Designation: D7651 – 10

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 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. 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D7650 Test Method for Test Method for Sampling of Particulate Matter in High Pressure Hydrogen used as a Gaseous Fuel with an In-Stream Filter

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

2.3 *Other Standards:*

California Code of Regulations , Title 4, Division 9, Chapter 6, Article 8, Sections 4180 – 4181[4]

ISO 14687 Hydrogen fuel -- Product specification -- Part 1: All applications except proton exchange membrane (PEM) fuel cell for road vehicles[5]

3. Terminology

3.1 *Acronyms:*

3.1.1 *FCV*—Hydrogen Fuel Cell Vehicle

3.1.2 *PSA*—Particulate sampling adapter for sampling particulate in hydrogen fuel.

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

3.1.4 *SAE*—Society of Automotive Engineering

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.

3.2.4 *weight monitoring filter, WMF*—This filter is put inside the glove box (7.3) with balance (7.1) and not removed from the glove box (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.

[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

[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.

3.4 *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 within 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 fuel passing through a filter. Every precaution should be taken to avoid contamination of particulates 10 µm or larger onto the filter coming from the PSA, 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 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, 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.

6. Interferences

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

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

6.3 Moisture content may affect polytetrafluoroethylene (PTFE) filter weight, even though the polytetrafluoroethylene (PTFE) filter is hydrophobic. Filters should be equilibrated for a minimum of 24 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) and a humidity range of 20 to 30% RH (± 5% RH).

7. Apparatus

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

7.2 *Calibration weight–Class 1 (Class S)*—category calibration weights with a tolerance of ± 0.1 mg. certified as traceable to NIST mass standards. The weights used for calibration are a 0.05 g and 0.2 g weight, of corrosion-resistant construction. Calibration weight is 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*—A glove box is a sealed container that, in this application, is designed to allow weight measurement by balance without particulate contamination from ambient air. Two gloves are generally built into the front sides of the glove box with entry arranged in such a way that the user can place their hands into the gloves and perform weight measurement, install filters and assemble the filter holder inside the box. The glove box must be maintained 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 at all times with clean dry N2 maintaining a RH of 30% or less inside the glove box as determined by a data logger or other device installed in the glove box.

7.4 *Static Charge Removal Device*—A static charge removal device, such as an ionization bar, must be placed inside the glove box (7.3) next to the balance. Before measurement of any material, such as standard weights and filters, the static charges on the material must be removed using a static charge removal device. 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.

7.5 *Moisture/Temperature Data Logger*—A data logger is placed inside the glove box to measure both moisture and temperature of atmosphere inside the glove box 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 stored in a data logger, which is downloaded into excel, or a similar program after completion of measurements.

[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