Designation: D7606 – 11

Standard Practice for Sampling of High Pressure Hydrogen and Related Fuel Cell Feed Gases[1]

This standard is issued under the fixed designation D7606; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes a hydrogen quality sampling apparatus (HQSA) and a procedure for the sampling of high pressure hydrogen at fueling nozzles of 35 or 70 Mega Pascal (MPa) fueling stations.

1.2 This practice does not include the analysis of the acquired sample. Applicable ASTM standards include but are not limited to test methods referenced in Section 2 of this practice.

1.3 This practice is not intended for sampling and measuring particulate matter in high pressure hydrogen. For procedures on sampling and measuring particulate matter see ASTM D7650 and D7651.

1.4 The values stated in SI units are standard. The values stated in inch-pounds are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D7650 Test Method for Sampling of Particulate Matter in High Pressure Hydrogen used as a Gaseous Fuel with an In-Stream Filter

D7651 Test Method for Gravimetric Measurement of Particulate Concentration of Hydrogen Fuel

2.2 *SAE Standards*[3]

SAE J2600 Compressed Hydrogen Surface Vehicle Refueling Connection Devices

SAE J2799 70 MPa Compressed Hydrogen Surface Vehicle Fuelling Connection Device and Optional Vehicle to Station Communications

SAE TIR J2719 Information Report of the Development of a Hydrogen Quality Guideline for Fuel Cell Vehicles

2.3 *California Code of Regulations:*[4]

California Code of Regulations Title 4, Division 9, Chapter 6, Article 8, Sections 4180 – 4181

3. Terminology

3.1 *Definitions:*

3.1.1 *absolute pressure*—Pressure measured with reference to absolute zero pressure, usually expressed in MPa, mm Hg, or pound per square inch (psi).

3.1.2 *contaminant*—impurity that adversely affects the components within fuel cell or hydrogen storage systems

3.1.3 *gauge pressure*—Pressure measured above ambient atmospheric pressure. Zero gauge pressure is equal to ambient atmospheric (barometric) pressure.

3.1.4 *gaseous fuel*—Material to be tested, as sampled, without change of composition by drying or otherwise.

3.1.5 *hydrogen quality sampling apparatus (HQSA)*—an apparatus used to collect hydrogen from a 35 or 70 MPa hydrogen fueling nozzle (SAE J2600 and SAE J2799) into a sample container.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *high pressure hydrogen*—For the purposes of this practice, high pressure hydrogen is hydrogen defined as hydrogen pressurized to 35 or 70MPa.

4. Summary of Practice

4.1 This practice describes an apparatus and procedure for the sampling of high pressure hydrogen from fueling nozzles conforming to SAE J2600 or SAE J2799. This practice is intended as a guideline for ensuring collection of a representative sample without introducing trace levels of contaminants. Samples collected using this practice should be suitable for trace analysis of contaminants, utilizing a variety of analytical techniques.

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[1] This practice is under the jurisdiction of ASTM Committee D03 on Gaseous Fuels and is the direct responsibility of Subcommittee D03.14 on Hydrogen and Fuel Cells.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

[4] Available from Office of Administrative Law, 300 Capitol Mall Suite 1250, Sacramento, CA 95814–4339.

5. Significance and Use

5.1 Hydrogen is delivered to fuel cell powered automotive vehicles and stationary appliances at pressures up to 87.5 MPa. The quality of hydrogen delivered is a significant factor in maximizing fuel cell efficiency and life span. Contamination can arise during the production of fuel cell feed gases, storage containers, station tubing and fuel lines up to the nozzle used for fuel delivery. Collection of a representative fuel sample without the introduction of contamination even as low as parts-per-billion (ppb) per contaminant during collection is crucial for assessing the quality of fuel in real world applications.

5.2 This practice is intended for application to high pressure, high purity hydrogen; however, the apparatus design and sampling techniques may be applicable to collection of other fuel cell supply gases. Many of the techniques used in this practice can be applied to lower pressure/lower purity gas streams.

6. Apparatus Design

6.1 The general design of the HQSA is shown in Fig. 1, which is a depiction of the apparatus with the nozzle hydrogen pressure regulated to approximate 6.9 MPa (1000 psi) before sampling. The pressure of 6.9 MPa (1000 psi) is selected as an example since it is, generally, the lowest pressure tolerated by hydrogen station safety shutoff systems while still providing a sample that analytical laboratories can safely handle routinely. All HQSA parts, including the ventilation tubes, are made of 316 grade stainless steel (SS).

6.2 *HQSA Metal Support Plate (1, Fig. 1)*—The HQSA metal support plate is utilized to mitigate damage during transportation and support the apparatus. The HQSA is firmly fixed to a metal support plate by tube supports (2, Fig. 1.).

6.3 *Movable Adjustable Platform*—Before sampling, the metal plate holding the HQSA is firmly clamped onto a height adjustable and movable platform, such as a heavy duty cart with a hydraulic adjustable horizontal platform and brakes on its wheels. The cart is moved to a position close to the fueling station and the height of platform is adjusted so that the fueling nozzle attaches easily to the receptacle of HQSA. The platform height is adjusted to provide a safe and comfortable work space. The cart is then locked into place using the cart wheel brakes.

6.4 SAE J2799 Receptacle (3, Fig. 1) – This receptacle can adapt to both 35 and 70MPa hydrogen fueling nozzles. For safety reason, the receptacle must be positioned vertically so that the fueling nozzle attaches to the receptacle from the top. To support the weight of the fueling nozzle, the receptacle must have an additional support (3.1, Fig. 1), which is fixed to the metal support plate (1, Fig. 1).

6.5 *Main Valve (4, Fig. 1)*—The functions of the main valve are explained as follows:

6.5.1 *Station and HQSA leak test*—The station leak test is performed before hydrogen fuel sampling to ensure there are no leaks in the hydrogen fuel delivery system. For sampling the station personnel must attach the fueling nozzle to the SAE J2799 receptacle (3, Fig. 1) first while the main valve is closed. The station leak test procedure is then initiated. A hand held

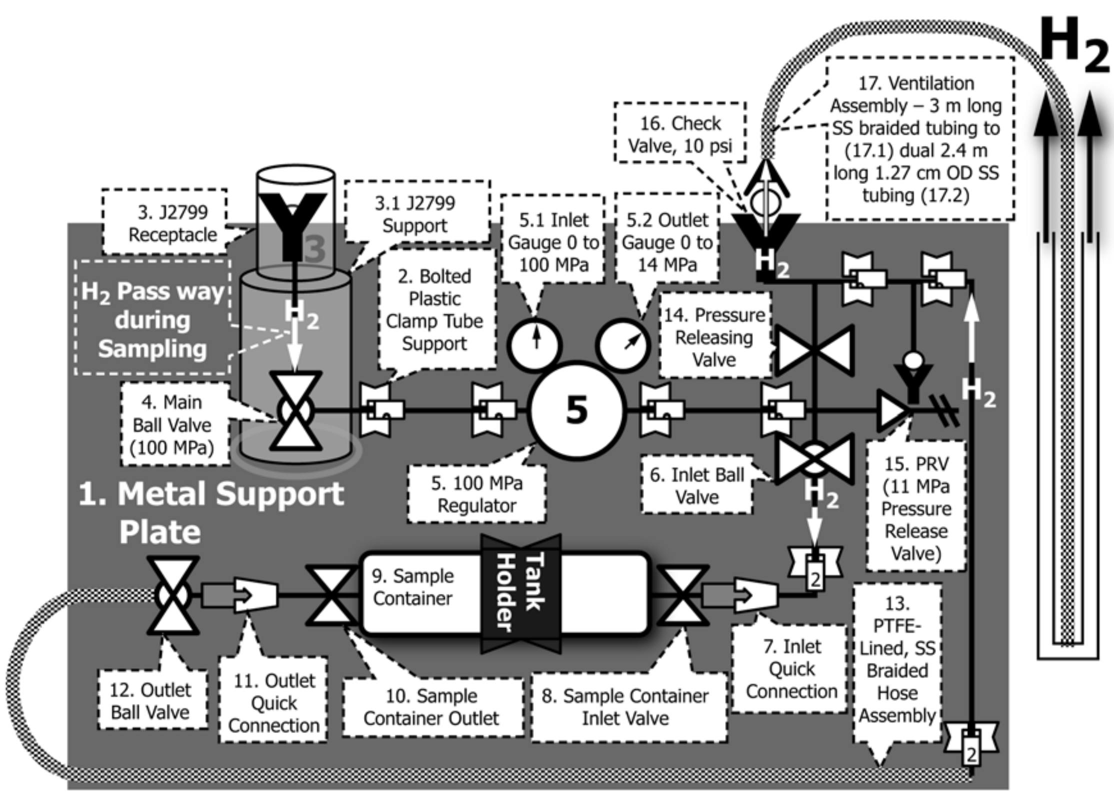

FIG. 1 Hydrogen Quality Sampling Apparatus

hydrogen leak detector is used to check for leaks around all the connections from nozzle to the main valve as in 10.7. There is residual high pressure hydrogen left in the station hose when the station is not fueling. The residual high pressure hydrogen can be used for checking the leakage in the complete HQSA system, including the sample container, as shown in 10.8.

6.5.2 *Prevention of High Pressure Hydrogen Passing through the Regulator*—If the main valve (4, Fig. 1) is not installed and station fueling starts, the diaphragm of the regulator (5, Fig. 1) may fail due to rapid hydrogen pressurization. In this case, pressure relief valve (PRV) 15, (Fig. 1) will open to release hydrogen pressure above 10.3 MPa (1500 psi). The main valve (4, Fig. 1) when closed is designed to contain high pressure hydrogen when the station sampling starts. The main valve is slowly turned to the open position and the high pressure hydrogen is regulated to 6.9 MPa (1000 psi).

6.6 *Regulator 5 (*Fig. 1*) and all connections from the* SAE J2799 *Receptacle to Regulator*—The regulator and all the connections, including tubing, tubing fittings, adapter fittings and unions from the SAE J2799 receptacle to the main valve and main valve to regulator must have a pressure rating of 103 Mpa (15,000 psi) or higher. The regulator should have two gauges, 5.1 and 5.2 in Fig. 1, to monitor both inlet and outlet pressures.

6.7 *Inlet and Outlet Valves (6 and 12,* Fig. 1*, respectively)*—The valves should be easily opened and closed, such as ball valves. The HQSA and sample container are cleaned by pressurizing and, releasing hydrogen from the HQSA and sample container (10.11) using these valves. This cleaning procedure can be repeated many times (10.11) but must be performed at least 10 times to ensure a valid sample under most sampling conditions.

6.8 *Sample Containers*—The pressure rating of the sample containers is 12.4 MPa (1800 psi). The sample containers and both inlet and outlet valves (8 and 10, Fig. 1, respectively) are internally coated with silicon (Si) since sulfur gas analysis to low ppb is required under SAE TIR J2719 and other hydrogen fuel quality specifications. To avoid air contamination during sampling, both inlet and outlet valves of sample containers are equipped with quick connects – one end of the container has a quick-connect stem and the other end a quick-connect body. These two quick-connections (7 and 11, Fig. 1) and the inlet and outlet valves provide a double seal on both ends of the sample container.

6.9 *HQSA Pressure Release Valve (14,* Fig. 1*)*—The valve is always closed before and during sampling. After collection, the pressure release valve is opened before removal of the pressurized and sealed sample container to release the hydrogen pressure in the HQSA through a check valve (16, Fig. 1). Hydrogen at 6.9 MPa (1000 psi) contained inside the HQSA must be released before next sample container can be safely connected to the inlet quick-connection (7, Fig. 1).

6.10 *Proportional Release Valve (PRV, 15,* Fig. 1*)*—The PRV is set at 10.3MPa (1500 psi) to protect the 12.4 MPa (1800 psi) pressure proof sample container.

6.11 *Check Valve*—The check valve (16, Fig. 1) with 69 Kilo Pascal (KPa) (10 psi) crack pressure is installed at the vent of the HQSA (Fig. 1) to prevent air from back diffusion into the HQSA.

6.12 *Ventilation Assembly (17,* Fig. 1*)*—The ventilation assembly contains a 3 meter (m) (10 ft) long SS braided tubing interfaced to a dual 2.4 m (8 ft) long 1.27 cm (½ in.) Outside Diameter (OD) SS tubing which is kept vertical to the ground. During sampling, the hydrogen fuel flows through HQSA, then the check valve (16, Fig. 1), through the a 3 m long SS braided tubing (17.1, Fig. 1) and dual 2.4 m long 1.27 cm (½ in.) OD SS tubing (17.2, Fig. 1), before venting to atmosphere at approximately 2.4 m (8 ft) above ground.

7. Additional Equipment Needed

7.1 *Hydrogen Leak Detector*—A hydrogen leak detector is a required as a safety device needed to detect hydrogen gas leaks when the HQSA is pressurized. Leak detection using soap bubbles must not be used due to possible moisture contamination.

8. Hazards

8.1 *High hydrogen pressure*—The hydrogen pressure can be as high as 87.5MPa and constitutes both an explosion and fire hazard.

8.2 The total mass of hydrogen passing through the HQSA during a sampling event is approximately 1 kilogram (kg). Smoking, camera flashes, or mobile phone usage is unsafe within 7.6 m (25 ft) of either the ventilation tubing (6.12) or hydrogen fueling station itself. Additional safety precautions must be taken as necessary to prevent fire or explosion, or both.

8.3 *Static Charges*—During gaseous sampling, the extremely high-speed hydrogen flow rate may generate a static charge on HQSA components. The static charge is removed by grounding the HQSA with a wire from hydrogen fueling station or other available grounding wire(s).

9. HQSA Cleaning

9.1 Do not clean the HQSA with water, iso-propanol or any other solvent.

9.2 The HQSA must be cleaned by purging during sampling. This is done by flowing one kilogram hydrogen fuel through the HQSA after the nozzle pressure is regulated to 6.9 MPa (1000 psi). The hydrogen flow rate at 1000 psi is approximately 33.3 grams per second for a total sampling time of around 30 s. At this flow rate, SAE TIR J2719 targeted constituents will be removed from within the HQSA and sampling lines. This procedure is the best way to conveniently dehydrate and remove residual sulfur gases from the HQSA, sampling line, and sample container. Dehydration of the apparatus cannot be safely achieved at the station through evacuation or heating, or both.

10. Sampling Procedures

10.1 *Safety Precaution*—During gaseous sampling, personnel must wear goggles, safety shoes and a flame resistant lab coat or other industrial flame resistant clothing. Personnel not directly involved in sampling should beat least 5 m away from the HQSA during sampling.

10.2 Attach a ground wire to the station ground.

10.3 Fix the HQSA onto a platform of a hydraulic cart (6.3).

10.4 Connect HQSA to the ventilation system (6.12).

10.5 Attach a 1-L 1800 psi stainless steel sample container to HQSA through quick connections (7 and 11, Fig. 1).

10.6 Make sure all the valves of HQSA (Fig. 1) are closed and attach the station 35 or 70 MPa nozzle to the SAE J2799 receptacle (3, Fig. 1).

10.7 Start the station leak test using a hand held hydrogen leak detector to check leaks around all the connections from the nozzle to the main valve (4, Fig. 1). Any leak must be eliminated before proceeding.

10.8 Open the main valve (4, Fig. 1) slowly and regulate the nozzle pressure (5, Fig. 1) down to 6.9 MPa (1000 psi).

10.8.1 Use a hydrogen leak detector to check for leaks around all the connections from the main valve to the inlet valve (6, Fig. 1), including the regulator.

10.8.2 If leaks are not present, open the inlet valve and check for leaks from the inlet valve to the sample container inlet valve (8, Fig. 1).

10.8.3 If leaks are not present, open the sample container inlet valve and check for leaks around the sample container inlet valve, especially at the moving stem of the valve and the connections between sample container body and both sample container valves.

10.8.4 If leaks are not present, open the sample container outlet valve (10, Fig. 1) to leak check down to the outlet valve (12, Fig. 1).

10.8.5 All the leaks found in this 10.8 must be eliminated before proceeding.

10.9 Close the main valve and open the outlet valve (12, Fig. 1) to vent hydrogen to atmosphere. After most of the hydrogen is released, close the outlet valve immediately.

10.10 Ensure the main valve is closed and start the station fueling process. Slowly turn the main valve to the open position and regulate the hydrogen pressure down to 6.9 MPa (1000 psi).

10.10.1 Open the inlet valve (6, Fig. 1) to pressurize the sample container with hydrogen. Close the inlet valve once the system has been pressurized.

10.10.2 Open the outlet valve (12, Fig. 1) to release hydrogen to ambient pressure and close the outlet valve when this has occurred.

10.10.3 Repeat the procedures 10.10.1 and 10.10.2 nine additional times.

10.11 Open the inlet and outlet valve in sequential order to let hydrogen flow through the sample container and record the time as the sample start time. Since the hydrogen flow is fast through the HQSA and sample container, the regulated hydrogen pressure should be lower than 6.9 MPa (1000 psi) during sampling. The station meter or fueling computer should show the total amount of hydrogen in kg being sampled. Close the sample container outlet valve and record the ending sampling time once 1 kg of hydrogen has flowed through the HQSA and sample container. The regulated hydrogen pressure should be back to 1000 psi. Close the sample container inlet valve. In general, the station fueling process will be shut down when closing the sample container outlet valve. If not, shut down the station fueling process.

10.12 If additional sampling is to be performed, do not remove the fueling nozzle. Instead, close the main valve. Open the HQSA pressure release valve (14, Fig. 1) to release the pressure inside the HQSA to atmospheric pressure, excepting the pressure remaining between the receptacle and main valve (3 to 4, Fig. 1). The check valve (16, Fig. 1) will prevent gas back flow from entering the HQSA and leave 10 psi hydrogen pressure inside the HQSA. Close the HQSA pressure release valve, inlet and outlet valves (6 and 12, Fig. 1, respectively).

10.13 Remove the sample container from the HQSA. Record the beginning and ending sampling time and the detailed sample identifications on a sample container label. The hydrogen nozzle pressure read from the inlet gauge (5.1, Fig. 1) during sampling and the weight of hydrogen sampled from the station meter or fueling computer are also recorded on the label.

10.14 For each additional sample container used, repeat the procedures of 10.5 and 10.7 – 10.13. In general, two to three sample containers are taken for a hydrogen sample at a fueling station since the analyses of two sample containers for each sample may be necessary to prove the existence and validate the amount of a contaminant in a hydrogen fueling system. For example, a sample container of a hydrogen sample is found to contain 5 ppmv oxygen and 20 ppmv nitrogen. It may be necessary to analyze another sample container of the same hydrogen source to validate whether the first sample container was contaminated by air. Likewise, if a sample container of a hydrogen sample contains hydrogen sulfide at high concentration, another sample container must be analyzed to validate the presence of hydrogen sulfide at this level.

10.15 After sampling, perform the following procedures to complete the sampling event.

10.15.1 Make sure hydrogen station fueling is discontinued and remove the fueling nozzle from the SAE J2799 receptacle (3, Fig. 1) of the HQSA.

10.15.2 Open the main valve, inlet valves, and the HQSA pressure release valve (4, 6, and 14, Fig. 1, respectively) to vent hydrogen inside the HQSA for 5 s. Once the hydrogen pressure has been reduced to ambient levels close all the valves of the HQSA.

10.15.3 Separate the 3 m long SS braided tubing (17.1, Fig. 1) from the dual 2.4 m long½ in. (1.27 cm) OD SS tubing (17.2, Fig. 1) and cap the end of the SS braided tubing.

10.15.4 Remove the ground wire.

10.15.5 Pack the sample containers firmly into a sample container suitcase or other sample transportation container for shipment. Label the suitcase and complete all necessary documentation as required by the shipping company for transporting compressed hydrogen.

10.15.6 Remove the HQSA from the movable platform (6.3).

11. Report

11.1 The following information is included in the analytical report:

11.1.1 Hydrogen weight sampled,

11.1.2 Initial and final sampling time. The sample duration is the difference of final (end) from initial (start) sampling time,

11.1.3 Average sampled hydrogen flow rate in grams per second, which is the weight of hydrogen sampled in kg, multiplying by 1000 and divided by the sample duration in s,

11.1.4 Hydrogen nozzle pressure, which is read from the regulator inlet gauge (5.1, Fig. 1) during sampling,

11.1.5 Initial regulated pressure, which should be 1000 psi. However, it may be lower, depending upon the hydrogen station,

11.1.6 Pictures of the HQSA during sampling, and

11.1.7 Any additional observations.

12. Keywords

12.1 HQSA; high pressure hydrogen fuel; hydrogen leak; safety; gaseous hydrogen sampling

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(1) D1945 Test Method for Analysis of Natural Gas by Gas Chromatography

(2) D1946 Practice for Analysis of Reformed Gas by Gas Chromatography

(3) D4150 Terminology Relating to Gaseous Fuels

(4) D5287 Practice for Automatic Sampling of Gaseous Fuels

(5) D7634 Test Method for Visualizing Particulate Sizes and Morphology of Particles Contained in Hydrogen Fuel by Microscopy

(6) D7649 Test Method for Determination of Trace Carbon Dioxide, Argon, Nitrogen, Oxygen and Water in Hydrogen Fuel by Jet Pulse Injection and Gas Chromatography/Mass Spectrometer Analysis

(7) D7652 Test Method for Determination of Trace Hydrogen Sulfide, Carbonyl Sulfide, Methyl Mercaptan, Carbon Disulfide and Total Sulfur in Hydrogen Fuel by Gas Chromatography and Sulfur Chemiluminescence Detection

ISO Standards [5]

(8) ISO 14687-2 Hydrogen Fuel- Product Specification- Part 2: Proton Exchange Membrane (PEM) fuel cell applications for road vehicles.

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