Designation: D7650 – 21

Standard Practice for Sampling of Particulate Matter in High Pressure Gaseous Fuels with an In-Stream Filter[1]

This standard is issued under the fixed designation D7650; 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 practice is primarily for sampling particulates in gaseous fuels up to a nominal working pressure (NWP) of 70 MPa (10 152 psi) using an in-stream filter. This practice describes sampling apparatus design, operating procedures, and quality control procedures required to obtain the stated levels of precision and accuracy.

1.2 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered 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

D7651 Test Method for Gravimetric Measurement of Particulate Concentration of Hydrogen Fuel

2.2 *NFPA Standard:*[3]

NFPA 2 Hydrogen Technologies Code

2.3 *CSA/ANSI/NGV Standards:*[4,5]

HGV 4.1 Hydrogen Dispensing Systems

NGV 1 Fueling Connection Devices

NGV 4.1 Natural Gas Vehicle (NGV) Dispensing Systems

2.4 *SAE Standards:*[6]

SAE J2579 Standard for Fuel Systems in Fuel Cell and Other Hydrogen Vehicles

SAE J2600 Compressed Hydrogen Surface Vehicle Fueling Connection Devices

SAE J2719 Hydrogen Fuel Quality for Fuel Cell Vehicles

2.5 *ISO Standard:*[7]

ISO 14687 Hydrogen Fuel Quality— Product Specification

2.6 *ASME Standard:*[8]

Boiler and Pressure Vessel Code

2.7 *UN Global Technical Regulation:*[9]

No. 13 Global Technical Regulation on Hydrogen and Fuel Cell Vehicles

2.8 *NIST Standard:*[10]

Handbook 44 Specifications, Tolerances, and Other Technical Requirements for Weighing and Measuring Devices

3. Terminology

3.1 *Definitions*—For definitions of general terms used in D03 Gaseous Fuels standards, refer to Terminology D4150.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *maximum allowable working pressure (MAWP), n*—the maximum gauge pressure of the working fluid (gas or liquid) to which a piece of process equipment or system is rated with consideration for initiating fault management above normal operation.

[1] This practice 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 National Fire Protection Association (NFPA), 1 Batterymarch Park, Quincy, MA 02169-7471, http://www.nfpa.org.

[4] Available from Canadian Standards Association (CSA), 178 Rexdale Blvd., Toronto, ON M9W 1R3, Canada, http://www.csagroup.org.

[5] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[6] Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

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

[8] Available from American Society of Mechanical Engineers (ASME), ASME International Headquarters, Two Park Ave., New York, NY 10016-5990, http://www.asme.org.

[9] Available from UNECE, https://unece.org/.

[10] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

3.2.2 *nominal working pressure (service pressure, working pressure), n*—the gauge pressure that characterizes typical operation of a pressure vessel, container, or system.

3.2.2.1 *Discussion*—For compressed hydrogen gas containers, NWP is the container pressure, as specified by the manufacturer, at a uniform gas temperature of 15 °C (59 °F) and full gas content.

3.2.3 *pinhole, n*—a small hole generated during sampling of particulates that can be identified by microscope.

3.3 *Abbreviations:*

MAWP—Maximum Allowable Working Pressure
PM—Particulate Matter
NWP—Nominal Working Pressure
PPE—Personal Protective Equipment
PRD—Pressure Relief Device

4. Summary of Practice

4.1 This practice provides a procedure for the sampling of particulate matter (PM) contained in gaseous fuels primarily used in motor vehicles. It is designed to collect all particulates 0.2 µm or larger contained in a known amount of fuel at a station dispenser nozzle in a way that simulates a vehicle fueling event.

4.1.1 The practice has two different approaches:

(1) The Tank Approach emulates a typical fueling by collecting the gas, which passes across the filter, into a tank.

(2) The Atmosphere Approach allows the gas to escape to atmosphere but sets a constant back pressure typically found during a fueling.

5. Significance and Use

5.1 This sampling procedure is used to collect a particulate filter sample containing particulates 0.2 µm or larger in size to be used to measure the size and concentration of particulates in a gaseous fuel stream.

6. Interferences

6.1 Dust and other environmental PM 10 µm or larger will interfere with the accurate measurement of particulates; therefore, every measure should be taken according to Section 13 to prevent contamination of the apparatus and all equipment, supplies, and gases used in these procedures.

7. Apparatus Design

7.1 Fig. 1 and Fig. 2 below show a recommended piping and instrumentation design of the two different sampling approaches. Both apparatuses have the following:

(1) Receptacle to attach to dispenser nozzle,

(2) Filter and filter holder,

(3) Pressure relief valve to protect against overpressurization,

(4) Control valves to regulate the gas,

(5) Pressure gauge to monitor the pressure of the apparatus, and

(6) Check valves to prevent back flow of the gas from the vent stack. If the apparatus has a tank, check valves should be placed upstream of the tank to prevent any gas from the tank from flowing backwards across the filter.

7.1.1 The requirements for these components and for components specific to sampling are discussed below. The design of the apparatus can vary if the requirements below are met.

7.2 *General Apparatus Requirements*—See CSA HGV 4.1 for general information on hydrogen and NGV 4.1 for general information on natural gas.

7.2.1 All equipment shall be designed to the maximum allowable working pressure (MAWP) of the dispenser.

7.2.2 All equipment shall be designed to the maximum peak flow rate of the dispenser. The peak flow rate for most dispensers for passenger cars is typically 60 g/s.

7.2.3 All apparatus components shall be constructed with materials compatible with the gaseous fuel being tested. High pressure hydrogen may alter the mechanical properties of common structural metal alloys. These effects are similar for all relevant structural metals and alloys in that hydrogen reduces the resistance to crack initiation and crack growth and reduces ductility. The reduction is dependent on several variables based on the material, the environment, and the mechanical loading conditions. This can lead to failure or hydrogen leaks. The sampling apparatus and all equipment used according to this test method must be closely inspected for signs of cracks or any other combination of signs of wear and damage.

7.2.4 The apparatus shall be built to withstand gaseous fuel temperatures ranging from −40 °C to 50 °C, except the tank which should have a temperature range of −40 °C to 85 °C.

7.2.5 The equipment shall be designed to operate in ambient conditions.

7.2.6 Refer to Section 10 (Hazards) for more information regarding safety requirements. A hazard analysis shall be performed on the apparatus. This shall be available to share with dispenser manufacturers/operators.

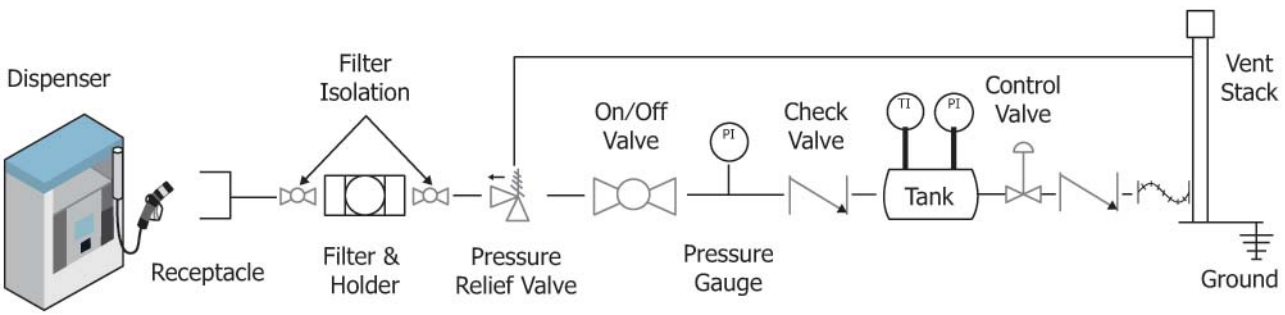

FIG. 1 Piping and Instrumentation Diagram Sampling Using the Tank Approach

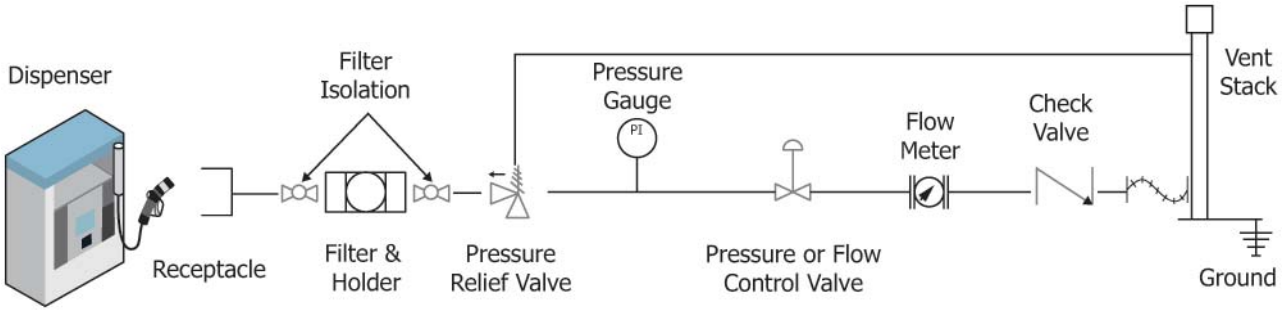

FIG. 2 Piping and Instrumentation Diagram Sampling Using the Atmosphere Approach

7.2.7 The flow path from the nozzle to the filter shall be designed to minimize trapping particulates on any surface.

7.2.8 All hose assemblies shall have an anti-whip that is attached to a suitable anchor point during sampling to prevent the hose from whipping should the connection become uncoupled.

7.3 *Receptacle*—The receptacles shall be designed in accordance with SAE J2600 or ISO 17268 for hydrogen or NGV1 for natural gas. Most receptacles have an integrated particulate filter installed in them. Receptacles for testing purposes shall not contain a filter.

7.4 *Filter Holder Requirements*—An example of a filter holder is shown in Fig. 3. The filter holder shall have an inlet adapter, item 1 from Fig. 3, that can be attached to a receptacle. The filter holder should be held together with holder screws, item 3 from Fig. 3, and inlet/outlet plates, items 4 and 8 from Fig. 3, or a combination of internal and external threads on the inlet/outlet plates (items 4 and 8). There are two O-rings, items 5 and 6 from Fig. 3, that ensure the filter holder is securely closed and no particulates can enter the system. The filter support screen, item 7 from Fig. 3, provides support to the filter and ensures it is not ruptured during a high-pressure pulse. The inlet and outlet adapters shall have a protective cap to maintain the sample integrity and keep particulates from entering the filter holder during transport.

7.4.1 *Filter Holder Support*—The filter holder support must be designed to securely hold the filter holder and dispenser hose nozzle in a downward vertical position. The nozzle shall be held firmly and not move or shake during particulate sampling.

7.5 *Pressure Relief Device (PRD)*—The apparatus should be equipped with a PRD to avoid over-pressurization. The PRD, including the release set pressures, shall meet the requirements of ASME Boiler and Pressure Vessel Code, Section XIII - Rules for Overpressure Protection. The PRD is not needed if all the equipment is rated to the MAWP of the dispenser.

7.6 *On/Off Valve (Tank Approach ONLY)*—A ball, needle, or plug valve shall be used to allow flow into the tank system.

7.7 *Pressure Gauge*—A pressure gauge which meets the general requirements listed above capable of providing a pressure reading up to the sampling apparatus MAWP.

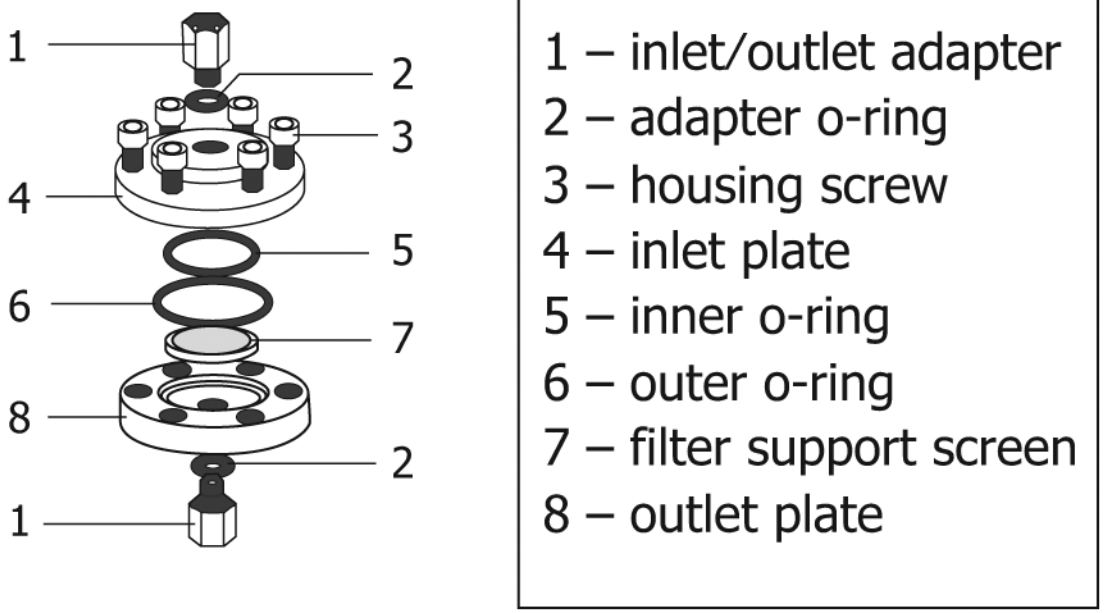

FIG. 3 Example of a High-pressure Filter Holder Design

7.8 *Pressure or Flow Control Valve (Atmosphere Approach ONLY)*—A pressure or flow control valve that can regulate the pressure during sampling to 20 MPa (2900 psi).

7.9 *Tank System (Tank Approach ONLY)*—For systems which use the Tank Approach, the tank shall meet the minimum requirements in one of the following standards: SAE J2579 or UN GTR 13. The high-pressure filter holder shall connect to the tank using a fueling hose made of material that conforms to the general requirements listed in 7.2. Unless the tank is Hazmat certified by local and government agencies, then it shall be exhausted completely before leaving the station.

7.10 *Vent Stack*—For systems which use the Atmosphere or Tank Approach, a portable vent stack or the refueling station vent stack shall be connected after the outlet of the particulate sampling holder for the atmosphere approach or used to exhaust the tank system; see 7.9.

7.10.1 *Location of Vent Stack*—Gaseous fuel must be vented at a safe distance from personnel and equipment. The ventilation outlet should be at a minimum distance from the sampling and the ground as calculated using NFPA 2, Chapter 7.3.2.3.1.1 tables based on the diameter of the vent stack. The vent stack should be placed clear of any overhead or nearby objects that may trap gas. Depending on the ambient and gas conditions, the gaseous fuel may not always flow upward. Ensure that venting is performed in accordance with applicable national, regional, and local laws and regulations. It is recommended to set the vent downwind of the dispenser and sampling event.

7.11 *Grounding*—All components of the apparatus shall always be electrically grounded to earth. Grounding using the nozzle and hose of the dispenser is not sufficient.

7.12 *Mass Flow Measurement Requirements*—The mass of gas which passes across the filter can be measured using several methods.

7.12.1 For systems which use the Tank Approach, the temperature and pressure measurement would be used. The tank shall be equipped with a temperature and pressure sensor to record the initial and final tank temperatures and pressures. Both sensors shall have an accuracy of at least ± 1%, or

7.12.2 For systems which use the Atmosphere Approach, a Coriolis mass flow meter, calibrated for the appropriate gas that can measure the grams per second of gaseous fuel sampled, shall be installed after the particulate sampling holder. This device should include a totalizer that will calculate the mass of gas sampled. The flow meter shall be installed downstream of the filter holder, measure the amount of gas sampled, and meet the requirements for dispensing in NIST Handbook 44, or

7.12.3 For systems which use the Tank or Atmosphere Approach, the dispenser may provide a total amount dispensed. Some dispensers may not be able to measure while in manual mode.

8. Additional Equipment Needed

8.1 *Leak Detector*—A leak detector is a required safety device needed to detect small gas leaks when the sampling apparatus is pressurized prior to particulate collection. Diluted soap bubbles or any liquid shall not be used to detect leaks due to possible contamination.

8.2 *Anti-static Bags*—Anti-static bags shall be used during transport of the filter and filter holder. This will prevent any static buildup during transport and ensure that no particulates will enter the filter holder.

8.3 *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 always be kept clean, and any visual PM must be removed immediately. The glove box should always have a steady flow of clean, dry nitrogen. The temperature and humidity should be kept consistent at 21 ± 2 °C and 35 ± 5 % relative humidity and should be monitored by a data logger or other device installed in the glove box.

8.4 *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 PM from the bottoms of shoes. The room must have a HEPA air filter on the inlet air system to remove particulates from the air.

9. Reagents and Materials

9.1 Materials need to be compatible with the gaseous fuel being tested and pressures being used.

9.2 *n-Heptane, ≥ 99 % Minimum Purity,* for cleaning the filter holder. Should be filtered through a 0.2 µm filter. (**Warning—**Flammable. Harmful if inhaled. Causes skin irritation.)

10. Hazards

10.1 *Personal Protective Equipment (PPE)*—The following PPE shall always be worn during particulate sampling: eye protection, hearing protection, flame resistant lab coat/clothing, safety vest, and steel-toed/composite and electro-static dissipative shoes. A hard hat and safety cones may be necessary. It may also be necessary to comply with any additional safety and PPE requirements of the station owner/operator.

10.2 Only trained personnel shall be involved with sampling. Personnel not directly involved in sampling should be at least 5 m (16 ft) away from the sampling site.

10.3 *Visual Inspection*—All the equipment used according to this practice must be closely inspected for signs of cracks, corrosion, or any other combination of signs of wear and damage.

10.4 Ignition sources, such as smoking and unclassified electronic devices, shall be avoided within 7.6 m (25 ft) of the fueling station and vent stack.

10.5 During particulate sampling, the high speed of gas flow may generate a static charge on the sampling apparatus.