Designation: D7651 – 17

Standard Test Method for Gravimetric Measurement of Particulate Concentration of Hydrogen Fuel[1]

This standard is issued under the fixed designation D7651; 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 intended for gravimetric determination of particulate concentration in hydrogen intended as a fuel for fuel cell or internal combustion engine powered vehicles. This test method describes operating and quality control procedures required to obtain data of known quality satisfying the requirements of SAE J2719. This test method can be applied to other gaseous samples requiring determination of particulates provided the user's data quality objectives are satisfied.

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

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4150 Terminology Relating to Gaseous Fuels

D7650 Practice for Sampling of Particulate Matter in High Pressure Gaseous Fuels with an In-Stream Filter

E617 Specification for Laboratory Weights and Precision Mass Standards

2.2 *SAE Standards:*[3]

SAE J2600 Compressed Hydrogen Surface Vehicle Refueling Connection Devices

SAE J2719 Hydrogen Quality Guideline for Fuel Cell Vehicles

2.3 *Other Standards:*

Code of Federal Regulations, Title 40, Part 50, Appendix L, Section 8.2[4]

ISO 14687–2 Hydrogen fuel -- Product specification -- Part 2: Proton exchange membrane (PEM) fuel cell applications for road vehicles[5]

3. Terminology

3.1 *Acronyms:*

3.1.1 *FCV*—Hydrogen Fuel Cell Vehicle

3.1.2 *HEPA*—High Efficiency Particulate Air

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

3.1.4 *PEMFC*—Proton Exchange Membrane Fuel Cell

3.1.5 *PTFE*—Polytetrafluoroethylene

3.1.6 *SAE*—SAE International

3.2 *Definitions:* For definitions of general gaseous fuel terms used in this practice, refer to D4150.

3.2.1 *weight monitoring filter, WMF*—This filter is put inside the glove box or clean room (7.3) with the balance (7.1) and never removed. The weight of this filter is always measured before and after each measurement event.

4. Summary of Test Method

4.1 This test method is used to determine the concentration of particulate matter and nonvolatile reside on filters collected from hydrogen fuel or other gaseous streams at fueling station dispenser nozzles (Test Method D7650, SAE J2600) or other

[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 Office of the Federal Register, 800 N. Capitol St., NW Suite 700 Washington, DC 20001.

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

gaseous fuel delivery system dispenser interfaces. The particulate concentration is determined by dividing the particulate weight, which is the difference of filter weights before and after sampling, by the total volume of hydrogen or other gaseous fuels passing through the filter. Every precaution should be taken to avoid contamination of particulates onto the filter coming from the particulate sampling adapter, the analytical system, ambient air, filter handling, or other environmental sources.

5. Significance and Use

5.1 Low operating temperature fuel cells such as proton exchange membrane fuel cells (PEMFCs) require high purity hydrogen for maximum material performance and lifetime. Measurement of particulates in hydrogen is necessary for assuring a feed gas of sufficient purity to satisfy fuel cell and internal combustion system needs as defined in SAE J2719. The particulates in hydrogen fuel for fuel cell vehicles (FCV) and gaseous hydrogen powered internal combustion engine vehicles may adversely affect pneumatic control components, such as valves, or other critical system components. Therefore, the concentration of particulates in the hydrogen fuel should be limited as specified by ISO 14687-2, SAE J2719, or other hydrogen fuel quality specifications.

5.2 Although not intended for application to gases other than hydrogen fuel, techniques within this test method can be applied to gas samples requiring determination of particulate concentration.

6. Interferences

6.1 Particulate matter on the filter from sources other than the hydrogen fuel will interfere with the determination of particulate concentration. Every precaution should be taken to avoid contamination of particulates onto the filter from the particulate sampling adapter, the analytical system, ambient air, filter handling, or other environmental sources.

6.2 To minimize contamination on the filters from body oils and moisture, wear powder-free gloves while handling filters.

6.3 Humidity may affect polytetrafluoroethylene (PTFE) filter weight. Filters should be equilibrated for a minimum of 24 hours (h) in a controlled environment prior to weighing. For reference, U.S. EPA filter conditioning requirements for PM10 samples are a temperature range of 21 ± 2 °C and a humidity range of 35 ± 5% relative humidity (RH).

7. Apparatus

7.1 *Balance*—The balance must have a readability of 0.01 milligrams (mg). The balance may have the capability to record the weight and calibration data into Microsoft Excel,[6,7] or a similar program. In order to prevent contamination of particulates from ambient air, the balance must be placed inside a glove box or clean room with a HEPA[8,7] air filter.

7.2 *Calibration Weight–ASTM Class 1 (E617)*—Category calibration weights with a tolerance of ± 0.01 mg certified as traceable to a national metrology institute (NMI) such as NIST mass standards should be used. The weights used for calibration are 0.05 g and 0.2 g, of corrosion-resistant construction. Calibration weights are to be certified on an annual basis.

7.3 *Glove Box (Option A)*—A glove box is a sealed chamber that, in this application, allows weight measurements to be taken without particulate contamination from ambient air. Two gloves are built into the front side of the glove box so that the user can place their hands into the gloves and perform weight measurements, install filters, and assemble the filter holders. A side evacuation port or antechamber should also be used to minimize contamination of the glove box environment. The glove box must be kept clean at all times and any visual particulate matter must be removed immediately. The glove box should have a steady flow of clean, dry nitrogen (N_2) at all times. The temperature and humidity should be kept consistent at 21 ± 2 °C and 35 ± 5% RH and should be monitored by a data logger or other device installed in the glove box.

7.4 *Clean Room (Option B)*—Analysis should occur in a climate-controlled, draft-free room constantly under positive pressure. The relative humidity must be maintained at 35 ± 5% and the temperature must be maintained at 21 ± 2 °C. If the temperature or humidity falls out of range, no weighing can occur for 24 h. Before entering the clean room, the analyst must step on "sticky" floor mats to remove any particulate matter from the bottoms of shoes. The room must have a HEPA air filter on the inlet air system to remove particulates from the air.

7.5 *Static Charge Removal Device*—A static charge removal device, such as an ionization bar, must be placed inside the glove box or clean room (7.3) next to the balance. The static charge on the materials, such as weights and filters, must be removed before weighing. Alternatively, anti-static strips which consist of radioactive (α - particle) Polonium-210 can be used to discharge static from weights and filters. Polonium strips should be replaced every 6 months (conservatively) or according to the useful life quoted by the manufacturer.

7.6 *Humidity/Temperature Data Logger*—A data logger is placed inside the glove box or clean room to measure both humidity and temperature of the atmosphere either continuously or at pre-defined intervals. The humidity is kept at 35 ± 5% RH. Temperature and humidity information may be stored in a data logger, which can be downloaded into Microsoft Excel,[6,7] or a similar program after completion of measurements.

7.7 *Storage*—A clean room or a glove box with a HEPA filter may be used to store new PTFE filters, the filter holder, and sampled filters at 35 ± 5% RH and 21 ± 2 °C.

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

[7] 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.

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