



Standard Test Method for Test Method for Sampling of Particulate Matter in High Pressure Hydrogen used as a Gaseous Fuel with an In- Stream Filter¹

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.

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.

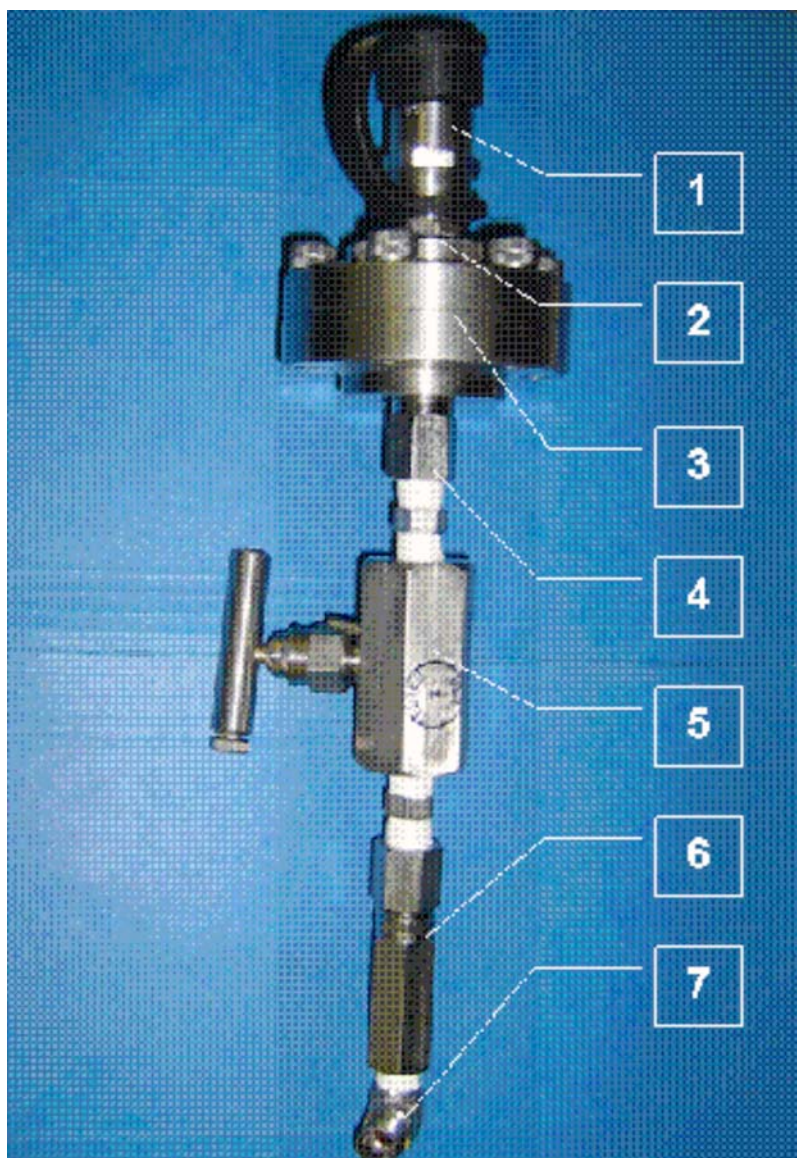
7.1.4.2 *PSA design for flowing hydrogen to gaseous hydrogen vehicle tank*—The configuration is the same as 7.1.4.1, except the downstream of the PSA at the elbow of the PSA (Item 7, [Fig. 1](#)) connects the inlet hose of a 2nd nozzle assembly. The 2nd [SAE J2600](#) nozzle is then attached to the receptacle 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

⁴ Swagelok is a trademark of Swagelok Fluid System Technologies, 29500 Solon Road, Solon OH 44139.

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-1-4 TH (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, ⅝ in. tube OD × ¼ MNPT, P/N SS-600-2-4

FIG. 1 PSA Components

glove box with entry arranged in such a way that the user can 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

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

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 downloaded into a Microsoft Excel^{7,5}, or a similar program, sheet after completion of measurements.

8.3 Mini-Clean Room—A small clean room with HEPA air filtration must be used to store unused polytetrafluoroethylene (PTFE) filters, filter holders, and sampled filters at moisture content less than 30%.

8.4 Ultrasonic Cleaner—Either an ultrasonic bath or probe is used in lab to shake off particulates on parts of PSA into reagent grade water.

8.5 Hydrogen Leak Detector—A hydrogen leak detector is a required safety device needed to detect small hydrogen gas leaks in particular when the PSA is pressurized prior to particulate collection. The diluted soap bubble should not be used to detect hydrogen leak from PSA.

8.6 HEPA Filter Horizontal Flow Hood—A HEPA filter horizontal flow hood blows filtered air through a HEPA filter horizontally, providing for an environment with minimal suspended particulates. The air velocity measured by an air flow meter (8.8) within the hood should be over 100 ft/minute. If below this velocity, the air velocity meter should trigger an alarm notifying the operator about a low air velocity.

8.7 Plastic tweezers—Used to manipulate filters without contamination.

8.8 Air Flow Meter—A meter to measure the air velocity going through the HEPA Filter Horizontal Flow Hood. The air flow meter can trigger alarm when the air flow rate is lower than 30 m/min.

8.9 Clean Room Air Filter Fan—A fan that blows air through a HEPA filter to improve particulate removal efficiency.

8.10 HEPA Vacuum—A vacuum with a HEPA filter that is used to remove dust from a glove box or general filters storage or work environments.

9. Reagents and Materials

9.1 Filters—A 47mm diameter polytetrafluoroethylene (PTFE) filter (PTFE Membrane Disc Filters. For example, a Pall TF-200 47mm 0.2 µm (P/N 66143) with a pore size of 0.2 µm, as described in 7.1.2, has been used.) This type filter has two sides: one is PTFE and the other is polypropylene. Only the PTFE side faces incoming hydrogen fuel and collects particulates in hydrogen. New filters must be demonstrated to be particle free. Filters must be inspected and conditioned before use. Inspection and conditioning must be performed in a temperature and humidity controlled environment free of suspended particulate matter, such as glove box (8.1).

9.2 De-Ionized or Regent Grade Water—Purified water with resistivity 18 megohms-cm at room temperature for ultrasonic cleaner (8.4).

10. Hazards

10.1 High Pressure Hydrogen:

10.1.1 Hydrogen fuel pressure can approach 6000 psi (414 Bar). All PSA components must be constructed from 316 stainless steel, or better, and rated for this application.

10.1.2 The total mass of hydrogen passing through the PSA during a sampling event is approximately 2 kg. Smoking, camera flashes, or mobile phones usage are an ignition hazard and are not allowed within 7.6 m from the both ventilation tubing (7.1.4.1) and hydrogen fueling station itself. Additional safety precautions must be taken as necessary to prevent fire or explosion.

10.2 Static Charges—During particulate sampling, the extremely high speed of hydrogen flow may generate a static charge on PSA components. The static charge is removed by grounding the PSA with a wire from hydrogen fueling station or other available grounding wire(s).

10.3 Hydrogen Embrittlement—High pressure hydrogen can cause embrittlement of contacting metal surfaces or may cause metal hydride formation on metal surfaces. This can lead to catastrophic PSA failure, hydrogen leaks or generation of pyrophoric particulates. The PSA and all equipment used according to this standard must be closely inspected for signs of cracks, metal oxide dust from metal hydride oxidation or any other combination of signs of wear and damage.

11. Sampling, Test Specimens, and Test Units

11.1 Sampling—Sampling of particulate matter in high pressure hydrogen fuel is at the delivery nozzle without using either a regulator or pressure reducing orifice to lower the pressure. The pressure usage range is generally from 14 Mpa to 42Mpa.

11.2 Test Specimens—Particulate matter 0.2 µm or larger.

11.3 Test Units—µg/L or mg/kg.

12. Preparation of Apparatus

12.1 PSA—Before assembly, visually examine the inside of each PSA component to verify it is free from particulates or contamination, and is undamaged inside a glove box (8.1). All parts should be cleaned using an ultrasonic bath such as per 8.4.

13. Conditioning

13.1 Filter Conditioning—Unused and new filters are inspected for pinholes and other damage. After inspection they are stored in a box or other appropriate particulate free container at 15 to 30% humidity and at 15 to 30 °C until ready for weighing.

13.2 Filter Holder Conditioning :

13.2.1 Filter holder components must be stored in either a mini-clean room (8.4) or a glove box (8.1). The inside of the filter holder must be carefully examined inside a glove box (8.1) where any obvious foreign material or dust is removed. If the filter holder is dirty it is cleaned using the procedure in 13.2.2.

13.2.2 Under a HEPA Filter Horizontal Flow Hood (8.6), the following clean-up procedures are performed if the inside of the filter holder is dirty. Both inner and outer o-rings of the high pressure filter holder are removed. The o-rings for both inlet and outlet unions (¼ in. FNPT – SAE 7/16 – 20 unions,

⁷ Microsoft Excel is a trademark of the Microsoft Corporation, One Microsoft Way Redmond, WA 98052-6399.

3-I and 3-O, **Fig. 1**) are also removed. The high pressure filter holder plus the unions are put into a 1000mL beaker. Enough de-ionized water is added to cover all filter holder components. Sonication using either a sonic probe or Ultrasonic Bath (8.4) is performed on the filter holder components for at least 20 min. The water is decanted and the ultrasonic cleaning process is repeated two more times. The filter holder components are dried in a mini-clean room (8.4) for two days at 15 to 30% relative humidity.

14. Procedure

14.1 Filter Installation procedure:

14.1.1 *Inside a glove box*—Using plastic tweezers remove a weighed filter and center the weighed filter onto the screen support affixed to the bottom of the filter holder with the polypropylene side of the filter facing downstream. The PTFE side of the polytetrafluoroethylene (PTFE) filter must face the receptacle. Align the top of the filter holder with bottom of the filter holder and attached the filter holder screws. Tighten the screws a little at a time by tightening one screw then tightening the screw opposite to it. This is necessary to ensure even pressure distribution across the filter and filter holder. Unscrew the screws and separate the top and bottom sections of filter holder. Visually examine the filter to ensure that has made contact with and is centered on the polytetrafluoroethylene (PTFE) o-ring. If it was not centered properly, repeat the centering procedure. When the proper contact and placement is achieved, align the top and bottom sections of the filter holder and tighten all the screws as before.

14.1.2 Remove the PSA from the glove box (8.1) and further tighten the screws on the filter holder by a hex head wrench.

14.2 Particulate Sampling Procedure:

NOTE 3—**Warning:** SAFETY PRECAUTION: During either particulate or gaseous sampling, personnel must wear goggles, safety shoes and a flame resistant Nomex^{8,5} lab coat. Personnel not directly involved in sampling should stand at least fifteen feet away from the sampling event.

14.2.1 Particulate Sampling Procedure - Venting to Atmosphere :

(1) Install the 47mm OD 0.2µm polytetrafluoroethylene (PTFE) filter into the PSA according to 14.1.

(2) In the field, the PSA is secured to a PSA Support (7.1.3) by trained personnel.

(3) Ground the PSA to the grounding wire on Hydrogen Fueling pump or other suitable grounding site.

(4) Attach the fueling nozzle to the receptacle on the PSA by station personnel.

(5) Close the high pressure needle valve (Item 5, **Fig. 1**) and start station hydrogen fueling leak checking procedure to fill the PSA with high pressure hydrogen.

(6) Perform a leak check by use of a hand held leak detector to verify there are no leaks around the nozzle and filter holder (1 to 5, **Fig. 1**) by moving the detector inlet around all connections. If a leak is detected, investigate and fix it before sampling being proceeded.

(7) Prior to opening the high pressure needle valve (Item 5, **Fig. 1**), prepare to begin timing the sample flowing through nozzle as well as record the initial hydrogen tank pressure if necessary.

(8) Open the needle valve (Item 5, **Fig. 1**) to atmosphere and flow at least 2 kg of Hydrogen Fuel through the filter to obtain a representative sample. Write down the sampling time and weight of the hydrogen sample from the station dispense meter.

(9) Close the needle valve (Item 5, **Fig. 1**) and the hydrogen fueling station should automatically stop hydrogen flow. Remove the station nozzle from the PSA.

(10) After the completion of particulate sampling, the PSA is removed from the PSA Support after removal of the ground wire.

14.2.2 Particulate Sampling Procedure —Hydrogen Fuel Pressurized into a FCV using a SAE J2600 Nozzle and Hose:

(1) The general sampling configuration is described in 7.1.4.2, in which the hydrogen:

- flows from the station SAE J2600 nozzle (1st nozzle),
- passes PSA into the inlet hose of 2nd SAE J2600 nozzle onto the receptacle on FCV, and finally
- pressurizes into FCV hydrogen storage tank.

However, every nozzle has venting tube. The vent tube of the 1st nozzle is venting through station vent and the vent tube of the 2nd nozzle is connected to the ventilation assembly described in 7.1.4.1.

(2) Connect the communication cable of the hydrogen station to FCV.

(3) Perform procedures 1 to 6 described in section 14.2.

*Note: Since the air is in the section from the high pressure needle valve (Item 5, **Fig. 1**) to 2nd SAE J2600 nozzle, the air must be removed before the hydrogen can be fueled into FCV. The next five steps are for removing air inside 2nd SAE J2600 nozzle.*

(4) Detach the 2nd SAE J2600 nozzle from the FCV receptacle and attach it to a receptacle, which is neither the one on PSA (Item 1, **Fig. 1**) nor on FCV and called the 3rd receptacle. A high pressure ball valve is attached to the 3rd receptacle in one end and the ventilation system in 7.1.4.1 in the other end.

(5) With the ball valve attached to the 3rd receptacle closed, open the PSA high pressure needle valve (Item 5, **Fig. 1**) to allow hydrogen into the 2nd SAE J2600 nozzle. Use hydrogen leak detector to perform leak check around all the connections from PSA high pressure needle valve (5, **Fig. 1**) to the ball valve attached to the 3rd receptacle. If leak is found, fix it before proceed.

(6) Open the ball valve attached to the 3rd receptacle to release most of hydrogen to atmosphere and, just before the hydrogen completely exhausted, close the ball valve.

(7) Start the station leak check.

(8) Repeat procedures 6 and 7 an additional nine (9) times.

Note: The next four (4) steps describe sampling:

⁸ Nomex is a trademark of DuPont, 1007 Market Street Wilmington, DE 19898

(9) Detach the 2nd **SAE J2600** nozzle from the 3rd receptacle and attach it to the receptacle of FCV hydrogen storage tank by station personnel. Start the station fueling cycle.

(10) Sampling or fueling time is measured and the weight of hydrogen fuel sampled is obtained from FCV fuel meter. At least 2 kg of hydrogen fuel must be sampled. Hydrogen fuel delivery is stopped.

(11) The hydrogen in the 2nd nozzle and PSA is vented by attaching the 2nd nozzle to the 3rd receptacle as in steps 4 and 6.

(12) *Filter preparation for further analyses* – Remove any dust on the surface of PSA, which is then placed inside a glove box. Carefully disassemble the filter holder avoiding shearing action that may damage the filter. Using plastic tweezers

remove the filter from the filter screen and place it on a balance. Record the weight by following Test Method **D7651**. Store the filter in a labeled small particulate-free plastic or other suitable container until ready for further analyses.

15. Report

15.1 Report sampling date and time, hydrogen weight, sampling duration, and additional comments as necessary. Calculate the hydrogen sampling flow rate by dividing the total weight of hydrogen sampled in grams by the total sampling duration in seconds.

16. Keywords

16.1 balance; clean room; glove box; high pressure hazard; hydrogen leak; hydrogen fuel; particulate; safety

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