 88 % of the pressure at which it opened.

<u>Pressure gauge</u>: The FTS tank pressure gauge is from High Pressure Equipment Company model #4PG5. The maximum operating pressure is 35 MPa (5000 psi). The temperature operating range is well within the FTS tank temperature operating range.

9. References

- [1] Dynecell Type 3 Cylinder Operation Manual S-OP-020 Rev D, March 2012.
- [2] San Marchi, C., Somerday, B. P., Technical Reference on Hydrogen Compatibility of Materials; Nickel Alloys: Solid Solution Alloys, *Ni-Cr Alloys* (code 5110), Sandia National Laboratories, Livermore CA.
- [3] San Marchi, C., Somerday, B. P., Technical Reference on Hydrogen Compatibility of Materials; Austenitic Stainless Steels: Type 316 (code 2103), Sandia National Laboratories, Livermore CA.
- [4] San Marchi, C., Somerday, B. P., Technical Reference on Hydrogen Compatibility of Materials; Nonmetals: Polymers (code 8100), Sandia National Laboratories, Livermore CA.
- [5] NTM Sensors: Installation Guidelines for the NTM Hydrogen Alarm System. Doc. No. 241004 Rev. 130920. <u>www.ntmsensors.com</u>
- [6] ESP Safety Inc.: IPES-IR/UV Flame Detector Operating Manual 80010-001 R07.
- [7] NSS 1740.16: Safety Standard for Hydrogen and Hydrogen Systems. Office of Safety and Mission Assurance, Washington, DC 20546
- [8] Mettler Toledo: IND560x Terminal Installation Guide. 64061929 (04/2011).R05. <u>www.mt.com/support</u>
- [9] Micro Motion Model 2400S Transmitters Installation Manual. P/N 20003402, Rev. D; April 2008
- [10] Zalosh, Robert. CNG and Hydrogen Vehicle Fuel Tank Failure Incidents, Testing, and Preventative Measures. Firexplo Wellesley, MA
- [11] Lemmon, E. W., Huber, M. L., and McLinden, M. O. Refprop 23: Reference fluid thermodynamic and transport properties. *NIST Standard Reference Database 23*, Version 9 2010; National Institute of Standards and Technology, Boulder, CO.