



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

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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

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>.

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.

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

⁶ Available from Deutsches Institut für 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 ($\geq 0.2 \mu\text{m}$ 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 $\rightarrow$ Filter Holder with integrated rotary valve, the Filter Element supported by a Sinter Metal Frit and a an integrated high pressure Bleed Plug $\rightarrow$ Refueling Hose $\rightarrow$ 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) $\rightarrow$ PSA-H70 $\rightarrow$ with hose and a 2nd SAE J2600 nozzle $\rightarrow$ 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