Designation: ~~D7651 – 10~~ 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 ~~particulates~~ 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 and the California Code of Regulations, Title 4, Division 9, Chapter 6, Article 8, Sections 4180 – 4181. The levels of precision and accuracy stated.~~ 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 and health 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 Test Method for Sampling of Particulate Matter in High Pressure Hydrogen used as a Gaseous Fuel with an In-Stream Filter

E617 Specification for Laboratory Weights and Precision Mass Standards

2.2 *SAE Standards:*[3]

~~SAE J2719 Hydrogen Quality Guideline for Fuel Cell Vehicles, April 2008~~

SAE J2600 Compressed Hydrogen Surface Vehicle Refueling Connection Devices

SAE J2719 Hydrogen Quality Guideline for Fuel Cell Vehicles

2.3 *Other Standards:*

~~California~~ Code of ~~Regulations~~Federal Regulations, ~~, Title 4, Division 9, Chapter 6, Article 8, Sections 4180 – 4181~~Title 40, Part 50, Appendix L, Section 8.2[4]

ISO ~~14687~~14687–2 Hydrogen fuel -- Product specification -- Part ~~1: All applications except proton~~ 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

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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.

[4] Available from Office of ~~Administrative Law 300 Capitol Mall, Suite 1250 Sacramento, CA 95814-4339~~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.

3.1.2 *PSA—HEPA*—Particulate sampling adapter for sampling particulate in hydrogen fuel. 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*—Society of Automotive EngineeringSAE International

3.2 *Definitions:*

3.2.1 *contaminant*—impurity that adversely affects the components within the fuel cell system or the hydrogen storage system

3.2.2 *density*—Mass per unit of volume of the fuel gas or air being considered.

3.2.3 *fuel cell hydrogen*—hydrogen satisfying the specifications in SAE J2719.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 not removed from the glove box (never removed. 7.3). The weight of this filter is always measured before and after each measurement event.

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 procedure is for the weight determination of filters before and after collection of particulates contained withintest 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 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 fuelfuels passing through athe filter. Every precaution should be taken to avoid contamination of particulates 10 µm or larger onto the filter coming from the PSA, 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 FCVs 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 14687,14687-2, 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 matter.concentration.

6. Interferences

6.1 Particulate matter 10 µm or larger originating in the environment or equipmenton the filter from sources other than the hydrogen fuel will interfere with the determination of total particulate matter collected on the filter. particulate concentration. Every precaution should be taken to avoid contamination of particulates 10 µm or larger onto the filter coming from the PSA, particulate sampling adapter, the analytical system, ambient air, filter handling, or other environmental sources.

6.2 To minimize contamination ofon the filters from body moistureoils and oils,moisture, wear powder-free gloves while handling filters outside of the glove box.filters.

6.3 Moisture content Humidity may affect polytetrafluoroethylene (PTFE) filter weight, even though the polytetrafluoroethylene (PTFE) filter is hydrophobic. weight. Filters should be equilibrated for a minimum of 24 h 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 25 °C (± 3 °C) 21 ± 2 °C and a humidity range of 20 to 30% RH (± 5% RH).35 ± 5% relative humidity (RH).

7. Apparatus

7.1 *Balance*—The balance must ~~measure to 10-5 g. The balance should~~have a readability of 0.01 milligrams (mg). The balance may have the capability to ~~download~~record the weight ~~measurement~~ and calibration data into Microsoft Excel,[6,7]; or a similar ~~program, for weight recording and calibration.~~ program. In order to prevent contamination of particulates from ambient air, the balance must be placed inside a glove box ~~in a small confined~~ or clean room with a HEPA[8,7] air ~~cleaner.~~filter.

7.2 *Calibration ~~weight–Class~~ Weight–ASTM Class 1 ~~(Class (E617S) —)—~~*~~category~~Category calibration weights with a tolerance of ± ~~0.1 mg.~~0.01 mg certified as traceable to ~~NIST mass standards.~~ a national metrology institute (NMI) such as NIST mass standards should be used. The weights used for calibration are ~~a~~ 0.05 g and 0.2 ~~g weight,~~ g, of corrosion-resistant construction. Calibration ~~weight is~~weights are to be certified on an annual basis. ~~The weight of the particular 0.2µm polytetrafluoroethylene (PTFE) filter used in this method test was around 0.1g.~~

7.3 *Glove ~~box—~~Box (Option A)*—A glove box is a sealed ~~container~~chamber that, in this application, ~~is designed to allow weight measurement by balance~~ allows weight measurements to be taken without particulate contamination from ambient air. Two gloves are ~~generally~~ built into the front ~~sides~~side of the glove box ~~with entry arranged in such a way~~ so that the user can place their hands into the gloves and perform weight ~~measurement,~~measurements, install filters, and assemble the filter ~~holder inside the box.~~ holders. A side evacuation port or antechamber should also be used to minimize contamination of the glove box environment. The glove box must be ~~maintained~~kept clean at all times and any visual particulate matter must be removed immediately. ~~A HEPA vacuum can be used for cleaning purposes. A side evacuation port or anti-chamber should also be used to minimize contaminating the glove box environment.~~ The glove box should ~~be flushed~~ have a steady flow of clean, dry nitrogen (N_2~~at all times with clean dry N2 maintaining a RH of 30% or less inside the glove box as determined~~) 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. ~~Before measurement of any material,~~ The static charge on the materials, such as ~~standard~~ weights and filters, ~~the static charges on the material~~ must be removed ~~using a static charge removal device.~~ before weighing. Alternatively, anti-static strips which consist of radioactive (α - particle) Polonium-210 ~~strips~~ 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 *~~Moisture/Temperature~~Humidity/Temperature Data Logger*—A data logger is placed inside the glove box or clean room to measure both ~~moisture~~humidity and temperature of ~~atmosphere inside the glove box~~ the atmosphere either continuously or at pre-defined intervals. The ~~moisture of the glove box is kept of 30% or less using reagent grade or better nitrogen flow. All the temperature and moisture information are~~ humidity is kept at 35 ± 5% RH. Temperature and humidity information may be stored in a data logger, which ~~is~~ can be downloaded into ~~excel,~~ Microsoft Excel,[6,7] or a similar program after completion of measurements.

7.7 *~~Mini-Clean Room—~~Storage*—A ~~small~~ clean room ~~with~~ or a glove box with a HEPA filter ~~must~~may be used to store new ~~polytetrafluoroethylene (PTFE)~~ PTFE filters, the filter holder, and sampled filters at ~~moisture less than 30%.~~ 35 ± 5% RH and 21 ± 2 °C.

7.8 *HEPA Vacuum*—A vacuum with a HEPA filter ~~is~~ may be used to remove dust from the glove box or areas where filters are stored or ~~manipulated.~~handled.

7.9 *Tweezers*—Filters must be handled using clean plastic or PTFE coated tweezers.

7.10 *Gloves*—Clean, non-powdered plastic gloves must be worn when handling filters outside of the glove box.

7.11 *Light ~~box~~Box (Optional)*—A light box may be useful for inspection of filters.

8. Reagents and Materials

8.1 *Filter*—A 47 ~~mm diameter polytetrafluoroethylene (PTFE)~~ millimeter (mm) diameter PTFE filter (PTFE Membrane Disc Filters) is used. One side of this ~~type~~ filter is composed of ~~polytetrafluoroethylene (PTFE)~~ PTFE and the reverse side is composed of polypropylene. ~~Installed~~ When the filter is installed in the filter holder, the PTFE side should face the hydrogen fuel stream. The

[6] Microsoft Excel is a trademark of the Microsoft Corporation, One Microsoft Way Redmond, WA ~~98052-639~~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 ~~9280~~92806.