Designation: D7650 – 10

StandardTest Method for
Test Method for Sampling of Particulate Matter in High Pressure Hydrogen used as a Gaseous Fuel with an In-Stream Filter[1]

This standard is issued under the fixed designation D7650; 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 test method is primarily for sampling particulates in hydrogen fuel used in hydrogen fuel cell vehicles or gaseous hydrogen powered internal combustion vehicle engines up to pressures of 35 MPa (350 Bars) using an in-stream filter. This test method describes sampling apparatus design, operating procedures, and quality control procedures required to obtain the stated levels of precision and accuracy.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.2.1 The values stated in Bars in 1.1, 7.1 and 10.1.1 are for information only.

1.3 *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]

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

2.2 *SAE Standards:*[3]

SAE J2719 Information Report on the development of a hydrogen quality guideline for fuel cell vehicles.

SAE J2600 Compressed Hydrogen Surface Vehicle Refueling Connection Devices

2.3 *ISO Standard:*

ISO/CD 14687–2 Hydrogen fuel — Product Specification — Part 2: Proton exchange membrane (PEM) fuel cell applications for road vehicles.

3. Terminology

3.1 *Acronyms:*

3.1.1 *FCV*—Hydrogen Fuel Cell Vehicle.

3.1.2 *HQSA*—Hydrogen quality sampling assembly for sampling gaseous hydrogen fuel.

3.1.3 *PEM*—Polymer Electrolyte Membrane also called a Proton Exchange Membrane

3.1.4 *PSA*—Particulate sampling adapter for sampling particulate in hydrogen fuel.

3.1.5 *SAE*—Society of Automotive Engineering

3.2 *Definitions:*

3.2.1 *pinhole*—a small hole generated during sampling of particulate in hydrogen that can be identified by microscope.

3.3 *SAE J2719*—Informational Report on the development of a hydrogen quality guideline for fuel cell vehicles. This report specifies PEM FCV hydrogen fuel quality from the fueling nozzle.

3.4 *SAE J2600 Compressed Hydrogen Surface Vehicle Refueling Connection Devices.*—This document specifies the design requirements for nozzles and receptacles used in high pressure hydrogen applications such as delivery from a fueling station to a FCV

4. Summary of Test Method

4.1 This test method provides a procedure for the sampling of particulate matter contained in hydrogen used as a FCV fuel. It is designed to collect all particulates 0.2 µm or larger contained in a known amount of hydrogen at a station dispenser nozzle in a way that simulates a FCV or a gaseous hydrogen powered internal combustion vehicle engine fueling event. The adapter used for sampling particulates in hydrogen fuel is called a Particulate Sampling Adapter (PSA) and is described in 7. Great care should be taken to avoid contamination and exposure of the PSA, filters, and other equipment with particles sized 10 µm or larger prior to use.

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[1] This test method 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.

5. Significance and Use

5.1 Fuel cells such as proton exchange membrane fuel cells require high purity hydrogen for maximum material performance and lifetime. Collection and measurement of particulate matter 0.2 µm or larger is necessary for assuring a feed gas of sufficient purity to satisfy fuel cell system needs. In addition, internal combustion engines using high pressure hydrogen fuel also require low particulate containing fuel. Specifically, particulate matter has been implicated in the premature failure of pneumatic control components, such as valves within vehicles. This sampling procedure is used to collect and measure samples containing particles 0.2 µm or larger in size as specified in ISO/CD 14687–2, SAE J2719, and other hydrogen fuel quality specifications.

5.2 Although not intended for application to gases other than hydrogen and related fuel cell supply gases, the techniques within this sampling procedure can be applied to other high pressure gaseous samples requiring particulate collection and measurement.

6. Interferences

6.1 Dust and other environmental particulate matter will interfere with the accurate measurement of particulates contained in FCV quality hydrogen; therefore, every measure should be taken to avoid contamination of the apparatus and all equipment, supplies and gases used in these procedures.

7. Apparatus Design

NOTE 1—The use of trade names in this section are not intended as an endorsement for use.

7.1 The PSA is designed for pressures at least up to 6000 psi (420 Bar) with appropriate safety factors built in and is designed for a flow rate of 38 g per second of hydrogen without damage to the filter or leakage from the PSA. The PSA possesses a receptacle as per SAE J2600 which is connected directly to the filter housing. A high pressure needle valve with working pressure at 42 Mpa is attached downstream of the filter holder to stop the hydrogen flow when leak testing the PSA. Downstream of the needle valve, a check valve is attached to prevent the back flow of hydrogen during sampling. In summary, the configuration of the PSA, as shown in Fig. 1, is:

A SAE J2600 compliant Receptacle → Filter Holder with Filter →Needle Valve → Check Valve.

Fig. 1 illustrates a PSA design that has been successfully used to collect particulate samples from 5075 psi (350 Bar) fuel cell quality hydrogen. The PSA should be rated above the operating pressure, and all materials used must be rated for high pressure hydrogen applications at a 1.5 times minimum margin of safety at the maximum operating pressure. The recommended working pressure of the PSA and associated materials is 42 Mpa. Contamination from polytetrafluoroethylene (PTFE) tape, lubrication or other sources must be avoided and the apparatus must be cleaned prior to use using appropriate cleaning techniques for high pressure hydrogen applications. The design of the PSA should include minimizing the distance and surface area between the nozzle and filter to minimize the particulates generated from the surface of this area by fast flow and high pressure hydrogen

7.1.1 *High Pressure Filter Holder*—The high pressure filter holder is a 47 mm, stainless steel housing with maximum inlet pressure 70 Mpa and a polytetrafluoroethylene (PTFE) inner 47mm diameter PTFE-O ring. The filter holder must be equivalent, similar or exceed performance characteristics of the filter holder shown in Fig. 1.

7.1.2 *Filter*—A polytetrafluoroethylene (PTFE) filter that tolerates flow rates of up to 38 g per s without damage and collects particulates with a minimum size of 0.2 µm.

NOTE 2—Hydrogen back flow must be avoided since the backflow of hydrogen can cause pinhole formation or other damage to filters. The design of the apparatus and sampling procedures must prevent fuel backflow, such as implementing the use of a check valve as shown in Fig. 1, Item 6.

7.1.3 *PSA Support*—The mechanical PSA support must be designed to securely hold the PSA and a station nozzle. The nozzle should be held firmly and not move or shake during particulate sampling.

7.1.4 *PSA Design variations*—The design of the PSA downstream of the stainless steel Swagelok[4,5] fitting (union 7 in Fig. 1) will vary with the sampling procedure. The procedure variations include:

(1) Sampling while the hydrogen is venting to atmosphere,

(2) Sampling while fueling a vehicle.

Sampling when fueling into a vehicle tank collects a sample more representative of the particulates seen by vehicles in service since the flow rate is much higher when fueling into a vehicle tank than when venting to atmosphere. The following sections describes the post Swagelok fitting designs in detail.

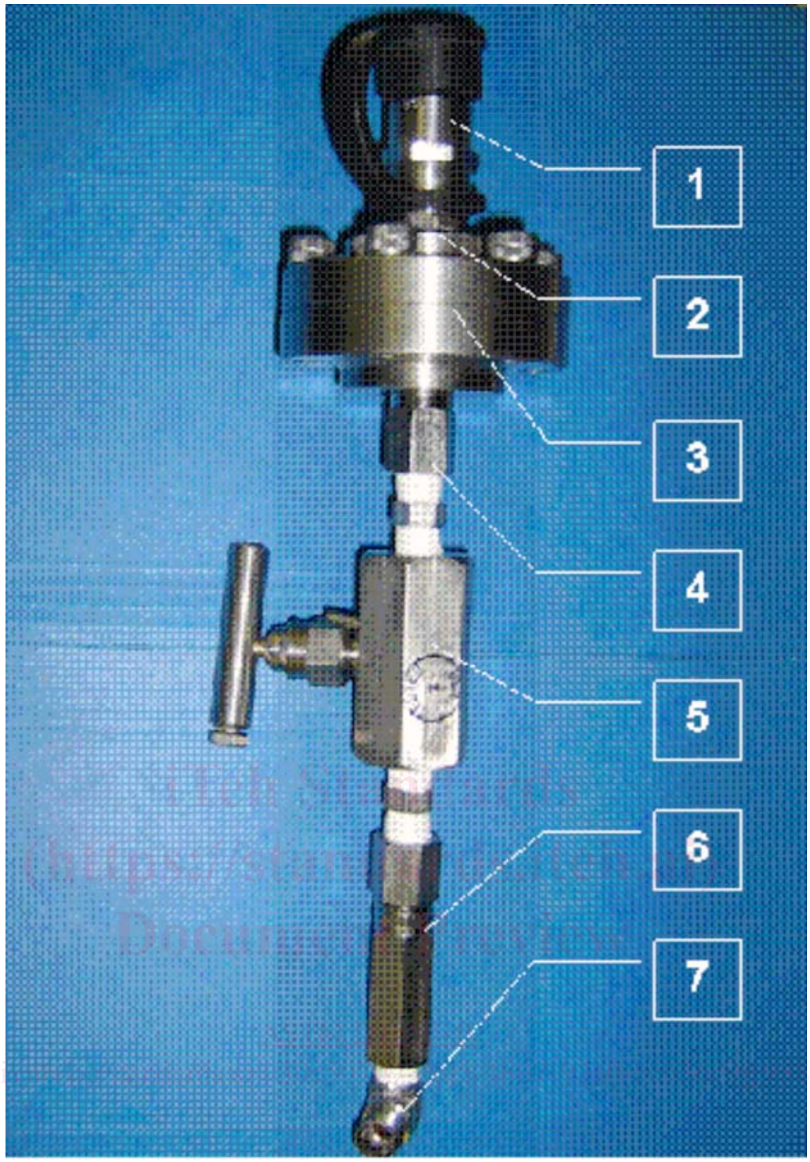
7.1.4.1 *PSA design for venting to atmosphere*—For application to systems requiring venting hydrogen through the PSA to atmosphere, a ventilation assembly contains a 3 m long SS braided tubing connects Item 7 of Fig. 1 in one end and another to a dual 2.4 m long 1.27cm OD SS tubing vertical to the ground. During sampling, the hydrogen fuel flows through PSA, then the ventilation assembly, before venting to air at 8ft above ground.

7.1.4.2 *PSA design for flowing hydrogen to gaseous hydrogen vehicle tank*—The configuration is the same as 7.1.4.1, except the downstream of the PSA at the elbow of the PSA (Item 7, Fig. 1) connects the inlet hose of a 2nd nozzle assembly. The 2nd SAE J2600 nozzle is then attached to the receptacle on a vehicle. While sampling, the hydrogen fuel flows from station SAE J2600 nozzle (1st nozzle) → PSA → inlet hose of a 2nd SAE J2600 nozzle assembly → receptacle of FCV tank.

8. Additional Equipment Needed

8.1 *Glove box*—A glove box is a sealed container that is designed to assemble PSA without particulate contamination from ambient air. Two gloves are built into the sides of the glove box with entry arranged in such a way that the user can

[5] The mention of trade names in this test method does not constitute endorsement or recommendation. Other manufacturers of equipment or equipment models can be used.



Item	Description
1	SAE J2600 hydrogen receptacle without filter
2	Swagelok[4] High pressure SS-6–SAEH-1–4TH (Pressure rating 9,000 psi)
3	Millipore High Pressure 316 SS Filter Housing (47mm, 02.μm) P/N XX4504700
4	3–O Outlet ¼ in. FNPT-SAE 7/16-20 union
5	High pressure needle valve
6	Check valve
7	SS Swagelok Tube fitting, female elbow, ⅜ in. tube OD × ¼ MNPT, P/N SS-600–2–4

FIG. 1 PSA Components

place hands into the gloves to install the filter and assemble the filter holder inside the box. The glove box must be maintained particulate free at all times. Any visual particulate material must be removed prior to working with the PSA or filters. A HEPA[6,5] vacuum can be used to remove particles from the glove bag and other equipment.

8.2 *Moisture/Temperature Data Logger*—A data logger is placed inside the glove box to measure both moisture and temperature continuously at pre-defined intervals such as once every two to five minutes. Moisture in the glove box is kept between 15 to 30% using reagent grade nitrogen. All temperature and moisture data are stored in a data logger, which are

[6] HEPA is a trademark of the HEPA Corporation, 3071 East Coronado Street Anaheim, CA 92806.