Designation: D7650 – 13 D7650 – 21

Standard Test Method Practice for Sampling of Particulate Matter in High Pressure Hydrogen used as a Gaseous FuelGaseous 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 test method practice is primarily for sampling particulates in hydrogen fuel used in hydrogen fuel cell vehicles or gaseous hydrogen powered internal combustion vehicle engines up to pressures gaseous fuels up to a nominal working pressure (NWP) of 70 MPa (700 bars)(10 152 psi) using an in-stream filter. This test method 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. No other units of measurement are included in this The values given in parentheses after SI units are provided for information only and are not considered standard.

1.2.1 *Exception*—In 7.1 and 10.1.1 the values stated in psi are for information only.

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.*

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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

[1] This test method 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.

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 ~~J2719~~J2579 ~~Hydrogen Fuel Quality~~ Standard for Fuel ~~Cell~~ Systems in Fuel Cell and Other Hydrogen Vehicles
SAE J2600 Compressed Hydrogen Surface Vehicle ~~Refueling~~Fueling Connection Devices
SAE J2719 Hydrogen Fuel Quality for Fuel Cell Vehicles
2.5 *ISO Standard:*[7]
~~ISO/CD 14687–2~~ISO 14687 Hydrogen ~~fuel — Product Specification — Part 2: Proton exchange membrane (PEM) fuel cell applications for road vehicles.~~Fuel Quality— Product Specification
2.6 ~~*EU*~~*ASME Standard:*[8]
~~97/23/EC Pressure Equipment Directive of the EU set out the standards for the design and fabrication of pressure equipment~~Boiler and Pressure Vessel Code
2.7 ~~*DIN Standard:*~~*UN Global Technical Regulation:*[9]
~~DIN EN 12266-1~~No. 13 ~~Industrial valves-Testing of metallic valves-Part 1: Pressure test, test procedures and acceptance criteria Mandatory Requirements~~Global Technical Regulation on Hydrogen and Fuel Cell Vehicles
2.8 ~~*API*~~*NIST Standard:*[10]
~~API 598~~Handbook 44 ~~Valve Inspection and Testing~~Specifications, Tolerances, and Other Technical Requirements for Weighing and Measuring Devices

3. Terminology

~~3.1 *Acronyms:*~~

~~3.1.1 *FCV*—Hydrogen Fuel Cell Vehicle.~~

~~3.1.2 *HQSA*—Hydrogen quality sampling assembly for sampling gaseous hydrogen fuel.~~

~~3.1.3 *PEM*—Polymer Electrolyte Membrane or Proton Exchange Membrane~~

~~3.1.4 *PSA-H70*—Particulate sampling adapter for sampling particulate in hydrogen fuel up to pressures of 70 MPa.~~

~~3.1.5 *SAE International*—Society of Automotive Engineering~~

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

3.2 ~~*Definitions:*~~*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.

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.

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[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 ~~European Committee for Standardization (CEN), Avenue Marnix 17, B-1000, Brussels, Belgium, http://www.cen.eu.~~American Society of Mechanical Engineers (ASME), ASME International Headquarters, Two Park Ave., New York, NY 10016-5990, http://www.asme.org.
~~[6] Available from Deutsches Institut fur Normung e.V.(DIN), Burggrafenstrasse 6, 10787 Berlin, Germany, http://www.din.de.~~
~~[7] Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, http://www.api.org.~~
~~[8] HEPA is a trademark of the HEPA Corporation, 3071 East Coronado Street Anaheim, CA 92806.~~
[9] ~~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.~~Available from UNECE, https://unece.org/.
[10] ~~Microsoft Excel is a trademark of the Microsoft Corporation, One Microsoft Way Redmond, WA 98052-6399.~~Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

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

3.3 ~~*SAE J2719 Hydrogen Fuel Quality for Fuel Cell Vehicles*—~~*Abbreviations:*
~~This~~ *MAWP* ~~document specifies allowable levels of constituents in the hydrogen fuel at the vehicle/station interface.~~—Maximum Allowable Working Pressure
PM—Particulate Matter
NWP—Nominal Working Pressure
PPE—Personal Protective Equipment
PRD—Pressure Relief Device

~~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~~Practice

4.1 This ~~test method~~ practice provides a procedure for the sampling of particulate matter (PM) contained in ~~hydrogen used as a FCV fuel.~~ gaseous fuels primarily used in motor vehicles. It is designed to collect all particulates ~~0.2 µm~~ 0.2 µm or larger contained in a known amount of ~~hydrogen~~fuel at a station dispenser nozzle in a way that simulates a ~~FCV or a gaseous hydrogen powered internal combustion vehicle engine fueling event. The adapter used for sampling particulates in hydrogen fuel is called a Particulate Sampling Adapter for pressures up to 70 MPa (PSA-H70) and is described in Section~~ vehicle fueling event. ~~7. Great care should be taken to avoid contamination and exposure of the PSA-H70, filters, and other equipment with particles sized 10 µm or larger prior to use.~~

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 ~~Fuel cells such as proton exchange membrane fuel cells require high purity hydrogen for maximum material performance and lifetime. Collection and measurement of particulate matter 0.2 µm or larger is necessary for assuring a feed gas of sufficient quality to satisfy fuel cell system needs. In addition, internal combustion engines using high pressure hydrogen fuel also require low particulate levels. Specifically, particulate matter has been implicated in the premature failure of pneumatic control components, such as valves within vehicles.~~ This sampling procedure is used to collect ~~and measure samples containing particles 0.2 µm~~ a particulate filter sample containing particulates 0.2 µm or larger in ~~size.~~size to be used to measure the size and concentration of particulates in a gaseous fuel stream.

~~5.2 Although not intended for application to gases other than hydrogen and related fuel cell supply gases, the techniques within this sampling procedure can be applied to other high pressure gaseous samples requiring particulate collection and measurement.~~

6. Interferences

6.1 Dust and other environmental ~~particulate matter~~ PM 10 µm or larger will interfere with the accurate measurement of ~~particulates contained in FCV quality hydrogen;~~ particulates; therefore, every measure should be taken according to Section ~~14~~13 to ~~avoid~~prevent contamination of the apparatus and all equipment, supplies, and gases used in these procedures.

7. Apparatus Design

~~NOTE 1—The use of trade names in this section are not intended as an endorsement for use.~~

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,

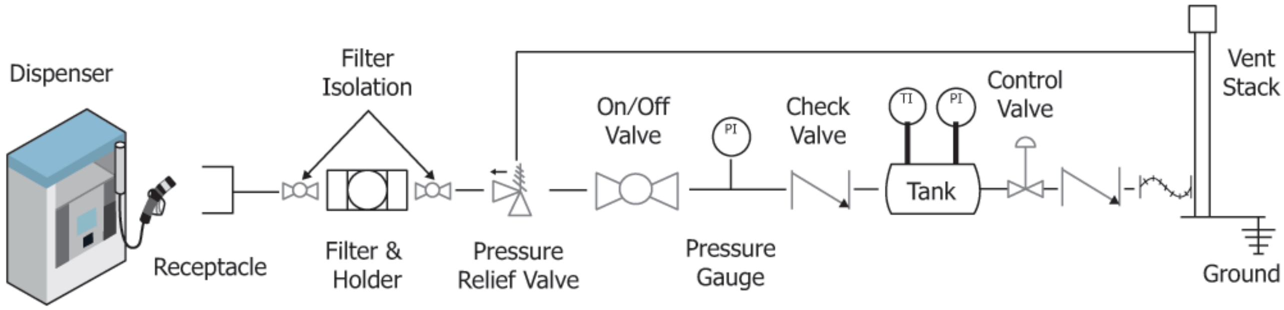


FIG. 1 Piping and Instrumentation Diagram Sampling Using the Tank Approach

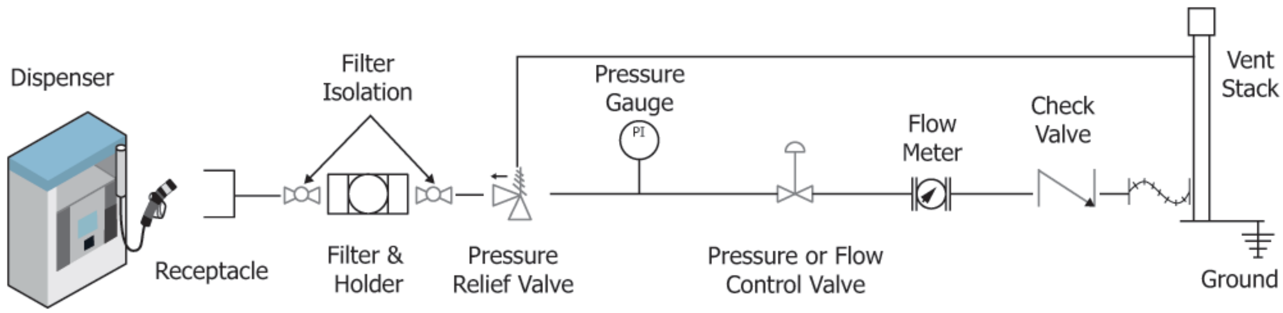


FIG. ~~1~~2 ~~PSA-H70 Components~~Piping and Instrumentation Diagram Sampling Using the Atmosphere Approach

(2) Filter and filter holder,

(3) Pressure relief valve to protect against over-pressurization,

(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.

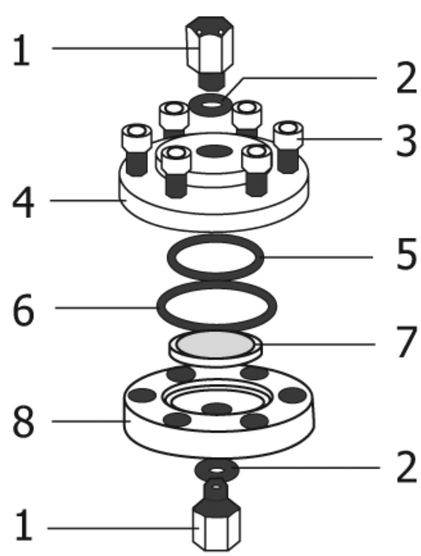
7.2.7 The flow path from the nozzle to the filter shall be designed to minimize trapping particulates on any surface.

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—*~~The PSA-H70 has a design pressure of 800 Bar (11600 psi) and is certified after 97/23/EC and API 598 (conform to DIN EN 12266-1) up to 1200 bar (17400 psi) shell test pressures with appropriate safety factors built in. It is designed for a flow rate of 60 g per second of hydrogen without damage to the filter or leakage from the PSA-H70. The PSA-H70 possesses a receptacle as per SAE J2600 followed by a rotary valve which is directly integrated~~An example of a filter holder is shown in Fig. 3~~to the filter housing inlet. The rotary valve provides the possibility to switch the inner diameter of the filter housing inlet~~. The filter holder shall have an inlet adapter, item 1 from Fig. 3~~4 mm to 1 mm. That prevents the filter element from high pressure pulse damage.~~, that can be attached to a receptacle. The filter holder ~~contains~~ should be held together with holder screws, item 3 from Fig. 3, and inlet/outlet plates, items 4 and 8 from Fig. 3~~a standard 47 mm diameter filter element (≥ 0.2 µm pore size) for particle collection, supported by a sinter metal frit with a pore size of 150 µm. Downstream the sinter metal frit a high pressure bleed plug is integrated into~~, 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 ~~followed by a standard hydrogen refueling hose and nozzle as per SAE J2600 to be connected to a FCV during sample collection. In summary, the configuration of the PSA-H70, as shown in~~ is securely closed and no particulates can enter the system. The filter support screen, item 7 from Fig. ~~1~~3, ~~is:~~provides ~~A SAE J2600 compliant Receptacle → Filter Holder with integrated rotary valve, the Filter Element supported by a Sinter Metal Frit and a an integrated high pressure Bleed Plug→Refueling Hose→ a SAE J2600 compliant Fueling Nozzle.~~ 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.

~~Fig. 1 illustrates a PSA-H70 design that has been successfully used to collect particulate samples from 875 bar (12691 psi) fuel cell quality hydrogen. The PSA-H70 should be rated above the operating pressure, and all materials used must be rated for high pressure hydrogen applications at a 1.5 times minimum margin of safety at the maximum operating pressure. The recommended working pressure of the PSA-H70 and associated materials is 80 MPa. Contamination from lubrication or other sources must be avoided and the apparatus must be cleaned prior to use using appropriate cleaning techniques for high pressure hydrogen applications. The design of the PSA-H70 should include minimizing the distance and surface area between the nozzle and filter to minimize the particulates generated from the surface of this area by fast flow and high pressure hydrogen~~



1 – inlet/outlet adapter
2 – adapter o-ring
3 – housing screw
4 – inlet plate
5 – inner o-ring
6 – outer o-ring
7 – filter support screen
8 – outlet plate

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

~~7.1.1 *High Pressure Filter Holder*—The high pressure filter holder is a 47 mm, stainless steel housing with maximum inlet pressure of 120 MPa a polytetrafluoroethylene (PTFE) inner 40.94 mm diameter PTFE-O ring and a hydrogenated acrylnitrilbutadiene caoutchouc as high pressure sealing. The filter holder must be equivalent, similar or exceed performance characteristics of the filter holder described in this test method, that is burst strength.~~

~~7.1.2 *Filter*—A polytetrafluoroethylene (PTFE) filter that tolerates flow rates of up to 60 g per s without damage and collects particulates with a minimum size of 0.2 µm.~~

~~NOTE 2—Hydrogen back flow must be avoided since the backflow of hydrogen can cause pinhole formation or other damage to filters. The design of the apparatus and sampling procedures must prevent fuel backflow.~~

7.4.1 ~~*PSA-H70*~~ *Filter Holder Support*—The ~~mechanical PSA-H70~~filter holder support must be designed to securely hold the ~~PSA-H70 and a J2600 nozzle. The nozzle should~~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.

~~7.1.4 *PSA-H70 design for flowing hydrogen to gaseous hydrogen vehicle tank*—As sampling during refueling into a vehicle tank collects a sample most representative of the particulates seen by vehicles in service, the PSA-H70 is designed for this purpose. Therefore, for particulates collection the refueling nozzle of the station is connected to the SAE J2600 receptacle of the PSA-H70 and the SAE J2600 nozzle of the PSA-H70 is attached to the FCV receptacle. While sampling, the hydrogen fuel flows from station SAE J2600 nozzle (1st nozzle) → PSA-H70 → with hose and a 2nd SAE J2600 nozzle → receptacle of FCV tank.~~

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.

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—~~Box (Option A)*—A glove box is a sealed ~~container that is designed to assemble PSA-H70~~chamber that, in this application, allows weight measurements to be taken without particulate contamination from ambient air. Two gloves are built into the ~~sides~~ front side of the glove box ~~with entry arranged in such a way~~ so that the user can place their hands into the gloves ~~to install the filter~~ and perform weight 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 particulate free at all times. Any visual particulate material~~ always be kept clean, and any visual PM must be removed ~~prior to working with the PSA-H70 or filters. A HEPA~~immediately. The glove box should always have a steady flow ~~vacuum can be used to remove particles from the glove bag and other equipment.~~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.

~~8.2 *Relative Moisture/Temperature Data Logger*—A data logger is placed inside the glove box to measure both moisture and temperature continuously at pre-defined intervals such as once every two to five minutes. Moisture in the glove box is kept between 15 to 30% using reagent grade nitrogen. All temperature and moisture data are stored in a data logger, which are downloaded into a Microsoft Excel[10,9], or a similar program, sheet after completion of measurements.~~

~~8.3 *Mini-Clean Room*—A small clean room with HEPA air filtration must be used to store unused polytetrafluoroethylene (PTFE) filters, filter holders, and sampled filters at moisture content less than 30%.~~

~~8.4 *Ultrasonic Cleaner*—Either an ultrasonic bath or probe is used in lab to shake off particulates on parts of PSA-H70 into reagent grade water.~~

~~8.5 *Hydrogen Leak Detector*—A hydrogen leak detector is a required safety device needed to detect small hydrogen gas leaks in particular when the PSA-H70 is pressurized prior to particulate collection. The diluted soap bubble should not be used to detect hydrogen leak from PSA-H70.~~

~~8.6 *HEPA Filter Horizontal Flow Hood*—A HEPA filter horizontal flow hood blows filtered air through a HEPA filter horizontally, providing for an environment with minimal suspended particulates. The air velocity measured by an air flow meter (8.8) within the hood should be over 30 m/min. If below this velocity, the air velocity meter should trigger an alarm notifying the operator about a low air velocity.~~

~~8.7 *Plastic tweezers*—Used to manipulate filters without contamination.~~