Designation: D7650 – 13

Standard 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 70 MPa (700 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 *Exception*—In 7.1 and 10.1.1 the values stated in psi 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 Hydrogen Fuel Quality for Fuel Cell Vehicles

SAE J2600 Compressed Hydrogen Surface Vehicle Refueling Connection Devices

2.3 *ISO Standard:*[4]

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

2.4 *EU Standard:*[5]

97/23/EC Pressure Equipment Directive of the EU set out the standards for the design and fabrication of pressure equipment

2.5 *DIN Standard:*[6]

DIN EN 12266-1 Industrial valves-Testing of metallic valves-Part 1: Pressure test, test procedures and acceptance criteria Mandatory Requirements

2.6 *API Standard:*[7]

API 598 Valve Inspection and Testing

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 or Proton Exchange Membrane

3.1.4 *PSA-H70*—Particulate sampling adapter for sampling particulate in hydrogen fuel up to pressures of 70 MPa.

3.1.5 *SAE International*—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 Hydrogen Fuel Quality for Fuel Cell Vehicles*—This document specifies allowable levels of constituents in the hydrogen fuel at the vehicle/station interface.

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.

[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.

[4] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

[5] Available from European Committee for Standardization (CEN), Avenue Marnix 17, B-1000, Brussels, Belgium, http://www.cen.eu.

[6] Available from Deutsches Institut fur Normung e.V.(DIN), Burggrafenstrasse 6, 10787 Berlin, Germany, http://www.din.de.

[7] Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, http://www.api.org.

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 for pressures up to 70 MPa (PSA-H70) and is described in Section 7. Great care should be taken to avoid contamination and exposure of the PSA-H70, filters, and other equipment with particles sized 10 µm or larger prior to use.

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 quality to satisfy fuel cell system needs. In addition, internal combustion engines using high pressure hydrogen fuel also require low particulate levels. 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.

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 according to Section 14 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-H70 has a design pressure of 800 Bar (11600 psi) and is certified after 97/23/EC and API 598 (conform to DIN EN 12266-1) up to 1200 bar (17400 psi) shell test pressures with appropriate safety factors built in. It is designed for a flow rate of 60 g per second of hydrogen without damage to the filter or leakage from the PSA-H70. The PSA-H70 possesses a receptacle as per SAE J2600 followed by a rotary valve which is directly integrated to the filter housing inlet. The rotary valve provides the possibility to switch the inner diameter of the filter housing inlet from 4 mm to 1 mm. That prevents the filter element from high pressure pulse damage. The filter holder contains a standard 47 mm diameter filter element (≥ 0.2 µm pore size) for particle collection, supported by a sinter metal frit with a pore size of 150 µm. Downstream the sinter metal frit a high pressure bleed plug is integrated into the filter holder followed by a standard hydrogen refueling hose and nozzle as per SAE J2600 to be connected to a FCV during sample collection. In summary, the configuration of the PSA-H70, as shown in Fig. 1, is:

A SAE J2600 compliant Receptacle → Filter Holder with integrated rotary valve, the Filter Element supported by a Sinter Metal Frit and a an integrated high pressure Bleed Plug→Refueling Hose→ a SAE J2600 compliant Fueling Nozzle.

Fig. 1 illustrates a PSA-H70 design that has been successfully used to collect particulate samples from 875 bar (12691 psi) fuel cell quality hydrogen. The PSA-H70 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-H70 and associated materials is 80 MPa. Contamination from 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-H70 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 of 120 MPa a polytetrafluoroethylene (PTFE) inner 40.94 mm diameter PTFE-O ring and a hydrogenated acrylnitrilbutadiene caoutchouc as high pressure sealing. The filter holder must be equivalent, similar or exceed performance characteristics of the filter holder described in this test method, that is burst strength.

7.1.2 *Filter*—A polytetrafluoroethylene (PTFE) filter that tolerates flow rates of up to 60 g per s without damage and collects particulates with a minimum size of 0.2 µm.

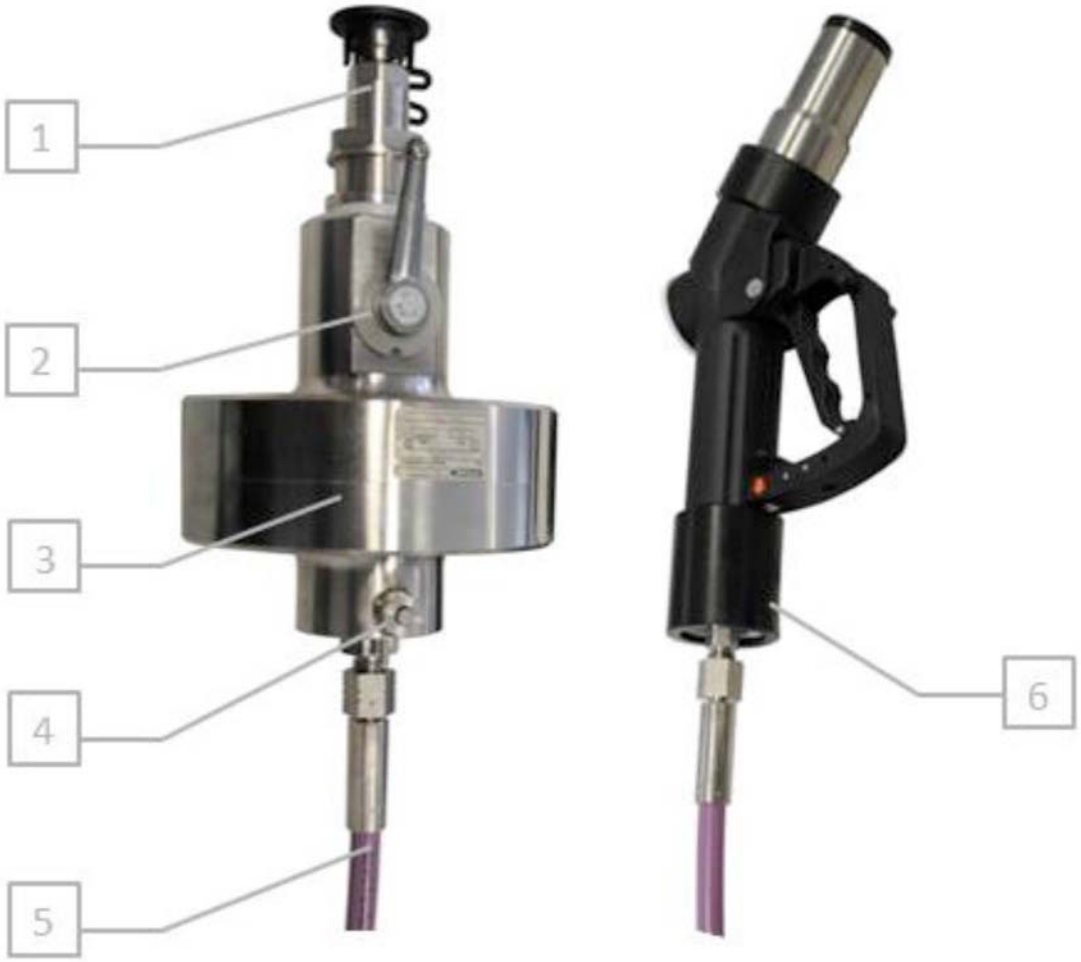
NOTE 2—Hydrogen back flow must be avoided since the backflow of hydrogen can cause pinhole formation or other damage to filters. The design of the apparatus and sampling procedures must prevent fuel backflow.

7.1.3 *PSA-H70 Support*—The mechanical PSA-H70 support must be designed to securely hold the PSA-H70 and a J2600 nozzle. The nozzle should be held firmly and not move or shake during particulate sampling.

7.1.4 *PSA-H70 design for flowing hydrogen to gaseous hydrogen vehicle tank*—As sampling during refueling into a vehicle tank collects a sample most representative of the particulates seen by vehicles in service, the PSA-H70 is designed for this purpose. Therefore, for particulates collection the refueling nozzle of the station is connected to the SAE J2600 receptacle of the PSA-H70 and the SAE J2600 nozzle of the PSA-H70 is attached to the FCV receptacle. While sampling, the hydrogen fuel flows from station SAE J2600 nozzle (1st nozzle) → PSA-H70 → with hose and a 2nd SAE J2600 nozzle → 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-H70 without particulate contamination from ambient air. Two gloves are built into the sides of the



Item	Description
1	SAE J2600 hydrogen receptacle without filter
2	Rotary valve DN04 (1 mm and 4 mm diameter)
3	Hydac PSA-H70 DN04 PN800 SO 1257 Filter Housing
4	Bleed plug
5	High pressure hydrogen fueling hose
6	SAE 2600 hydrogen fueling nozzle

FIG. 1 PSA-H70 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-H70 or filters. A HEPA[8,9] vacuum can be used to remove particles from the glove bag and other equipment.

8.2 *Relative 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 downloaded into a Microsoft Excel[10,9], 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-H70 into reagent grade water.

8.5 *Hydrogen Leak Detector*—A hydrogen leak detector is a required safety device needed to detect small hydrogen gas

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

[9] 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.

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

leaks in particular when the PSA-H70 is pressurized prior to particulate collection. The diluted soap bubble should not be used to detect hydrogen leak from PSA-H70.

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 30 m/min. 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 875 bar (12691 psi). All PSA-H70 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-H70 during a sampling depends on how many FCVs are refueled over one filter element. Smoking, camera flashes, or mobile phones usage are an ignition hazard and are not allowed within 7.6 m 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-H70 components. The static charge is removed by grounding the PSA-H70 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-H70 failure, hydrogen leaks or generation of pyrophoric particulates. The PSA-H70 and all equipment used according to this test method 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 by default without using either a regulator or pressure reducing orifice to lower the pressure. In case of very high pressure pulses up to 875 Bar at the beginning of the refueling which may in rare cases damage the sinter metal frit, the PSA-H70 provides the option to reduce the inner diameter from 4 mm to 1 mm. The pressure usage range is generally from 2 MPa to 87.5 MPa.

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-H70*—Before assembly, visually examine the inside of each PSA-H70 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 cleaned 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) or in a mini-clean room (8.4) 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 or through a professional cleaning service in laboratory.

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 high pressure filter holder plus the unions are put into a 1000 mL 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-H70 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[11,9] lab coat. Personnel not directly involved in sampling should stand at least fifteen feet away from the sampling event.

(1) Install the 47mm OD 0.2µm polytetrafluoroethylene (PTFE) filter into the PSA-H70 according to 14.1.

(2) Assemble the PSA-H70 refueling hose with SAE J2600 nozzle to the PSA-H70 filter holder

(3) Close the PSA-H70 bleed plug.

(4) In the field, the PSA-H70 is secured to a PSA-H70 Support (7.1.3) by trained personnel.

(5) Ground the PSA-H70 to the grounding wire on Hydrogen Fueling pump or other suitable grounding site.

(6) Attach the station fueling nozzle to the receptacle on the PSA-H70.

(7) Change the rotary valve to diameter 1 mm and start station hydrogen refueling to fill the PSA-H70 volume with high pressure hydrogen. The refueling stations stops the fill automatically.

(8) 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 or by spraying the leak check liquid onto all connections. If a leak is detected, investigate and fix it before sampling being proceeded.

(9) Slowly open the bleed plug to release the hydrogen with air fraction from the PSA-H70. Leave the bleed plug open.

(10) Attach the PSA-H70 SAE J2600 nozzle to the FCV receptacle and then close the bleed plug at the PSA-H70.

(11) Decouple station refueling nozzle from PSA-H70, hang it up at the station dispenser and then re-connect the station nozzle to the SAE J2600 receptacle of the PSA-H70. This procedure is necessary as the station needs to start a new refueling procedure.

(12) Start station hydrogen refueling and change the rotary valve from 1 mm to the 4 mm position right after the pressure pulse. The handle is now in the direction of the flowing hydrogen and the station will refuel the FCV over the PSA-H70 until the maximum tank system pressure is reached and the refueling is automatically stopped by the station. Refuel at least 2 kg of hydrogen through the filter to obtain a representative sample. Write down the sampling time and weight of the hydrogen sample from either the station dispenser meter and from the FCV fuel meter, or both.

(13) Remove the station nozzle from the PSA-H70 and put the dust cover onto the PSA-H70 SAE J2600 receptacle to avoid receptacle contamination with condensing water due to the cold PSA-H70 after refueling.

(14) Slowly open the bleed plug of the PSA-H70 to depressurize the device. Then remove PSA-H70 SAE J2600 nozzle from FCV receptacle. To refuel more than one FCV over one filter element, which increases the representativeness of the sample, repeat steps (10) to (14).

(15) After the completion of particulate sampling remove the ground wire, remove the PSA-H70 from the PSA-H70 support close the bleed plug and dissemble the PSA-H70 into filter holder unit and fueling hose plus SAE J2600 nozzle. Be careful not to tip or rotate the filter holder to avoid particulate distribution.

If a mobile glove box is available at the sampling location perform step (16) otherwise proceed with step (17) (see also D7651 Test Method for Gravimetric Measurement of Particulate Concentration of Hydrogen Fuel).

(16) Filter preparation for further analysis: Remove any dust on the surface of PSA-H70, 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. Store the filter in a labeled small particulate-free plastic or other suitable container until ready for further analyses.

(17) Put the filter holder unit in the transportation case for transportation to the laboratory.

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

[11] Nomex is a trademark of DuPont, 1007 Market Street Wilmington, DE 19898