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.

Designation: D7634 – 10 (Reapproved 2017)

Standard Test Method for Visualizing Particulate Sizes and Morphology of Particles Contained in Hydrogen Fuel by Microscopy[1]

This standard is issued under the fixed designation D7634; 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 visualizing and measuring the sizes and morphology of particulates in hydrogen used as a fuel for fuel cell or internal combustion engine powered vehicles. This test method describes procedures required to obtain size and morphology data of known quality. This test method can be applied to other gaseous samples requiring determination of particulate sizes and morphology provided the user's data quality objectives are satisfied.

1.2 Mention of trade names in standard does not constitute endorsement or recommendation. Other manufacturers of equipment, software or equipment models can be used.

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

1.4 *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.*

1.5 *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]

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

2.2 *SAE Standards:*[3]

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

SAE J6000 Compressed Hydrogen Surface Vehicle Refueling Connection Devices

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *constituent*—component (or compound) found within a hydrogen fuel mixture

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

3.1.3 *fuel cell grade hydrogen*—hydrogen satisfying the specifications in SAE TIR J2719.

3.1.4 *gaseous fuel*—material to be tested, as sampled, without change of composition by drying or otherwise.

3.1.5 *HEPA Filter*—A high efficiency particulate air filter which, by definition, removes at least 99.97% of airborne particles 0.3µm in diameter.

3.1.6 *SAE TIR J2719*—Information Report on the development of a hydrogen quality guideline for fuel cell vehicles

3.2 *Acronyms:*

3.2.1 *FCV*—Fuel Cell Vehicle

3.2.2 *PSA*—Particulate sampling adapter for sampling particulate in hydrogen fuel

3.2.3 *HQSA*—Hydrogen quality sampling adapter for sampling gaseous hydrogen fuel

3.2.4 *SAE*—Society of Automotive Engineers International

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

3.2.6 *PEMFC*—proton exchange membrane fuel cells

4. Summary of Test Method

4.1 This procedure is for visualizing and measuring, by microscopy, the sizes and morphology of particulates after

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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.

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collection of particulates contained within hydrogen fuel at fueling station dispenser nozzles (Test Method D7650, SAE J2600) or other gaseous fuel delivery system dispenser interfaces. Every precaution should be taken to avoid contamination of particulates 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 temperature fuel cells such as proton exchange membrane fuel cells (PEMFCs) require high purity hydrogen for maximum material performance and lifetime. The particulates in hydrogen used in FCVs and hydrogen powered internal combustion vehicles may adversely affect pneumatic control components, such as valves or other critical system components. The visualization of the size and morphology of particles is an important tool for determining particle origin as well as for devising particle formation reduction strategies.

6. Interferences

6.1 Particulate matter originating in the environment or equipment will interfere with the determinations. Every precaution should be taken to avoid contamination of particulates onto the filter coming from the analytical system, ambient air, filter handling, or other environmental sources.

6.2 The potential effect of body moisture or oils contacting the filters is minimized by using powder-free gloves while handling filters outside the glove box.

7. Apparatus

7.1 *Microscope*—A microscopy system is necessary to have reflectance and transmittance illuminations, built-in polarization system and a digital camera with an USB connection to a computer. The microscope is covered with a plastic cover when not in use and placed on a table top inside a horizontal flow hood containing a HEPA filter (7.3).

7.2 *Mini-Clean Room*—A small clean room with HEPA filter should be used to store unused TFE-flourocarbon filters, filter holders, and sampled filters at atmospheric moisture less than 30%.

7.3 *HEPA Filter Fitted Horizontal Flow Hood*—A flow hood that blows filtered air through a HEPA filter horizontally. This eliminates or reduces environmental particulates that can interfere with microscope visualization. The air velocity measured by Vaneometer (7.4) should be over 80 ft/minute (1.46 km/hour); otherwise, an electronic air velocity meter (7.5) should alarm the operator.

7.4 *Vaneometer*—This metering device is used to measure air velocity passing through the HEPA Filter fitted Horizontal Flow Hood.

7.5 *Electronic Air Velocity Meter*—An Electronic air velocity meter is used to notify the analyst if the horizontal air flow behind the microscope falls below approximately 80 ft per minute.

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

8. Reagents and Materials

8.1 *Filter*—A 47 mm diameter polytetrafluoroethylene filter (PTFE Membrane Disc Filters) is used. An example of a suitable filter is a Pall TF-200 47mm 0.2 µm (P/N 66143) with a pore size of 0.2 µm. 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, visualizing, handling, and weighing filters, the side facing the gas stream and collecting particulates must always face up. Before visualizing a filter by microscopy, examine it carefully to ensure the filter is not damaged and record the condition and appearance of the filter. Filters are always stored in a small particulate free plastic container in a mini clean room (7.2) when not in use.

9. Test Specimens and Test Units

9.1 *Test specimens*—Particulate.

9.2 *Test units*—µm.

10. Preparation of Apparatus

10.1 *Microscope*—The microscope, when not in use, must be covered with particulate free plastic and remain in a Horizontal Flow Hood (7.3) fitted with a HEPA Filter. The surface of the hood must be cleaned using a HEPA filter fitted vacuum (7.6) before visualization activity and the flow in the hood is turned on at least an hour before this activity.

11. Conditioning

11.1 *Filter Conditioning*—New filters are stored in their original packaging and the filters ready for visualization are stored in a mini-clean room as described in 7.2.

12. Procedure

12.1 Always clean horizontal flow hood HEPA filter air inlet surfaces using a HEPA Vacuum before handling filters.

12.2 Clean the surface area around microscope with a HEPA vacuum before performing visualizations.

12.3 Remove the plastic microscope covering inside the HEPA filter fitted horizontal flow hood. Place a Vaneometers (7.4) on one side of the microscope and an electronic air velocity meter (7.5) on the other side to ensure the air linear velocity is greater than 80ft/min.

12.4 Transfer filters stored in a plastic container from the mini clean room to the hood and adjacent to the microscope.

12.5 Use plastic tweezers to remove a filter from the plastic container and place it onto a clean glass surface under the microscopes objective lens. The glass is placed on a stage, which can be moved in different directions so that different portions of filter can be visualized.

12.6 Adjust the coaxial coarse and fine adjustments to focus the surface of filter. Use the lowest magnification and move the stage to locate particulates on the polytetrafluoroethylene filter.

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12.7 Use a higher magnification, reflectance polarizing light or transmittance illumination as needed to get the best visualization of particulates on the filter. The images of the particulates are taken by a digital camera interfaced to the microscope.

12.8 The digital image is input into Adobe Acrobat[4], or similar, software. The grid measurement tool of Adobe Acrobat is used to measure the size of the particulate. Select a scale ratio such that the length of 1.00 mm microscope calibration grid provided by microscope manufacturer to 1 mm, as shown in Fig. 1 which is an example of measurement of the sizes of particulates by Adobe Acrobat software. The scale ratio as shown in Fig. 1 of the Adobe Acrobat measurement tool is set so as the microscope calibration grid (1.00 mm total length) to be 1 mm. We found the distance between grids on polytetrafluoroethylene filters is close to 1 mm. After measurement, one can use "Export Measurement Makeup to Excel" tool to download all the measurements to an Excel[5] file for data process.

12.9 Use the largest diameter or measurement of the particle to associate a size to that particle. Particle size and any other observations, such as, pinholes, are recorded and submitted with the final report.

13. Report

13.1 Report particulate sizes, and any other observations or comments. Include images of particulates and their size measurement in the final report. However, there are several cases encountered in the particulate sizes analyses, which should be reported accordingly as described below.

13.2 *A few particulates on filter*—In this case, all the particulates images with their sizes should be reported with an example given in Fig. 2. However, if the transmission microscope cannot give clear image of the particulate, the reflective polarized light microscope should be used to give clear image and particulate size. An example is shown in Fig. 3 and Fig. 4, in which the image of particulate is taken by a transmission and polarized light reflective microscope, respectively. The polarized light reflective microscopic image apparently shows clearer image of the particulate.

13.3 *Pinhole on filter*—Polytetrafluoroethylene filter is in general not damaged; however, occasionally particulates with metallic nature, such as the one shown in Fig. 4, can penetrate the filter with pinholes left behind. Most of pinholes usually locate close to the center of the filter. In case pinholes are detected, the sizes and images of pinholes should be reported. An example of pinhole image and size is shown in Fig. 5.

13.4 *A lot of 100µm or smaller particulates at the center of filter*—Most of 100µm or smaller particulates usually locate at the centric circle on the filter of approximate 8mm OD. In this case, all the sizes of the particulates within this circle should be measured and their sizes downloaded into an Excel file, in which the particulate sizes are rearranged from small to large sizes. The number of particulates found in the different ranges of particulate sizes should be reported along with the images of the centric center on the filter containing most of small particulates. A portion of images of the centric circle on the filter with many 100µm or smaller particulates is shown as an example in Fig. 6. Any particulates found outside the 8 mm OD centric circle on filter should be reported as in 13.2.

[4] Trademarked by Adobe Systems Incorporated.

[5] Trademarked by Microsoft Corporation.

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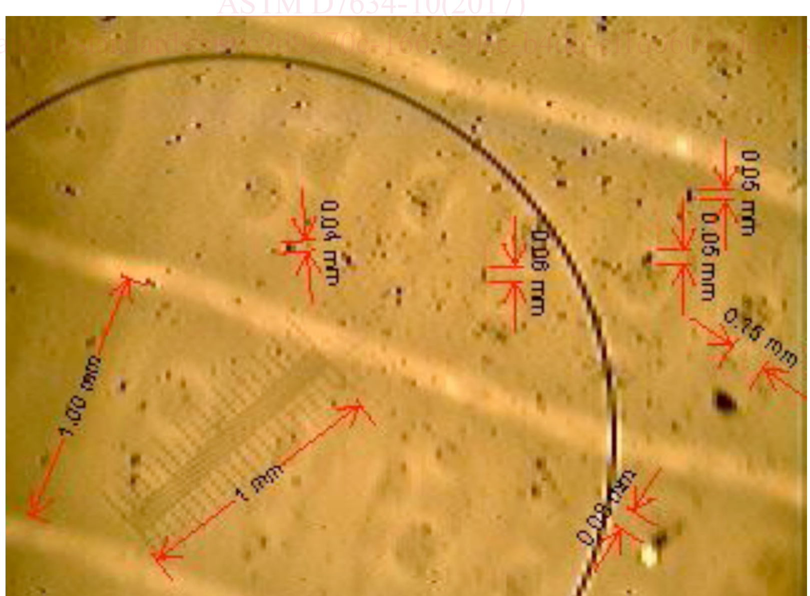

FIG. 1 An Example of Particulate Size Measurement