



# Standard Test Method for Gravimetric Measurement of Particulate Concentration of Hydrogen Fuel<sup>1</sup>

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 ( $\epsilon$ ) 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:<sup>2</sup>

[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:<sup>3</sup>

[SAE J2719 Hydrogen Quality Guideline for Fuel Cell Vehicles, April 2008](#)

[SAE J2600 Compressed Hydrogen Surface Vehicle Refueling Connection Devices](#)

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, <http://www.sae.org>.

### 2.3 Other Standards:

[California Code of Regulations, Title 4, Division 9, Chapter 6, Article 8, Sections 4180 – 4181<sup>4</sup>](#)

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

## 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.

<sup>4</sup> Available from Office of Administrative Law 300 Capitol Mall, Suite 1250 Sacramento, CA 95814-4339

<sup>5</sup> 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  $\mu\text{m}$  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  $\mu\text{m}$  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  $\mu\text{m}$  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  $^{\circ}\text{C}$  ( $\pm 3$   $^{\circ}\text{C}$ ) and a humidity range of 20 to 30% RH ( $\pm 5$  % 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<sup>6,7</sup>, 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<sup>8,7</sup> air cleaner.

7.2 *Calibration weight—Class 1 (Class S)*—category calibration weights with a tolerance of  $\pm 0.1$  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 0.2 $\mu\text{m}$  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 N<sub>2</sub> 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 ( $\alpha$  - 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.

<sup>6</sup> Microsoft Excel is a trademark of the Microsoft Corporation, One Microsoft Way Redmond, WA 98052-6399

<sup>7</sup> 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.

<sup>8</sup> HEPA is a trademark of the HEPA Corporation, 3071 East Coronado Street Anaheim, CA 92806

7.6 *Mini-Clean Room*—A small clean room with HEPA filter must be used to store new polytetrafluoroethylene (PTFE) filters, the filter holder, and sampled filters at moisture less than 30%.

7.7 *HEPA Vacuum*—A vacuum with HEPA filter is used to remove dust from the glove box or areas where filters are stored or manipulated.

7.8 *Light 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) filter (PTFE Membrane Disc Filters) is used. One side of this type filter is composed of polytetrafluoroethylene (PTFE) and the reverse side is composed of polypropylene. Installed in the filter holder, the PTFE side should face the hydrogen fuel stream. The polypropylene side of the filter is generally shinier than the PTFE side, which is dull when viewed under a bright light. When examining, handling, and weighing filters, the side facing the gas stream and collecting particulates must always face up. Before usage of any new filter, examine it carefully inside a glove box to ensure the filter is not damaged and that there are no particulates on the filter. Filters containing particulates should be discarded as they are unsuitable for usage.

## 9. Sampling, Test Specimens, and Test Units

9.1 *Test Specimens*—Particulates.

9.2 *Test Units*— $\mu\text{g/L}$ ,  $\text{mg/kg}$ .

## 10. Preparation of Balance

10.1 *Balance*—Balance must be put in a glove box described in 7.3 and remain there throughout the procedures described in this standard.

## 11. Calibration and Standardization

11.1 *Balance Calibration*—The balance is calibrated at the beginning of a weighing session using a 0.05 and 0.2 g standard weights. A filter blank is also weighed. In addition the balance is internally calibrated with internal standard weights automatically at pre-determined intervals.

11.2 QC weighing of the first filter in a batch of ten filters is recommended.

## 12. Conditioning

12.1 *Filter Conditioning*—New filters are stored in their original packaging and are considered ready for use when stored in mini-clean room as described in 7.6.

## 13. Procedure

13.1 Filter Weighing Procedure

13.1.1 Transfer new polytetrafluoroethylene (PTFE) filter in a box from a mini clean room (7.6) to a glove box (7.3) containing a balance inside. Turn on the nitrogen flow at the flow rate of 0.5 L per minute for about an h to lower moisture content inside the glove box below 30%. Keep nitrogen flowing throughout the measurement process.

13.1.2 The analyst must wear gloves when handling filters and filter holders. Clean non-powdered plastic gloves with plastic sleeves are used for activity outside the glove box. polytetrafluoroethylene (PTFE) filters must be handled using clean plastic or polytetrafluoroethylene (PTFE) coated tweezers.

13.1.3 *Inspect the filters:*

13.1.3.1 Filters must not be contaminated with particulates as seen by eye under a light box or under a microscope.

13.1.3.2 Filter must not be torn or nicked. If damaged discard filter.

13.1.4 Set up the balance so that the output of balance can directly download to Excel or a similar data logging system. Perform a calibration so that all the calibration data is downloaded.

13.1.4.1 Set up the balance so that the weight number of each measurement can be automatically downloaded every two seconds.

13.1.5 Since the weight for each measurement can shift back and forth at five places after decimal point (10-5 g), a rule must be set up to determine which weight number should be selected. The rule suggested is that the first value, that persists for at least 12 s without change will be selected.

13.1.6 The typical sequence for measurement is as following:

13.1.6.1 *Internal Calibration:*

13.1.6.2 200 mg,

13.1.6.3 50 mg,

13.1.6.4 WMF,

13.1.6.5 Filter#1,

13.1.6.6 Filter#2,

13.1.6.7 ...,

13.1.6.8 Repeat weighing all the Filters in the above sequence nine additional times,

13.1.6.9 WMF.

13.1.7 The average weight of the ten measurements of each filter is the weight of the filter for particulate concentration calculation. The percent relative standard deviation of the ten weights for each filter should be less than 0.02%.

13.1.8 The reason to monitoring weight of WMF is to make sure the environment of the glove box (7.3) does not affect weight of the polytetrafluoroethylene (PTFE) filter used for particulate monitoring in hydrogen fuel.

## 14. Calculation or Interpretation of Results

14.1 The polytetrafluoroethylene (PTFE) filter before and after sampling is weighed according to 13.1. The difference of the weight in gram multiplied by 1000 divided by hydrogen fuel sampled in kg is the particulate concentration in mg per Kg of hydrogen.

$$\text{Particulate Concentration} \left( \frac{\text{mg}}{\text{Kg}} \right) = \quad (1)$$

$$\frac{\text{Difference in Weight (g) of Filter before and after Sampling} * 100}{\text{Hydrogen fuel Sampled in Kg}}$$

14.2 The hydrogen in kg multiplied by 1000 and then divided by 2 gives the moles of hydrogen. The volume of a mole of hydrogen is 24.4 L at 25 °C. Therefore, the total

volume of hydrogen is hydrogen in kg \* 1000/2 \* 24.4 L. The weight of particulate in gram multiplied by 1 000 000 divided by total volume of hydrogen gives the particulate concentration in µg/L.

$$\text{Particulate Concentration} \left( \mu \frac{\text{g}}{\text{L}} \right) = \quad (2)$$

$$\frac{\text{Difference in Weight (g) of Filter before and after Sampling}}{\text{Hydrogen fuel Sampled in Kg} * \frac{1000}{2} * 24.4 \text{ L}}$$

## 15. Report

15.1 Report concentration of particulate calculated in 14 in both µg/L and mg/kg.

## 16. Precision and Bias

NOTE 1—Statements of precision and bias for this method will be provided as a result of inter-laboratory testing which will be performed within five (5) years.

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16.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials.

16.2 *Repeatability*—1% full scale for successive identical samples.

16.3 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test materials.

16.4 Reproducibility data to added within five (5) years of method approval.

16.5 *Bias*—A statement of bias will be developed through inter-laboratory testing by the responsible study group.

## 17. Keywords

17.1 gravimetric; hydrogen fuel; particulate concentration; particulate