A Guide to Passive Air Sampling, **Using Canisters**



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Thank you for requesting this Restek technical guide.

One of our standing goals is to provide you with practical information to help you obtain reliable data from your chromatographic and peripheral systems. This guide describes equipment needed and practical techniques to follow when collecting passive air samples, using canisters. It is a compilation of information based on our experience and that of experts in this field. We would like to thank the individuals who provided invaluable assistance in the development of this guide: Dr. Eric Winegar of Applied Measurements Science, Joachim Pleil of the US Environmental Protection Agency, John Yokoyama of Performance Analytical, and Jerry Winberry of Envirotech Solutions.

We hope you enjoy this first edition guide to passive air sampling. If you have any questions, or have input for future editions, please feel free to contact us at Restek Corporation.

David M. Shelow Air Monitoring Products

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Introduction

Ambient air sampling involves collecting a representative sample of ambient air for analysis. If the environment is not changing, or if only a qualitative sample is needed, a simple "grab" sample can be obtained. For example, an evacuated sample canister can be opened and sample rapidly collected at a non-controlled rate, usually over several seconds, until the container attains equilibrium with atmospheric pressure. Generally this qualitative approach is used when unknown analytes must be identified, when the air contains high concentrations at certain (short) times, or when an odor is noticed and a sample must be obtained quickly. Paired grab samples (before/after or smell/no smell) often are employed to qualitatively diagnose a perceived problem.

To obtain a more representative sample requires time-integrated sampling. A flow restrictor is used to spread the sample collection flow over a specific time period, to ensure an "average" composited or time-weighted average (TWA) sample. A TWA sample will accurately reflect the mean conditions of the ambient air in the environment and is preferred when, for regulatory or health reasons, a typical exposure concentration is required for a situation that may have high variability, as in an occupational setting.

There are two general approaches to collecting air samples: 1) "whole air" sampling with canisters or Tedlar[®] bags and 2) "in-field concentration" sampling using sorbent tubes or cold traps. In this guide we focus on collecting whole air samples in canisters. Within this approach, two sampling techniques commonly are used: passive sampling and non-passive (active) sampling, distinguished by the absence or use of an active pumping device, respectively.

In passive sampling an air sample is pulled through a flow controller into an evacuated canister over a chosen period of time, ranging from 5 minutes to 24 hours. The sampling period and the flow rate determine the canister volume required. In non-passive (active) sampling, a pump is used to push the sample through a mass flow controller and into the canister. Additional sample can be collected, relative to the amount that can be collected by passive sampling, by pressurizing the canister with sample. Commonly the sample is pressurized to 15psig, effectively doubling the sample volume. Sampling can be time-integrated (e.g., an 8-hour sample), or a dip tube design can be used to establish a flow through the system and flush the sample container with sample, then, after a specified time, the exit valve is closed and the container is pressurized with sample.

Although active sampling is very flexible, a drawback to using a pump is the need for additional quality assurance requirements for sample integrity (i.e., no artifacts or loss of analytes). Additionally, a pump requires a battery or line power source, which may pose logistic difficulty in remote field-site sampling.

II. Equipment Used for Passive Air Sampling

To ensure a valid sample when using a passive sampling technique, it is important that the flow rate not change greatly during the time interval specified for the integrated sample. The proper sampling equipment helps accomplish this objective. A typical passive sampling train should include the following components, all constructed of stainless steel: a sampling inlet, a sintered metal particle filter, a critical orifice, a flow controlling device, a vacuum gauge, and a canister (Figure 1).

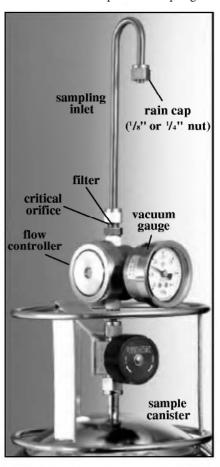
Sampling Inlet

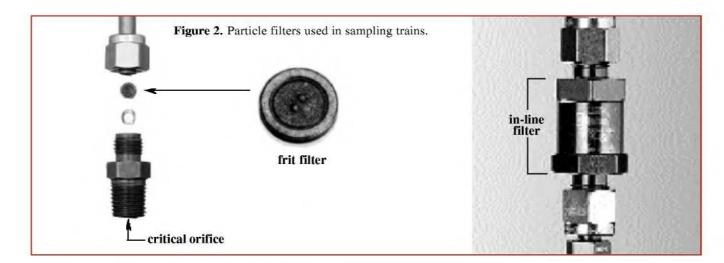
The sampling inlet - the entrance to the sampling train - typically is cleaned stainless steel tubing, either 1/4" ID or 1/8" ID. US EPA Compendium Method TO-14/15 recommends sampling at a height of 2 meters above the ground. In a highly trafficked area, this would minimize the problem of dust particles entering the sampling train. This height is not mandatory, however, and it is common practice to use an inlet that is 12" (approximately 1/3 meter) high. The EPA also recommends having the entrance of the sampling inlet face downward, to prevent raindrops from entering the inlet. In some sampling trains a 1/8" or 1/4" nut at the entrance of the inlet keeps water droplets away from the edge of the inlet, where they could be drawn into the sampling train with the sample.

Particle Filter

Installed in the sampling train prior to the flow-controlling device, the particle filter prevents airborne particles from entering the sample flow path. Particles could partially obstruct the flow path and alter the flow rate during sampling. In extreme cases, particles could plug the flow path and stop the sample flow. The smallest orifice commonly used in a passive sampling train is 0.0012" (approximately 30 micrometers). Without a particle filter, dust particles could occlude this opening as they accumulate in the orifice fitting. Particles also can affect the leak integrity of the valve, and possibly can damage the valve. Two types of filters are used for this application, frit filters and in-line filters (Figure 2). A variety of models of each type are available; most are of sintered stainless steel and have 2-, 5-, or 7-micron pores. Obviously, the smaller the pores, the less likely are potential problems from airborne particles. EPA Compendium Method TO-14A/15 recommends using a particle filter with 2-micron pores.

Figure 1. A complete sampling train is needed for reliable passive sampling.

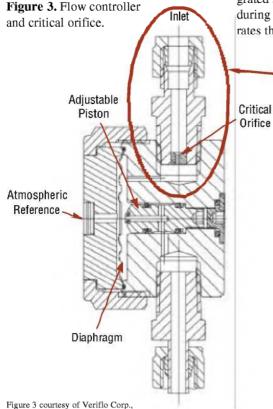




Critical Orifice

The critical orifice (Figure 3) restricts the flow to a specified flow range. In conjunction with the flow controller, this allows the canister to fill at a specified rate over a specified time period. The most common critical orifice design is a series of interchangeable stainless steel 1/4" NPT to 1/4" compression unions, each fitted with a precisely bored sapphire orfice. Each orifice provides a specific flow range (Table 1). Stability over a wide range of temperatures makes sapphire the construction material of choice. Typically during field sampling, the sampling train is subjected to temperature fluctuations that would cause metals to contract or expand, affecting the diameter of the aperture and thereby affecting flow. Sapphire will not expand or contract across any ambient temperature extremes incurred during sampling.

A critical orifice can be used as the sole flow-restricting device, but it cannot ensure uniform flow. The source pressure of the flow changes during sampling, and the flow rate through the orifice also would change, producing an invalid time-integrated sample. It is important that a highly consistent flow rate be maintained during passive sampling. This is accomplished by the flow controller that incorporates the critical orifice.



Outlet

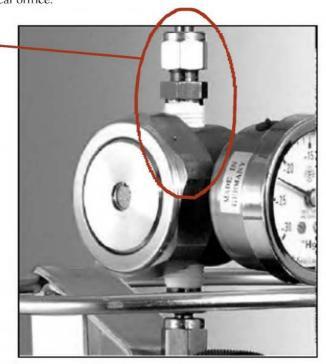


Table 1. Critical orifice diameter vs flow rate.

Orifice Diameter	Flow Rate Range	Canis	ster Volume	/ Samplin	g Time
(in.)	(seem)	1L	3L	6L	15L
0.0012	2-4	4 hr.	12 hr.	24 hr.	60 hr.
0.0016	4-8	2 hr.	6 hr.	12 hr.	30 hr.
0.0020	8-20	1 hr.	4 hr.	8 hr.	20 hr.
0.0030	20-40		2 hr.	3 hr.	8 hr.
0.0060	40-80		1,77	1 hr.	3 hr.

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Flow Controller

The flow controller (Figure 3) maintains a constant sample flow over the integrated time period, despite changes in the vacuum in the canister or in the environmental temperature (Figure 4). In the Veriflo™ Model SC423 XL Flow Controller shown in Figure 3, the critical orifice acts as a flow restrictor, upstream of a constant back pressure. This constant back pressure is established by the balance between the mechanical spring rate of the diaphragm and the pressure differential across the diaphragm. The latter is established by the pressure difference between the atmospheric pressure and the vacuum in the canister and the flow through the critical orifice. The net result is a constant flow.

The critical orifice determines the flow range. The adjustable piston is used to set a specific, fixed flow rate within the flow range. An adjustment to the position of the piston changes the back pressure, which changes the pressure differential across the critical orifice. If the piston is lowered away from the diaphragm, the flow rate will increase. If the piston is raised toward the diaphragm, the flow rate will decrease. This flow controller will accurately maintain a constant flow despite changes in vacuum over a range of 30" Hg to 7" Hg. Flow is constant until the vacuum range of the device is exceeded, making the flow controller unable to maintain the constant pressure differential. In Figure 5, for example, the flow rate is constant from 29.9" Hg to approximately 7" Hg, at which point the flow rate decreases because the flow controller is unable to maintain the proper pressure differential. This control will allow the user to collect approximately 5 liters of sample in a 6-liter canister. This is an extremely important factor in obtaining valid time-integrated samples through passive sampling. We will discuss this point further in the *Field Sampling* section of this guide.

Figure 4. A flow controller will maintain a constant sample flow despite changes in canister pressure or environmental temperature.

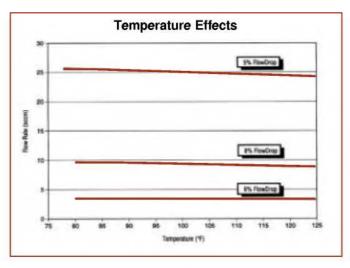
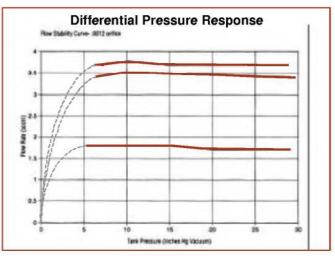


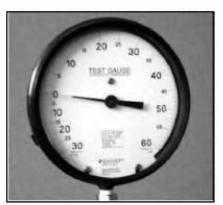
Figure 5. A flow controller will maintain a constant sample flow until it is unable to maintain a stable pressure differential across the critical orfice.



Figures 4 and 5 courtesy of Veriflo Corp., a division of Parker Hannifin Corp.

Figure 6. Conventional vacuum gauge





Vacuum Gauge

A vacuum gauge enables sampling personnel to visually monitor changes in the vacuum in the canister during sampling. If the flow rate changes unexpectedly (e.g., due to a leak or an incorrect setting), the vacuum gauge will indicate a disproportionately high or low vacuum in the canister, and corrective action can be taken (i.e., flow adjusted) in time to ensure a valid sample. This type of vacuum gauge is attached to the sampling train, for use in the field. The vacuum gauge should be of high quality, to ensure that it does not introduce contaminants into the sample. All wetted parts in the vacuum gauge shown in Figure 6 are constructed of stainless steel; the gauge is accurate to within 1% of full scale.

To monitor pressure in the canister before and after sampling, use a more accurate measuring device. Test gauges built by Ashcroft are accurate to 0.25% of full scale. These sensitive gauges should not be used in the field - they typically are wall mounted in the lab. Once used for sampling, a gauge must be cleaned, and should be certified clean. Procedures are described later in this guide.

Canister

The canister is a stainless steel vessel designed to hold vacuum to less than 10 mTorr or pressure to 40 psig. Canisters are available in a range of volumes: 850 mL, 1.0 liter, 1.8 liter, 3.0 liter, 6.0 liter, 15 liter, and 35 liter. The size of canister used usually depends on the concentration of the analytes in the sample, the sampling time, the flow rate, and the sample volume required for the sampling period (Table 1, page 4). Typically the smaller canisters are used for more concentrated samples, such as soil gas collection, 3-liter and 6-liter canisters are used to obtain integrated (TWA) ambient air samples at sampling times of up to 24 hours, and the large 15-liter and 35-liter canisters are used for reference standards. Sampling time will be limited by the combination of canister size and the flow rate at which the sample is to be collected.

A well-designed canister is essential to the success of the sampling project. First, the canister should be made of stainless steel, so the collected sample will not permeate through the vessel wall or degrade due to exposure to light during shipment to the analytical laboratory. Second, the interior surface of the canister should be inert, to reduce the potential for interactions with the analytes in the sample. Third, all canisters involved in a particular application should be of consistent volume, to simplify calculating sample volumes. Finally, the canister should have a high quality valve that resists abuse in the field (e.g., overtightening that potentially could cause leaks). An inferior valve can fail, causing sample loss and incurring replacement costs. It can be more expensive to sample again than to replace a valve. Two types of canisters are available, the difference being the interior surface. The traditional canister is the stainless steel SUMMA® canister. The interior of a SUMMA® canister is electropolished, using a polishing procedure (developed by Molectrics) that enriches the nickel and chromium surface and makes it more inert than untreated stainless steel. The new generation of sampling canister is typified by the SilcoCan[™] canister. Like the SUMMA® canister, the SilcoCan[™] canister is made of stainless steel, and the interior is electropolished, but in an additional step - Silcosteel® treatment - an inert layer is chemically bonded to the interior surface. Silcosteel® treatment makes the surface inert not only for relatively inactive organic compounds, but also for compounds that are reactive with metal surfaces, such as sulfur-containing compounds. Thus, surface deactivation for SilcoCan™ canisters exceeds that for SUMMA® canisters.

Canister Valve

The valve on a sampling canister must be of high quality, with the following characteristics: leak integrity, a metal seat (to eliminate offgassing of seat components

into the sample and memory effects in the seat material), stainless steel wetted surfaces, and a packless design (a completely enclosed system, to ensure no contamination from lubricants or packing material). Various valves are used on various models of canisters; the most commonly used valves are the Nupro 4H4 series metal bellows valve and the Parker diaphragm valve with metal seat. At Restek we incorporate the Parker diaphragm valve on canisters because of its ability to hold vacuum, it's durability and longevity, and a maximum temperature limit (250°C) that is much higher than that for a bellows valve. Further, a Parker diaphragm valve can be rebuilt if it is damaged; it does not have to be replaced.

The connection of the valve to the canister is critical. The connection must be leak tight, to ensure a correct sampling flow rate, but extreme caution must be taken to prevent overtightening the tube compression fittings.

Silcosteel® and Sulfinert™ Treatment

Silcosteel® treatment is a proprietary process, developed by Restek Corporation, through which an inert layer is chemically bonded to a metal surface. The surface produced by this treatment is virtually inert to active compounds. The stainless steel pathway described on pages 3-7 of this guide is sufficient for sampling atmospheres containing only non-reactive compounds, but for reactive compounds the entire sampling pathway should be Silcosteel® treated to eliminate contact between the reactive analytes and the metal surfaces. Silcosteel® treatment can be applied to the interior surfaces of the canister and valve, to ensure an inert sample pathway. If the samples will contain reduced sulfur-containing analytes, an alternative proprietary Restek deactivation process, Sulfinert™ treatment, is the most effective means of deactivating the sample pathway and canister.

III. Preparing the Sampling Train for Use

The sampling train must be prepared in the laboratory before it can be used in the field. The train must be assembled and leak tested, the flow rate must be set, and the train must be certified clean. All of the following information should be documented for the chain of custody for the passive sampling train and the sample collected with it.

Assemble, Leak Test, and Set the Flow Rate of the Passive Sampling Train

Choose the critical orifice according to the sampling period and flow rate you anticipate using (Table 1, page 4). This will ensure an accurate and valid sample. There should be a marking on the outside of the critical orifice fitting indicating the size of the orifice. In a clean environment, assemble the sampling train components as shown in Figure 1 (page 3). It is imperative that you leak test the assembled train. If the sampling train leaks during sampling, the final partial pressure in the canister will not be the desired final partial pressure, making the sample invalid. The most common reason for invalid samples is leaks within the sampling train. There are two ways to leak test the train:

- Pass helium gas through the flow controller and use a sensitive helium leak detector to test for leaks (e.g., Restek Leak DetectiveTM II, cat. #20413).
- 2. Cap the inlet, attach the sampling train to an evacuated canister, open the valve on the canister and evacuate the sampling train.
 - Close the valve and monitor any pressure change in the static sampling train. Leaks of less than 1 mL/min. can be detected in 1-2 minutes.

This is a good practical test - the small internal volume of the passive sampling train, combined with even a small leak, will produce a large change in monitored pressure.

After you are certain the sampling train is leak-free, set the desired sampling flow rate.

Where can I find EPA Air Toxic Methods?

pdf files of US EPA Air Toxic Methods are available at this web address:

http://www.epa.gov/ttn/amtic/airtox.html

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To set the desired flow rate follow these steps:

- Remove the protective cap from the back of the Veriflo™ Flow Controller SC423XL body.
- 2. Connect either an evacuated canister or a vacuum source to the outlet of the sampling train.
- Connect a high quality calibrated flow meter (i.e., mass flow meter, rotameter, GC-type flow sensor; e.g., Restek Flowmeter 6000, cat. #21622) to the inlet of the train.
- 4. Apply vacuum by opening the canister or turning on the vacuum source.
- 5. With a 3mm hex (Allen) wrench, adjust the piston gap screw to achieve the desired flow rate (Table 2). Between adjustments allow the flow to equilibrate for several minutes.
- 6. Replace the protective cap onto the back of the Veriflo™ Flow Controller body.

Table 2. Flow rates for integrated sampling, when using a 6-liter canister and sampling on the flat portion of the flow curve for the flow controller (Figure 5). Collected volume is 4-5 liters (flow = volume in mL / sampling time in min.).

Sampling Pe	riod								
(hours)	0.5	0.75	1	2	4	8	12	16	24
Flow Rate Ra	ange								
(mL/min.)	133-167	89-111	67-83	33-42	17-21	8-10	5.6-6.9	4.2-5.2	2.8-3.5

Cleanliness: Certifying the Sampling Train for Use

US EPA Compendium Method TO-14A/TO-15 requires that the sampling train be certified clean prior to use. Certify the train by passing a humidified, high-purity air stream through the train, concentrating the exit gas on a trap, and analyzing the gas by gas chromatography / mass spectroscopy or other selective detector. For the sampling train to pass certification the analytical system should not detect greater than 0.2ppbv of any target VOC.

The certified sampling train should be carefully packaged in aluminum foil or in a clean container for storage or for shipment into the field. Care in packaging is critical. Careless handling could affect the preset flow rate. When the sampling train is ready for sampling, prepare the canister.

IV. Preparing the Canister for Sampling

Preparing a canister for sampling involves certifying the canister clean, evacuating the canister to final pressure for use, and identifying the canister. All information acquired during these processes is needed for the chain of custody.

Certifying the cleanliness of the canister is important toward ensuring that results reported are solely from the site sampled, and not admixed with residue from a previous site or from contaminated laboratory air. To certify a canister clean, fill the canister with humidified air, pass the air from the canister through an adsorbent trap and analyze the adsorbent for target VOCs by GC/MS or other selective detector. Two US EPA methods discuss canister certification: EPA Compendium TO-12 and EPA Compendium TO-14A/TO-15. To comply with EPA Compendium Methods TO-14A/TO-15, the analytical system should not detect greater than 0.2ppbv of any target VOC. To comply with EPA Compendium Method TO-12 the analytical system, GC/FID, should not detect greater than 0.02ppmvC hydrocarbons. Although

omtech.net.au/pdf/59203 Stiffation of Capister signification in Gapister signification of Capister significant individually. Detailed cleaning



instructions are presented in VIII. Cleaning the Canister.

Some laboratories certify a canister for VOC stability by introducing a low concentration test mixture into the canister and measuring degradation over a specified time period. If the canister meets the specification, it is certified for use. We recommend using such studies to ensure the effectiveness of a canister or group of canisters for a proposed application.

Once the canister is certified clean, evacuate the canister to a final vacuum of 10-50mtorr, using either the canister cleaning system or a clean final vacuum system. This vacuum is critical to ensure the correct amount of sample is collected. Use an accurate test gauge or digital pressure tester to ensure final vacuum has been reached and document the final vacuum reading for the chain of custody. Install a brass plug nut into the canister valve to ensure no contamination can enter the sample pathway during shipment to the field.

Allot an individual identity to the canister, either with a label and serial number or with a bar code.

Some analysts prefer to introduce surrogate standards into the canister prior to sampling. Debate on this practice revolves around theories that there are potential loss issues due to low humidity and inadequate surface passivation by water. Neither Restek chemists nor our consulting experts recommend adding surrogates to the canisters. If you choose to introduce surrogates into your canisters prior to sampling be sure to recheck the vacuum reading for each canister after adding the surrogates, and record the reading.

V. Field Sampling, Using a Passive Sampling Train and Canister

It is important to mention again that the sampling train and canister must be leak tested and certified clean prior to use. To properly begin field sampling, we recommend bringing a "practice" evacuated canister and a flow measuring device with you to the field. Use this canister to verify the flow rate through the passive sampling train prior to using the train to obtain samples of record. To verify the flow rate, connect the passive sampling train to the "practice" canister. Attach a flow meter to the inlet of the sampling train. Open the canister, and measure the flow rate through the sampling train. If the flow rate is within +/- 10% of the flow rate set in the lab, the train is ready to be used on the formal sampling canister. If the flow rate is not within these limits, adjust the flow rate by adjusting the piston gap screw.

When the flow rate is confirmed, record the rate as the canister flow rate for the chain of custody form.

To begin sampling, using the formal sampling canister, follow these steps:

- 1. Remove the brass plug nut from the canister valve.
- If you are using a test gauge, attach the gauge to the canister and record the vacuum reading. If you choose not to use a test gauge under field conditions, record the reading on the vacuum gauge that is part of the passive sampling train.
- 3. Attach the verified passive sampling train to the canister.
- 4. Record the begin sampling time and necessary meteorological data.
- 5. Open the canister valve and begin sampling.
- Periodically check the canister throughout the sampling period to ensure the partial pressure reading is accurate and sampling is proceeding as planned.
- 7. Once the sampling period is complete, close the valve and remove the sampling train. Check the final partial pressure within the canister, using the test gauge or the vacuum gauge in the sampling train.



There are four possible scenarios:

- A. Ideally there will be a vacuum of 7"-4" Hg in the canister.
- B. If more than 7" Hg vacuum remains, less sample was collected than initially anticipated. The sample will be valid, but the detection limit may be higher than expected. You might have to pressurize the canister prior to the analysis, which will dilute the sample, then you will have to use a dilution factor to determine final concentrations of target compounds.
- C. A vacuum of less than 4" Hg indicates the sample might be skewed toward the initial part of the sampling period. This assumption usually is valid because the flow rate through the flow controller will fall once the vacuum falls below 5" Hg (Figure 5, page 5), when the change in pressure across the flow controller diaphragm becomes too small and the flow controller is unable to maintain a constant flow. Although flow was not constant over the entire sampling period, the sample might be usable because sample was collected over the entire interval.
- D. If the ending vacuum is less than 1" Hg the sample should be considered invalid because it will be impossible to tell when the sample flow stopped.
- 8. Record the final partial pressure in the canister and replace the plug nut.

Information that should be acquired at the sampling site includes the start time and interval time, the stop time, atmospheric pressure and temperature and, for ambient sampling, wind direction. Include elevation if it is a factor. These parameters often prove very useful toward interpreting results.

After sampling, the canisters are sent back to the laboratory, where the final vacuum is measured once again, with a test gauge. Using the initial vacuum and final vacuum, the sample volume collected can be determined from Equation 1:

Equation 1:

Sample Volume =

math reference pressure

tinitial pressure - final pressure

pressure change*

x canister volume

tinitial pressure - final pressure

Example: A sample is collected in a 6-liter canister.

Example: A sample is collected in a 6-liter canister. The initial gauge pressure reading when the canister left the lab was 29" Hg vacuum; the final gauge pressure reading when the canister was returned to the lab was 7" Hg vacuum.

Sample volume = $[(29^{\circ}\text{Hg} - 7^{\circ}\text{Hg}) / 29^{\circ}\text{Hg}] \times 6L = 4.55 \text{ liters collected.}$

Table 3	 Final 	vacuum	and	volume	of samp	le collected	l in 6-liter	canister.

Final Vacuum ("Hg)	29"	27"	25"	23"	20"	17"	15"	12"	10"	7"	5"	3"	O"
Sample Volum (liters)	e 0	0.414L	0.83L	1.24L	1.86L	2.48L	2.90L	3.52L	3.93L	4.55L	5.0L	5.38L	6L

It is also a good practice to recheck the flow rate after sampling, because this will affect the sample volume (Equation 2). Laboratories typically allow a maximum deviation of $\pm 10\%$ to $\pm 10\%$ between the initial flow rate and the post-sampling flow rate.

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Equation 2:

Sample Volume = [(initial flow rate + post-sampling flow rate)/2] x sampling time

Example: A flow controller was set at 3.3mL/min. After obtaining a 24 hour sample the flow rate was 3.0mL/min.

Sample volume = $[(3.3 \text{mL/min.} + 3.0 \text{mL/min.}) / 2] \times 1440 \text{ min.} = 4536 \text{mL}.$

VI. Analysis of Collected Samples

Once received by the lab, each canister is identified from the information in the chain of custody report. The final partial pressure is checked to ensure no leaks appeared during transport. It might be necessary to pressurize a canister prior to the analysis; do this by adding humidified nitrogen or air to the canister to a pressure greater than 5 psig or higher, depending on the sample volume needed for analysis or for suitably diluting the sample. The need to dilute is determined by the preconcentrator instrument. Some air preconcentrators can be operated while the canister is under slight vacuum. Check with your instrument manuals, or with the manufacturer, to determine if you must dilute your samples prior to analysis.

Equation 3:

Dilution Factor = (Patter dilution + Plab atmosphere) / (Plab atmosphere - Pbefore dilution)

The dilution factor is calculated from the post-sampling pressure (before dilution), the final pressure (after dilution), and the atmospheric pressure in the laboratory. The factor for converting "Hg to psi = 0.491.

Example: At the end of a sampling period the gauge pressure in a canister was 7"Hg. The canister was pressurized with nitrogen to 14.7psig (1 Atm.).

The dilution factor is $(14.7 + 14.7) / (14.7 - (7 \times 0.491)) = 2.61$

Final Vacuum													
("Hg)	29"	27"	25"	23"	20"	17"	15"	12"	10"	7"	5"	3"	0"
Sample Volume													
(liters)	0	0.414L	0.83L	1.24L	1.86L	2.48L	2.90L	3.52L	3.93L	4.55L	5.0L	5.38L	6L
Dilution Factor													
	63.71	20.37	10.12	8.63	6.02	4.63	4.01	3.34	3.00	2.61	2.40	2.22	2.00

To analyze the sample, withdraw an aliquot of the sample from the canister. For low level ambient air analysis, withdraw 250-500 mL of sample from the canister and concentrate the analytes by using a mass flow controller and a cryogenically cooled trap (e.g., glass beads and/or a solid sorbent). Desorb the concentrated analytes from the trap and deliver them to a cryofocuser, to focus the sample bandwidth prior to introduction onto the GC column. A 60m x 0.32mm ID x 1.0 μ m Rtx*-1 column typically is used for EPA Method TO-14A or Method TO-15 ambient air analysis; an MSD is a common detector. Figure 7 shows a typical TIC spectrum for a TO-14A/TO15 ambient air analysis.

through the frit from the outlet surface toward the inlet surface. After the larger particles are removed sonicate or rinse the filter parts in methanol, then bake the parts in an oven at 130°C to remove any residual organic vapors.

The critical orifice and flow controller can be cleaned in either of two ways. The first method is to disassemble the flow controller and clean all the metal parts with methanol. This will remove any high boiling compounds that have condensed onto the wetted areas of the controller. Heat the cleaned parts in an oven at 130°C to remove residual organic vapors. Do not sonicate in solvent or bake any of the non-metallic parts, such as O-rings; they will be damaged during these steps. Do not rinse the vacuum gauge with methanol. The vacuum gauge may be heated, but do not exceed 80°C; higher temperatures will damage the face and the laminated safety glass lens. Heating to 80°C will not affect the mechanical operation of the spiral bourdon tube in the vacuum gauge.

A less involved method of cleaning the flow controller is to use a heating jacket or heat gun to heat the components of the assembled sampling train, while purging the system with nitrogen. As organic compounds are heated and desorbed from the interior surfaces the nitrogen gas sweeps them out of the sampling equipment.

Preparing the Clean Passive Sampling Train for Re-use

After the sampling train components have been cleaned, reassemble the system, check for leaks, set the desired flow rate, and certify the sampling system clean. Follow the procedures described previously in this guide. Package the clean sampling train to prevent contact with airborne contaminants.

VIII. Cleaning the Canister

Every air sampling canister, whether new or previously used, must be cleaned and certified before it is used for sampling. Some laboratories batch test and certify canisters, in which after cleaning, one canister out of 10 is tested and certified clean. We recommend certifying each canister clean prior to use, however, especially if there is potential for litigation.

For many years there has been much discussion as to what constitutes a proper procedure for cleaning canisters. US EPA Method TO-14A has provided guidance, and in the last 5-10 years many automated commercially available canister cleaning systems have evolved. Unfortunately, because these systems are quite expensive, and some designs have limitations, many analysts design their own systems and methodologies for cleaning canisters. The cleaning procedure described in this section is a practical approach that will ensure canisters are suitably cleaned for ambient air sampling, whether you are using a commercially available cleaning system or a system of your own design. There are minor differences when cleaning SUMMA® or SilcoCan™ canisters. We will discuss these differences in this procedure.

Air Versus Nitrogen

The two gases recommended for cleaning canisters are humidified ultra-high purity air and ultra-high purity nitrogen. The water in the humidified gas hydrolyzes impurities in the canister and, according to theory, will occupy the active sites on the interior surface, displacing the impurities and allowing them to be removed. Air is recommended when oxidation of the interior surface is desired. The oxygen content of air, 21%, is sufficient for this surface oxidation; it is not necessary to use pure oxygen gas. Nitrogen is equally effective for cleaning ambient air canisters, but, of course, nitrogen will not oxidize the surface of the canister.

Free while supplies last!

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air

US EPA TO-Methods on CD-ROM

- Methods T0-1 through T0-17
- Version 1 and Version 2
- Adobe® Acrobat Reader® software TO-Methods on CD-ROM cat.# 24280



Heat or No Heat

Many user-designed canister cleaning systems do not heat the canisters. Typically this does not create a problem when cleaning canisters that are used in ambient air collection, but as a safeguard we recommend heating the canisters during the cleaning process. Compounds collected in most ambient air samples are in the low ppbv range, and can be removed from a canister by multiple cycles of pressurization with humidified air or nitrogen followed by evacuation. If there are higher concentrations of contaminants in the canister, however, heat might be required to clean the canister satisfactorily.

Be aware that adding heat and humidified gas to a canister potentially can create a steam pressure vessel. Some commercial cleaning systems incorporate a pressure release valve to ensure the pressure does not exceed the pressure rating of the canisters.

A heating option can be added to a user-designed canister cleaning system by using an oven, heat bands, insulated jackets, or an infrared source.

Oven

Some canister cleaning systems are incorporated within an oven. The supply line for the humidified air stream and the line to the vacuum system are plumbed directly into the oven. In this arrangement the entire canister, including the valve, will be heated, and this will help remove contaminants if both the valve and the canister are dirty. Typically, when using heat, it is helpful to create steam from the humidified air stream. An oven temperature of at least 120°C is required, but higher temperatures often are used. Remember that heat can shorten the lifetime of the Nupro valve on a SUMMA® canister (see step 3 in *Cleaning Method*, below).

Space is a concern with oven systems. Most commercial ovens are not very large and this restricts the number of canisters that can be cleaned at one time. However, clean-up times are shorter with heat than without heat, so more cleaning cycles can be completed in a week.

Heat Bands

A band heater placed around the equator of the canister typically will be capable of heating the canister to approximately 130°C. There is a heat gradient, and the valve might only receive radiant heat (approximately 70-100°C). In most sampling situations, however, this lower temperature should be sufficient for effectively removing contaminants from the valve.

Insulated Heat Jackets

Insulated heat jackets can be obtained to surround and heat each canister. These jackets typically have a silicone or Teflon®-coated fiberglass fabric exterior and a fiberglass insulation interior. Some operate at a fixed temperature; others can provide variable temperature, up to 400-500°F. Commercially available designs do not encompass the valve area, however, so a jacket would have to be customized to include the valve.

Infrared Heat

An infrared heating system includes an infrared heat source and a reflective panel similar to the cylinder drying rack on a gas cylinder system. The infrared source and the reflective panel are placed on opposing sides of the canisters. Infrared rays from the source heat the canisters; rays that pass the canisters strike the reflective panel and heat the canisters from the opposing side.

The Cleaning System

The cleaning system must provide a humidified air stream and include a good vacuum source, a cold trap to collect impurities during cleaning, and accurate gauges to read vacuum and pressure. A heat source is optional, but is highly recommended in some circumstances, as discussed above. The system can be designed to clean 4 to 24 six-liter canisters. Figure 8 is an example of a "homemade" system designed to clean 24 six-liter canisters. This design does not employ heat, but a heater easily can be added (see *Heat or No Heat*, above). It provides a humidified air stream to all canisters and the roughing pump on the bottom shelf is the vacuum source. This system is computer operated to minimize labor, but this is not necessary.

Cleaning Method

- 1. Connect all canisters to the cleaning system, then release any pressure within any of the canisters. Put the system under vacuum, to evacuate the canisters. US EPA Method TO-14A/15 recommends evacuating the system to 50 mTorr for 1 hour, but a reduced pressure of 23-25" Hg is sufficient for general cleaning.
- 2. After the canisters have been under vacuum for approximately 1 hour, pressurize the canisters with humidified air or nitrogen. Pressurization will dilute the impurities and the moist air will hydrolyze them. Pressurize canisters to 5 psig if they will be heated or to 30 psig if they will not be heated. Proceed to step 3 when the system has equilibrated at the designated pressure.
- 3. Heat the pressurized canisters to 120 250°C, depending on the type of canister being cleaned. Do not allow the temperature of a SUMMA® canister to exceed 155°C, because the Nupro valve it employs has Viton® O-rings and requires greases that cannot be exposed to high temperatures. Many commercial cleaning systems avoid this problem by ensuring the valve is not within the heated zone. The canister below the valve is heated but the valve receives only radiant heat. In contrast, the Parker diaphragm valve in a SilcoCan™ canister is far less heat sensitive, allowing the canister to be cleaned at temperatures up to 250°C, to help remove less labile impurities.

Heat the canisters filled with humidified air for at least 1 hour.

- 4. Re-evacuate the canisters to remove the desorbed impurities. Allow the canisters to equilibrate for 1 hour.
- 5. Determine if the canisters have been cleaned effectively by following the procedure in *Certifying the Canister*, below. US EPA methods recommend testing every canister until a reliable procedure is developed.

Repeat steps 1-5 as necessary; the number of cycles will be determined by how dirty the canisters are and how easily they are cleaned. We recommend developing a cleaning procedure that matches your specific sampling procedure, by testing the canisters for cleanliness after each cycle and determining the number of cycles necessary for proper cleaning. If the canisters are not heated, the number of cycles required to clean the canisters might be higher.

6. Once a canister is clean, prepare it for collecting a sample by evacuating it to 10-50 mTorr. If your system is leak-tight, you can do this by using a roughing pump, but many commercial systems include a molecular drag pump to reach final vacuum quickly.



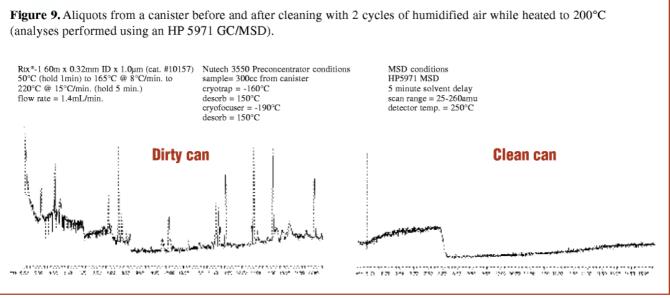
Figure 8. User-designed system for cleaning 24 six-liter canisters.

Pressure C	Conversion Tab	ole	Multiply uni the conversi	ts in the left-mos on factors listed	st column by to the right.		0.068 = 0.68atm 29.5300 = 295.300 inch	es Hg
Pressure	PSI	atm	kg/cm²	torr	kPa	bar	inches Hg	
PSI =	1	0.068	0.0703	51.713	6.8948	0.06895	2.0359	
atm =	14.696	1	1.0332	760	101.32	1.0133	29.921	
kg/cm ² =	14.223	0.967	1	735.5	98.06	0.9806	28.958	
torr =	0.0193	0.00132	0.00136	1	0.1330	0.00133	0.0394	
kP <i>a</i> =	0.1450	0.00987	0.0102	7.52	1	0.0100	0.2962	
bar =	14.5038	0.9869	1.0197	751.88	100	1	29.5300	
in. Hg =	0.49612	0.0334	0.0345	25.400	3.376	0.03376	1	

IX. Certifying the Canister

We recommend certifying canisters for both cleanliness and for analyte stability. To certify a canister clean, pressurize the canister to 14.7 psig with humidified ultra-high purity air or nitrogen after it has gone through the cleaning cycles. The humid air or nitrogen stream must be certified clean before it can be used for canister certification. Analyze an aliquot of the canister content by GC/MS or GC/FID/ECD. US EPA Method TO-14A/15 specifies a canister must contain less than 0.2 ppbv of any target VOC compound (Figure 9); EPA Method TO-12 specifies less than 0.02 ppmC, as detected by GC/FID. If a canister does not meet specification, it must be re-cleaned and re-tested for certification.

To certify a canister for analyte stability, introduce a low working concentration of a characterized test mix into the canister. Analyze an aliquot of the contents of the canister immediately after introducing the test mixture and at periodic intervals. We recommend monitoring for changes for a minimum of 2 weeks, or for a timeframe similar to your anticipated holding period. Responses should not decrease more than 20% over this period. Commercially available standards are available for stability testing, but we recommend you make your own test mixture that is comparable to the target compound list that the canister will hold. For example, if you are analyzing sulfur compound content in ambient air, prepare a sulfur-specific test mix and evaluate the canister's performance for sulfurs. Maintain a log sheet for each canister, and record the test results and certification. This will be a permanent record for each canister. Some labs certify canisters for certain compounds and use a canister only for this specific application.



X. Conclusion

A well designed and properly prepared passive sampling system helps ensure accurate, useful information is obtained from an air sampling project. In this guide, we describe the components of the system, procedures for assembling the system and preparing it for sampling, and the sampling procedure. Cleaning system options and procedures for cleaning a used sampling train and canister for certification prior to a subsequent sampling also are presented. The following section describes Restek products designed to help collect and analyze air samples.

Products

SilcoCan™ Silcosteel™-treated Canisters

Ideal for TO-14A/TO-15 and low-level (1ppb-20ppb) reactive sulfur compounds

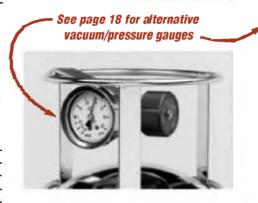
Feature		Benefit			
High-purity, ² / ₂ -turn valve with stainless steel valve diaphragms	No sample adsorption, for more accurate re- easy to use.				
Vacuum/pressure gauge (optional).		Indicates internal conditions.			
Variety of sizes available.	Meet extensive range of sampling needs.				
Temperature stability to 250°C.	Can be cleaned at higher temperature, producing a cleaner can.				
Silcosteel® valve available (add suffix "-650" to cat, #).		A completely passive sample pathway ensures sample stability.			
Size	qty.	cat.#			
1L	ea.	24112			
3L	ea.	24113			
6L	ea.	24114			
15L	ea.	24115			











www.restekcorp.com

SilcoCan™ Canisters Feature a Silcosteel® Valve

Many sample components, such as sulfur compounds and certain polar organics, react with metal surfaces. This is easily remedied by requesting a Silcosteel* valve for your SilcoCan™ canister. All valve parts that come in contact with the sample are passivated for unsurpassed inertness.

size	qty.	cat.#
1L	ea.	24112-650
3L	ea.	24113-650
6L	ea.	24114-650
15L	ea.	24115-650

New Generation Replacement Diaphragm Valves

If you need to replace the valve on your SilcoCan™ canister, use this Parker Hannifin diaphragm valve.* Each valve is helium leak-tested to 4 x 10°cc/sec. The all-stainless steel construction eliminates contamination while withstanding temperatures from -100°C to 250°C. Each valve is equipped with a compression outlet fitting and an indicator plate to determine open or closed position.

Descriptien	qty.	cat.#	
Silcosteel® Replacement Diaphragm Valve	ea.	24223	
Replacement Diaphragm Valve	ea.	24122	_

^{*}All canisters are originally equipped with high-quality diaphragm valves made by Parker Hannifin Corporation.

SilcoCan™ Canister with Vacuum/Pressure Gauge

- · Monitor pressure inside the canister.
- · Accurately measure from 30" Hg vacuum to 60psig pressure (other gauges available).
- · Gauge fully protected by canister frame.
- · Gauges available in three reading ranges (see page 18).

size	qty.	cat.#
1L	ea.	24116
3L	ea.	24117
6L	ea.	24118
15L	ea.	24119

^{**}Silcosteel* valves are available for SilcoCan™ canisters at an additional cost. Add the suffix number "-650" to the catalog number if ordering a Silcosteel valve.



Quickly confirm vacuum or pressure using a high-quality gauge mounted on your TO-Can" canister.



Alternative Vacuum/Pressure Gauges

To order a different vacuum/pressure gauge*** for the canister, add the appropriate suffix number to the canister catalog number

Gauge	Suffix
30" Hg/15psi	-651
30" Hg/30psi	-652

^{***}No price difference for these substituted gauges.

TO-Can™ Air Monitoring Canisters

Optimized for EPA Methods TO-14A and TO-15 (SUMMA® canister equivalent)

Feature		Benefit	
High-purity, metal to metal seat, %-turn valve with stainless steel diaphragms.		No sample adsorption, for more accurate results easy to use.	
Vacuum/pressure gauge (optional).		Indicates internal conditions.	
Variety of sizes available.		Meet a variety of sampling needs.	
Temperature stability to 250℃.	Higher temperature cleaning saves time.		
O-Can™ Canisters size	qty.	cat.#	
1L	ea.	24150	
3L	ea.	24152	
6L	ea.	24153	
15L	ea.	24154	

TO-Can™ Canister with Vacuum/Pressure Gauge

- Continuously monitor pressure inside a TO-Can[™] canister.
- Accurately measure from 30" Hg vacuum to 60psig pressure (other gauges are available).
- · Gauge fully protected by the canister frame.
- Gauges available in three reading ranges.

TO-Can™ Canisters with Vacuum/Pressure Gauge

size	qty.	cat.#
1L	ea.	24155
3L	ea.	24156
6L	ea.	24157
15L	ea.	24158



TO-14A Internal Standard/Tuning Mix

bromochloromethane chlorobenzene-d5 4-bromofluorobenzene 1.4-difluorobenzene In nitrogen, 104 liters @ 1800psig

1ppm	100ppb
34408 (ea.)	34425 (ea.)

TO-14A GC/MS Tuning Mix

4-bromofluorobenzene In nitrogen, 104 liters @ 1800psig

1ppm	100ppb
34406 (ea.)	34424 (ea.)

TO-14A Calibration Mix (39 components)

henzene halocarbon 12 bromomethane carbon tetrachloride chlorobenzene chloroethane chloroform chloromethane 1,2-dibromoethane 1,2-dichlorobenzene 1,3-dichlorobenzene 1 4-dichlorohenzene 1,1-dichloroethane 1,2-dichloroethane 1.1-dichloroethene cis-1,2-dichloroethene 1,2-dichloropropane cis-1,3-dichloropropene trans-1,3-dichloropropene ethylbenzene

halocarbon 114 hexachloro-1.3-butadiene methylene chloride styrene 1,1,2,2,-tetrachloroethane tetrachloroethene toluene 1,2,4-trichlorobenzene 1.1.1-trichloroethane 1,1,2-trichloroethane trichloroethene 1,2,4-trimethylbenzene 1,3,5-trimethylbenzene vinyl chloride m-xylene o-xylene p-xylene

halocarbon 113

In nitrogen 104 liters @ 1800nsig

1ppm	100ppb
34400 (ea.)	34421 (ea.)

For a complete product listing refer to our air monitoring catalog (lit. cat. #59661A) or the Restek general catalog (lit. cat. #59662)

TO-15 62 Component Mix

(62 components)

acetone henzene benzyl chloride** bromoform bromomethane bromodichloromethane 1,3-butadiene 2-butanone (MEK) carbon disulfide**
carbon tetrachloride chlorobenzene chloroethane chloroform chloromethane cyclohexane dibromochloromethane 1.2-dichlorohenzene 1,3-dichlorobenzene 1.4-dichlorohenzene 1,1-dichloroethane 1,2-dichloroethane 1,1-dichloroethene cis-1,2-dichloroethene trans-1,2-dichloroethene 1,2-dichloropropane cis-1,3-dichloropropene trans-1,3-dichloropropene 1,4-dioxane ethanol** ethyl acetate ethyl benzene ethyl dibromide

4-ethyltoluene Freon® 11 (trichlorofluoromethane) Freon® 12 (dichlorodifluo romethane) Freon® 113 (1,1,2-trichlorotrifluo-Freon® 114 (1.2-dichlorotetrafluoroethane) heptane hexachloro-1,3-butadiene hexane 2-hexanone (MBK) 4-methyl-2-pentanone (MBK) methylene chloride methyl tert-butyl ether (MTBE) 2-propanol propylene styrene 1,1,2,2-tetrachloroethane tetrachloroethene tetrahydrofuran toluene 1,1,1-trichloroethane 1.1.2-trichloroethane trichloroethene 1,2,4-trichlorobenzene 1,2,4-trimethylbenzene 1,3,5-trimethylbenzene vinyl acetate vinyl chloride m-xylene o-xylene

In nitrogen, 104 liters @ 1800psig

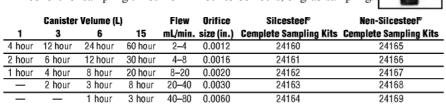
1ppm	100ppb	
34436 (ea.)	34437 (ea.)	

^{**}Stability of these compounds cannot be guaranteed.

Improved Passive Air Sampling Kits*

Better Performance at a Better Value

- · Improved design eliminates leaks at the filter.
- Silcosteel®-treated components ensure a very inert surface.
- · Excellent for sampling times from 1 hour to 60 hours, or grab sampling.



^{*}Air sampling canisters sold separately.

Replacement Orifices

Use these orifices to change the flow range for alternative sampling times. These orifices are interchangeable with Veriflo™ 423XL orifices.

Flew (sccm)	Orifice size (in.)	Silcesteel® cat.#	Nen-Silcesteel® cat.#
2–4	0.0012"	24233	24245
4-8	0.0016"	24234	24246
8–20	0.0020"	24235	24247
20-40	0.0030"	24236	24248
40-80	0.0060"	24237	24249

Replacement Frits

For use in critical orifice fitting. Includes washers.

Descriptien	qty.	cat.#
Silcosteel® Replacement Frit	3-pk.	24171
Non-Silcosteel® Replacement Frit	3-pk.	24170

Veriflo™ Flow Controllers

Veriflo ** SC423XL flow controllers are offered in a Silcosteel* and a non-Silcosteel* version. The flow device is available with or without a critical orifice.

Flew	Orifice	Silcesteel®	Nen-Silcesteel®
(sccm)	size (in.)	cat.#	cat.#
2–4	0.0012"	24255	24260
4–8	0.0016"	24256	24261
8–20	0.0020"	24257	24262
20-40	0.0030"	24258	24263
40-80	0.0060"	24259	24264
	no orifice	24238	24239

7µm In-Line Filter

This 316 stainless steel filter is designed to collect particles larger than 7 microns. We offer a Silcosteel version and a non-Silcosteel version.

Descriptien	qty.	cat.#
Silcosteel® 7µm In-Line Filter	ea.	24265
Non-Silcosteel® 7µm In-Line Filter	ea.	24266

2-Inch Vacuum Gauge

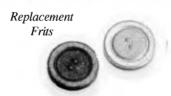
Restek's 2-inch vacuum gauge is a high-quality gauge that incorporates 316 stainless steel wetted surfaces.

Description	qty.	cat.#
¹/₅" NPT	ea.	24269
1/4" NPT	ea.	24270

Buy only the parts you need!

Frit filter and critical orifice





Veriflo™ SC423XL flow controller



Silcosteel® 7µm in-line filter



Silcosteel* tee



High-quality vacuum gauge

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