



Designation: D8283 – 19

Standard Practice for Cleaning and Certification of Specially Prepared Canisters¹

This standard is issued under the fixed designation D8283; 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 describes procedures to clean and verify the cleanliness of reusable sample containers used in air sampling and analysis applications, which includes but is not limited to passivated stainless steel canisters and rigid containers of other material specifically designed for whole-air sampling. This practice describes the steps used to clean volatile substance from canister interiors and document cleanliness prior to re-use.

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, 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

[D1193 Specification for Reagent Water](#)

[D1356 Terminology Relating to Sampling and Analysis of Atmospheres](#)

[D5466 Test Method for Determination of Volatile Organic Compounds in Atmospheres \(Canister Sampling Methodology\)](#)

¹ This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

Current edition approved Dec. 15, 2019. Published February 2020. DOI: 10.1520/D8283-19.

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.

2.2 EPA Documents:³

[EPA TO-15 Compendium Method TO-15, Determination of Volatile Organic Compounds \(VOCs\) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry \(GC/MS\), Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air](#), second edition, January 1999

[NATTs EPA Technical Assistance Document for the National Air Toxics Trends Stations Program](#), revision 3, October 2016

2.3 DoD Document:⁴

[DoD Department of Defense Quality Systems Manual for Environmental Laboratories](#), version 5.1.1, 2018

3. Terminology

3.1 Definitions—

3.1.1 For definitions of terms used in this practice, refer to Terminology [D1356](#).

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *cleaning batch, n*—a grouping of canisters cleaned concurrently on the same cleaning “system” (heated isothermal or unheated).

3.2.1.1 *Discussion*—Typical sizes range from 4 to 20 canisters/batch.

3.2.2 *cleaning cycle, n*—a standardized duration in which canisters are serially filled and evacuated.

3.2.2.1 *Discussion*—This process can be automated or manual depending on application.

3.2.3 *contaminated canister, n*—a canister pending cleaning and cleanliness verification/re-certification.

3.2.4 *other prepared containers or vessels, n*—other rigid containers designed for re-use, which may include but is not limited to inert-coated glass bottles used for collecting whole air samples.

³ Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

⁴ Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, <http://www.access.gpo.gov>.

3.2.5 *passivated canister, n*—a stainless steel air sampling canister whose inner surface is first electropolished to decrease surface roughness.

3.2.5.1 *Discussion*—The electropolishing may be followed by application of either a coating of nickel/chromium oxide (or similar) or a layer of fused silica (silicon dioxide) so as to increase the canister's inertness as compared to untreated stainless steel.

3.2.6 *quality control canister (QC canister), n*—canister that is selected and analyzed from a cleaning batch to verify canister cleanliness requirements have been satisfied.

3.2.7 *virgin canister, n*—a canister that is pending its initial cleaning, verification and certification.

3.2.8 *zero-grade air or nitrogen, n*—“hydrocarbon-free” air or nitrogen used as purge/fill gas, with typically <0.10 ppmv of total hydrocarbons.

4. Summary of Practice

4.1 This procedure describes *Best Practices* for cleaning and certifying reusable sample vessels designed for volatile organic compound collection. Contaminated and virgin canisters are first evacuated to remove the bulk contents from each canister. Once evacuated, the canisters are connected to a cleaning system, where they are serially evacuated and filled with humidified zero air or nitrogen. Depending on canister cleanliness and the system used, this process may take anywhere from a few hours to a few days to complete. Following the cleaning process, a minimum of one canister per batch or 5 % of the total batch, whichever is larger, is selected as the QC canister(s). The QC canister is analyzed for target compounds of concern using the same analytical test method as applied to the intended samples, or other accepted analytical method that has demonstrated equivalent performance. Following the cleaning procedure, the canisters are either pressurized or evacuated and stored for a minimum of 24 hours to verify that the canisters do not leak. The pressure of each canister is evaluated using a calibrated pressure/vacuum gauge and the results are recorded. All canister batches that pass both the batch QC and individual leak check are verified as ready for use.

5. Significance and Use

5.1 Whole-air sampling vessels are cited and utilized throughout a variety of internationally, nationally, and regionally applied documents and test methods, yet the methods generally fail to uniformly specify criteria used to qualify the use and re-use of sample containers. This practice describes a set of best practices for reusable containers and canister media cleaning, quality control (QC) objectives, and corrective actions necessary to ensure equipment that meets cleanliness needs of their intended use.

6. Reagents

6.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I or II of Specification D1193.

7. Hazards

7.1 *Electrical Shock*—All electrical wiring should be periodically checked for insulation cuts and chafing as electrical shock can occur from exposed wires.

7.2 *Fires*—All flammable materials, especially solvents, shall be kept clear of the cleaning system and pumps since the canister heaters generate considerable heat.

7.3 *Pollution Prevention and Waste Management*—Air purged from canisters via vacuum pumps should be discharged under a fume hood or to the outside air.

8. Equipment and Supplies

8.1 *Initial Evacuation System*—A manifold system for evacuating the canister constituents prior to cleaning may be used. While initial evacuation is not necessary, it may assist expediting the cleaning process and may be used to prevent gross contamination of the canister cleaning system.

8.1.1 The vacuum manifold consists of a vacuum pump and may include a gas supply connected to a series of canister positions, which connect directly to the canister to be evacuated.

8.1.2 The purge cycles may be controlled automatically with a three-way solenoid valve; one position connects to the gas source, the second position connects to a pump, and the third position connects to the manifold. The canisters are then purged by serial evacuation and dilution.

8.1.3 Recommendations on the quantity and duration of cycles recommendation is not given within this practice, as the parameters are dependent on the container contents.

8.2 *Canister Cleaning System*—The canister cleaning system is comprised of cleaning manifolds, a gas source, high vacuum pump(s), vacuum/pressure gauges, humidification apparatus if necessary, and an oven capable of maintaining $100^{\circ}\text{C} \pm 10^{\circ}\text{C}$.

8.3 *Canister Manifolds*—Manifolds are, typically, constructed of tubing made of stainless steel. Canisters are attached to the oven manifold with a compression fitting. Each manifold has its own pump and vacuum gauge, but may share a common gas source.

8.3.1 *Vacuum Pumps*—Typical pumps that can be used include, but are not limited to: dual-stage direct driven rotary-vane type, two-stage oil-less pumping system comprised of a molecular drag pump backed by a diaphragm-type roughing pump with integrated vacuum controller, or other pump capable of allowing the cleaning and final evacuation process to meet the intended use cleanliness objectives.

8.3.1.1 A definition criteria table for various vacuum levels can be found in Table 1.

8.3.2 *Electric Ovens with Single or Multi-Connection Manifold*—Canisters are heated during the cleaning process. Degradation of the canister coating can occur if the canister exceeds manufacturers specified limits, and so a typical maximum heating temperature is $100^{\circ}\text{C} \pm 10^{\circ}\text{C}$, but may be as high as $110^{\circ}\text{C} \pm 10^{\circ}\text{C}$.

8.3.2.1 The maximum allowable oven temperature may be lower than $100^{\circ}\text{C} \pm 10^{\circ}\text{C}$ depending on canister manufacturer recommendations.

TABLE 1 Pressure Ranges According to ‘Degree of Vacuum’^A

Absolute Pressure, Pa	Absolute Pressure, Pa	‘Degree of Vacuum’
1×10^5 to 3×10^3	100 000 to 3 000	low vacuum
3×10^3 to 1×10^{-1}	3 000 to 0.1	medium vacuum
1×10^{-1} to 1×10^{-4}	0.1 to 0.000 1	high vacuum
1×10^{-4} to 1×10^{-7}	0.000 1 to 0.000 000 1	very high vacuum
1×10^{-7} to 1×10^{-10}	0.000 000 1 to 0.000 000 000 1	ultra-high vacuum (UHV)
$<1 \times 10^{-10}$	$<0.000\ 000\ 000\ 1$	extreme-ultrahigh vacuum (EHV or XHV)

^A National Physical Laboratory (NPL), “What Do ‘High Vacuum’ and ‘Low Vacuum’ Mean? (faq – Pressure),” Mar. 10, 2010, date of access November 2018, available from: [http://www.npl.co.uk/reference/faqs/what-do-high-vacuum-and-low-vacuum-mean-\(faq-pressure\)](http://www.npl.co.uk/reference/faqs/what-do-high-vacuum-and-low-vacuum-mean-(faq-pressure)).

9. Procedure

9.1 *Initial Evacuation*—Evacuation is a desirable step to be taken with all contaminated canisters, but it may not be required if anticipated concentrations are known to be low, or when cleaning virgin canisters.

9.2 *Cleaning System Preparation:*

9.2.1 Establish the number of cleaning cycles needed to achieve cleanliness objectives. For automated systems configure the system to perform the desired number of evacuation and fill cycles. This will require setting a vacuum and pressure needed for each cleaning cycle. On completion of the prescribed cleaning cycle a canister(s) is chosen for cleanliness verification.

9.2.2 Canisters chosen for cleanliness verification are filled with zero-grade air or nitrogen (source gas) prior to final QC analysis.

9.2.3 Verify that the source gas is free of contaminants exceeding intended use criteria by performing analysis of the gas according to the intended analytic method for samples to be analyzed. Scrubbing of source gas with additional hydrocarbon traps or catalytic oxidation, or both, may be necessary to obtain sufficiently clean source gas. When using zero-air as the source gas, lower temperatures should be maintained during the cleaning process in order to avoid oxidation of interior canister surfaces.

9.2.4 When required or as needed, the source gas should be humidified to approximately 30 % to 70 % relative humidity (NATTs). The water assists in removal of compounds which may otherwise remain adsorbed to interior canister surfaces. Water also generates a sample matrix typical of environmental samples. Most commercial canister cleaning systems incorporate an up-stream humidifier, however these may not provide a sufficient level of humidity. If a bubbler-type humidifier is employed, care should be taken to ensure the downstream pressure is lower than the humidifier upstream pressure to avoid backflow of the water. It is recommended that the relative humidity of the gas being introduced into the canister cleaning manifold be measured with a calibrated hygrometer so that desired relative humidity percentage is attained. Verify the humidification system/water used will not introduce contamination above intended use tolerance to canisters.

9.2.5 Verify the cleanup system is leak free according to manufacturer specifications.

9.2.6 After starting the pump and opening valves to the cleaning manifold, allow the system to stabilize over several minutes.

9.2.7 The oven temperature should be set no higher than 80°C for inert-coated canisters and 100°C for electropolished canisters to prevent damage to either the lining or the canister valves.

9.3 *Canister Cleaning:*

9.3.1 Connect canisters to be cleaned to the cleaning manifold. Record data pertinent to the vacuum and pressure cleanup cycles as they are completed.

9.3.2 Start the cleaning cycle program or perform the cycle manually.

9.4 *QC Canister Selection*—A minimum of one canister or 5 % of the total batch, whichever is larger, must be analyzed as the QC canister(s). Typically, 10 % of the canister batch is considered optimal, and canisters are chosen based off of the criteria outlined in 9.5.

9.5 *QC Canister Selection:*

9.5.1 The most general practice is to select the canister in the batch with the highest contaminant concentrations from its previous use. This protocol assumes that if this canister passes the QC criteria, canisters with lower contaminant concentrations will also pass. This general recommendation is made assuming all of the canisters of the batch have similar concentrations and the previous sample concentration is available for review.

9.5.2 Depending on user application, a QC canister may also be chosen based on prior sample analysis showing contamination of semi-volatile target analytes. This protocol assumes that compounds with higher boiling points require the greatest effort in removal, and by removing the semi-volatile species, lesser volatile species will be removed as well.

9.5.3 If batches are created out of canisters with similar analytes and concentrations, then a “random selection” approach may be acceptable for QC canister selection. Random selection is not an optimal decision when previous contaminant concentrations are known, and the user should aim to ascertain selection based on the above criteria.

9.6 The QC canister is analyzed for analytes that are the targets for subsequent samples collected in the canister. Instrument quantitation report, chromatogram, and other instrument data along with the associated canister cleaning batch log must be stored and available for review. Typical archiving duration for cleaning batch information is five years as outlined by many regulatory body recommendations, but this practice does not specify required archival periods.