

INSTRUCTIONS FOR EPA METHOD-25 FIELD SAMPLE COLLECTION USING AN M25LAB HEATED PROBE AND FILTER ASSEMBLY

Introduction: Contact us to order equipment. All VOC sources are not created equal. You will be asked to provide a description of the process and source as well as estimates for the stack gas temperature, CO₂ and moisture content, expected VOC concentration range (<100, 100-1000, 1,000-10,000, >10,000 ppmC) and the most likely organic compounds present. Based on this information, we will supply you with the equipment necessary to properly collect and handle the samples. Step by step instructions for collecting samples using our equipment, including EPA-Approved alternate filter holders, are listed below. ***Field data and Chain of Custody sheets are attached for you to copy and use.***

A. Leak-check the Method-25 sample tank.

1. We ship all sample tanks under vacuum (<10 mm Hg), ready for leak check and use, unless requested otherwise. Each tank is equipped with a shutoff valve and a male Swagelok QF-4 quick-connect (QC) fitting. The tank absolute pressure and temperature (P/T) and the time and date of the readings are recorded on a wire tag attached to each tank. This acts as the first P/T point in the required tank leak-check ***Be sure to place tanks in an area where their temperature can equilibrate with the surroundings and remain stable prior to measuring the tank pressure and surface temperature.***
2. ***AFTER the tank temperature equilibrates with its surroundings,*** tape a thermocouple to the side of the tank and allow a few minutes for the temperature reading to stabilize.
3. Connect the evacuated sample tank to your leak-free vacuum manifold. Evacuate the manifold and tank QC to <2 mm Hg. Be sure to use a calibrated digital manometer for all pressure readings. An absolute manometer is preferred over a relative manometer. If you use a relative manometer, you will need to record an accurate station pressure (barometric pressure corrected for elevation) with each tank pressure reading. Although Method 25 allows the use of a mercury –filled manometer, DUE TO SAFETY CONSIDERATIONS, WE DO NOT RECOMMEND THE USE OF MERCURY (HG) MANOMETERS.
4. Close the valve to the vacuum pump, and then open the valve on the tank. The absolute pressure in the tank should be <10 mm Hg. Record the tank pressure and tank temperature on your field data sheet and on the wire tag attached to the tank.

If the tank pressure is >10 mm Hg, evacuate the tank to <10 mm Hg, close the tank valve and detach it from the vacuum manifold. Wait one hour or more, then reattach the tank to the vacuum manifold and check its pressure as described above. If the pressure has not changed, the tank is ready to use. If the tank pressure has increased over an hour, label the tank's wire tag with "LEAK" and place the tank in the return shipping crate. Do not use a leaky tank to collect a sample. Use one of the spare sample tanks provided.

B. Assemble the Method-25 sample train with a heated probe/filter.

5. Place a leak-checked sample tank and a half-gallon plastic insulated container in a standard milk crate. They should fit snugly. Attach a rope to the crate if needed.
6. Place the controller on top of the sample tank and couple the QC. Attach the heated probe/filter umbilical to the controller module. First, carefully attach the multi-pin electrical connectors to the controller and the

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probe, then plug in the thermocouple to the “Filter” plug on each end, then plug in the “Probe” thermocouple on each end, then connect the 1/8” sample line fitting to the controller, then connect the purge line QC to the controller and the purge line fitting to the purge valve on the probe. You will have to push back the outer sleeve on the purge port female QC to get the purge line male QC to seat properly.

7. Remove the plastic seal and wire tag from a sample trap and place it in the plastic insulated container. Be sure to place all caps and plugs removed from the trap in a clean container. **Do not reuse a cap or plug that has been contaminated (e.g., inadvertently dropped or placed on a dirty surface or in your pocket).** If this happens, use one of the clean spare caps and plugs provided. Connect the longer 1/8” Teflon line from the controller umbilical to the trap outlet fitting. Snug the fitting about 1/8 to 1/4 turn past finger tight. **Do not over-tighten or damage the fittings. This could result in the loss of the sample.**
8. If the stack gas temperature is ~500°F or lower, you are finished assembling the sample train. If the stack gas temperature is >500°F, remove the probe tip and attach a 15” long narrow-bore 1/8” O.D. inconel probe extension to the probe using a Swagelok 1/4” to 1/8” union. When collecting a sample, only the probe extension is placed in the hot stack with the heated probe placed in the port, or just outside the port, as needed to maintain the probe gas exit temperature at >265°F.

C. Leak Check the Method-25 sample train with heated controller.

9. Note that you are also checking the leak rate of your vacuum manifold when you perform the leak check. It is necessary to assure the integrity of the manifold before proceeding with the sample train leak check. So, first leak check your vacuum manifold and repair if needed, then proceed to step 10.
10. Attach a clean Swagelok or other appropriate fitting supplied by us to the end of the probe or probe extension. Never attach a dirty fitting to the sample train.
11. Attach a 1/4” line from your vacuum manifold to the fitting.
12. Open the sample (glass) rotameter valve several turns. Turn both purge valves (one on the controller and one on the probe) to the “OFF” position.
13. Turn the controller power on and allow the probe and filter temperature to stabilize. This will take ~6 minutes.
14. Next, open the valve to the vacuum manifold and evacuate the sample train to <10 mm Hg (<13 mbar or <0.4 in. Hg). It should take less than 5 minutes to pump down the assembled sample train.
15. When the sample train pressure has been <10 mm Hg for several minutes, close the valve to your vacuum pump and properly shut off the pump. (Most vacuum pumps require venting to atmosphere when stopping or starting to avoid drawing pump oil into the vacuum line.)
16. Wait one minute for the pressure in the assembled sample train to equilibrate, and then record the starting pressure and time on the field data sheet.
17. Wait ten minutes, and then record the ending pressure and time on the field data sheet. The allowed 1% leak rate for the assembled sample train with heated controller and probe (88 ml total volume) at standard atmospheric pressure (1013 mbar) and a sample rate of 80 ml/min is:

$$\begin{aligned}\text{Allowed pressure increase} &= (0.01) (80 \text{ ml/min}) (10 \text{ min}) (1013 \text{ mbar}) / (88 \text{ ml}) \\ &= 92 \text{ mbar (or 69 mm Hg, or 2.7 in. Hg)}\end{aligned}$$

The volume of your vacuum manifold will decrease the allowed pressure increase proportionally. Add the volume of your manifold to 88 ml in the above equation, substitute the station pressure, and calculate the allowed pressure increase for your particular assembly. Record this calculation and result on the field data sheet. In practice, if a pressure increase of >50 mbar is noted, it's best to find the leak. Always start by snugging the 1/8" nuts on the trap line to trap body connection.

18. If the sample train does not pass the leak check, snug all connections, and retest. *Be careful not to over-tighten or damage fittings. This could result in the loss of the sample.* If the system still does not pass, replace the trap with one of the spare traps provided and try again. (Always make sure your vacuum manifold is not leaking.)
19. After a successful leak check, close the glass rotameter needle-valve, then ***slowly remove the leak-check fitting from the probe tip and allow ambient air to fill the system. Do not relieve the system vacuum any other way.***

D. Collect a Method-25 VOC sample.

20. Add dry ice to the insulated container holding the trap at least 30 minutes prior to the start of sample collection. ***Record the sample location, run number, trap number and tank number on the field data sheet and on the wire tags attached to the trap and tank.***
21. Position the sample train near the stack. Make sure the tank valve and glass rotameter needle-valve are closed, both purge valves are in the "ON" position, the controller power is on, and the probe and filter temperatures are stable at >265°F and 250 ±5°F, respectively.
22. Next, place the heated probe or the probe extension (as appropriate for your sample) into the stack such that the probe is perpendicular to the duct or stack axis; locate the probe tip at a preselected point of average velocity facing away from the direction of gas flow. Be sure to seal the port using aluminum foil or clean ceramic-fiber cloth, as needed (not rags!!).
23. Purge the system with stack gas at ~100 ml/min for at least 10 minutes. Then, turn off the purge pump, close the plastic rotameter needle-valve, and turn both purge valves to the "OFF" position. ***These settings are critical in order to assure that stack gas is pulled through the trap during sampling.***
24. Open the valve to the tank, record the tank starting vacuum indicated as inches of Hg by the controller gauge. Refer to the rotameter calibration chart displayed beneath each rotameter to select the proper setting, then open the rotameter needle valve until the desired setting is reached. A sample collection rate of ~80 ml/min is ideal for most samples. Use a higher sample rate of 100 ml/min for low concentration (<200 ppmC) VOC samples. Use a lower sample rate of 60 ml/min for high concentration (<10,000 ppmC) VOC samples.
25. Record the tank vacuum, rotameter flow rate, filter temperature and probe temperature every 5 minutes on the field data sheet for the duration of sample collection period.
26. Method-25 sample duration is at least one hour. At the conclusion of sample collection, close the glass rotameter needle valve; record the final tank vacuum from the controller gauge, then close the tank valve. Remove the sample train from the stack and ***transport the sample train to a clean location for sample recovery.***

E. Recover and Transport the Method-25 VOC sample.

27. Disconnect the sample trap line from the probe/filter assembly and seal the trap tip with a clean Swagelok plug.
28. Disconnect the controller line from the trap outlet and cap the trap fitting with a clean Swagelok cap.
29. Coil the trap line NEATLY in a similar fashion to the “as received” sample traps, then remove the trap from dry ice. ***Use the wire tag to bundle the trap and line together.*** DO NOT TANGLE THE TRAPS AND LINES!
30. Place the bundled trap in the large plastic Ziplok bag provided, place in an insulated cooler, and add dry ice on top.
31. Disconnect the controller from the tank. Place the tank in an area where its temperature can stabilize.
32. Measure the final tank pressure and temperature as described above in steps 2-4 except the manifold pressure should be adjusted to approximate the sample tank ending pressure before closing the vacuum valve and opening the tank valve. ***Record the values on your field data sheet and on the wire tag on the tank, and on the Chain of Custody Form.*** Replace the silicon rubber cap on the tank QC fitting.
33. Record the approximate moisture, CO₂ and VOC concentrations for each run on each source on the Chain of Custody form.
34. Place the tanks in the return shipping container. Use the original packing materials and methods to protect the tanks. Tanks can be returned by ground or air.
35. **Ship the traps overnight express or second day air in our return shipping cooler or equivalent with *at least 30 lbs. of dry ice on top of the traps. Do not put any other equipment in the dry ice cooler. Only traps containing samples or blanks are to be in the cooler.***

If you use your own cooler, we recommend the 20-quart Igloo MaxCold plastic cooler, or the Coleman 5-day style cooler. We cannot start your sample analyses until we have both tanks and traps in our possession. Put a copy of the chain of custody sheet in each package. Normal turnaround time is 10 working days. Be sure to insure the samples and equipment to cover damage or loss.

36. Note that all equipment is provided for up to 15 days, including transit times, at no additional charge. Rental fees may apply after 15 days according to the current price list and contract. Call us if your job is delayed, we may be able to waive some or all rental charges.

HEATED METHOD-25 FILTER CHANGEOUT PROCEDURE

Filters should not be used for more than three test runs on a single source. If PM loading is high, it may be necessary to change the filter after each test run. Always change the filter between emission sources. Follow the instructions below to change the filter quickly (< 1 minute) and easily. It is not necessary to cool the filter block before changing filters. ***For safety always turn the heater power off and unplug the unit before changing the filter.***

- (1) For safety always turn the heater power off and unplug the unit before changing the filter. You must be in a dry environment when changing the filter (e.g. not in the rain!). Remove the filter housing plug with a 9/16” wrench, and then remove the used filter cartridge using the custom T-handle extraction tool. Insert the tool with the cross-bar aligned with the grooves on the back of the filter cartridge. Push the blue button in to “set” the tool, and release the button to “lock” the tool onto the filter cartridge. Once inserted into the back slot of the filter cartridge, GENTLY rotate the t-handle tool clockwise slightly until it stops. Then pull the filter cartridge straight out using the t-handle. Once out, press the blue-colored release button on the top of the t-handle tool.

- (2) When uncovering a clean filter cartridge for use, grasp the spiral end of the aluminum foil wrapper with a clean, ungloved hand and pull the folded foil off the other end. Attach the T-bar insertion tool and then remove the rest of the aluminum foil while holding the filter cartridge by the T-bar tool.

- (3) Next, insert the clean filter cartridge into the filter block using the custom insertion tool. Align the notch straight up-and-down. Use the probe handle as a guide. BE GENTLE! The locking notch MUST face the top of the filter block. This bayonet style notch will latch onto the filter thermocouple. DO NOT USE FORCE. Once inserted, rotate the filter cartridge counterclockwise very slightly until it stops. If the notch has not engaged the latch, the filter cartridge will rotate freely until the notch engages the latch. To test the lock, try to pull the filter cartridge out GENTLY. If it does not budge, it’s locked-in properly. Release the insertion tool from the filter cartridge by pushing the blue-colored button on the t-handle. Re-install the filter housing plug hand tight.