

INSTRUCTIONS FOR EPA METHOD-25 FIELD SAMPLE COLLECTION USING A M25Lab UN-HEATED PROBE AND IN-STOCK FILTER ASSEMBLY

Introduction: Contact *M25Lab* 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 Method 25 samples.

Method 25 at 6.1.1 and at 6.1.3, includes the following instruction:

“Note: If it is not possible to use a heating system for safety reasons, an unheated system with an in-stack filter is a suitable alternative.”

This alternative is not subject to approval by the Administrator. It’s your call. Be safe. No VOC sample is worth risking life, limb or property.

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

1. *M25Lab* ships all M25 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 micro-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 barometric pressure with each tank pressure reading. Although M25 allows the use of a mercury-filled manometer, DUE TO SAFETY CONSIDERATIONS, *M25Lab* DOES 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 an un-heated probe and In-stack filter.

5. Place a 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 quick-connect.
7. Remove the plastic seal and wire tag from a Method-25 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). Connect the 1/8" Teflon line from the controller to the trap outlet fitting. Use a 5/8" wrench and a 7/16" wrench to attach this fitting. Snug the fitting about 1/8 to 1/4 turn past finger tight. *Do not over-tighten or damage this fitting. This could result in the loss of the sample.*
8. Connect the 1/8" stainless steel M25 sample trap line to the back of an in-stack filter using a 5/8" and 7/16" wrench. *Do not over-tighten or damage this fitting.*

If the stack gas temperature is 500°F or lower, you are finished assembling the sample train. The entire in-stack filter will be placed in the stack to collect a sample.

If the stack gas temperature is >500°F, attach a 15" long narrow-bore 1/8" outside diameter Inconel probe extension to the in-stack filter. When collecting a sample, only the probe extension is placed in the hot stack with the "in stack filter" placed in the port, or just outside the port, as needed to maintain the filter holder temperature at ~250°F. *M25Lab* recommends that you attach a micro thermocouple to the outside of the filter holder to monitor and record its temperature during sampling (not required by M25).

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

9. Attach a 1/4" line from your vacuum manifold to the in-stack filter tip, or to the tip of the probe extension, as appropriate, using an Ultra-Torr or other appropriate clean fitting supplied by *M25Lab*. If using an Ultra-Torr fitting, finger tighten only. Never attach a dirty fitting to the M25 sample train.
10. Open the sample (glass) rotameter needle-valve several turns.
11. Next, open the valve to your vacuum manifold and evacuate the sample train to <10 mm Hg (<13 mbar). *M25Lab* recommends that you evacuate the train to <2 mm Hg for this application. *It will take several minutes to pump down the assembled sample train. Be patient.*
12. When the sample train pressure has been <10 mm Hg (<13 mbar) for several minutes, close the valve to your vacuum manifold and disconnect the vacuum pump from the manifold.
13. 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.
14. Wait ten minutes, and then record the ending pressure and time on the field data sheet. The allowed 1% leak rate for the assembled *M25Lab*-M25 sample train with un-heated controller and in-stack filter (95 ml total

volume) at atmospheric pressure (1013 mbar) and a sample rate of 85 ml/min is: Allowed pressure increase with unheated *M25Lab* controller at one atmosphere pressure is:

$$\begin{aligned} &= (0.01)(85 \text{ ml/min})(10 \text{ min})(1013 \text{ mbar}) / (95 \text{ ml}) \\ &= 91 \text{ mbar} = 68 \text{ mm Hg} = 2.7 \text{ in. Hg} \end{aligned}$$

The volume of your vacuum manifold will decrease the allowed pressure increase proportionally. Add the volume of your manifold to 95 ml in the above equation, substitute the actual local barometric 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.

15. 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 spares provided and try again. After a successful leak check, close the glass rotameter needle-valve, then disconnect the vacuum line from the probe tip and allow ambient air to fill the system.
16. After a successful leak check, close the glass rotameter needle-valve, then disconnect the vacuum line from the probe tip and allow ambient air to fill the system.

D. Collect a Method-25 VOC sample.

17. Add dry ice to the insulated container holding the M25 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.
18. Position the sample train near the stack. Make sure the glass rotameter needle-valve is closed. Place the in-stack filter 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 in-stack filter or probe extension tip at a preselected point of average velocity facing away from the direction of gas flow. For high-temperature sources, place the probe extension in the stack and place the in-stack filter assembly in the port, or just outside the port, as needed to maintain the filter temperature at ~250°F. Be careful not to let the probe tip touch any surfaces. Be sure to seal the port using aluminum foil or clean ceramic-fiber cloth, as needed (not rags!!).
19. To start sample collection, open the valve to the tank, and 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, and then open the rotameter needle valve until the desired setting is reached. A sample collection rate of ~80 ml/min is ideal for most M25 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.
19. Record the tank pressure and rotameter setting every 5 minutes on the field data sheet. If available, record the filter holder temperature as well (not required by M25).
20. 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 pressure, and then close the tank valve.

E. Recover the Method-25 VOC sample.

22. Transport the M25 sample train to a clean location for sample recovery.
23. Disconnect the trap line from the in-stack filter and seal the tip with a clean Swagelok plug.
24. Disconnect the controller line from the trap outlet and seal the trap fitting with a clean Swagelok cap.
25. 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.
26. Place the bundled trap in the large plastic Ziploc bag provided, place in the cooler and add dry ice on top.
27. Disconnect the controller from the tank. Place the tank in an area where its temperature can equilibrate.
28. *After the tank temperature has equilibrated with its surroundings,* measure the final tank pressure and temperature as described above in steps 2-4 except the manifold pressure should be adjusted to approximate the ending tank pressure before closing the vacuum valve and opening the tank valve. Record the values on the field data sheet *and* on the wire tag on the tank, *and* on the M25Lab Chain of Custody Form. Replace the silicon rubber cap on the tank QC fitting.
29. Record the approximate moisture, CO2 and VOC concentrations for each run on each source on the M25Lab M25 Chain of Custody form.
30. Place the tank, controller and probe in the return shipping containers. Use the original packing materials and methods to protect the equipment. Tanks can be sent overnight express or by ground.

Ship the traps overnight express or second day air in a our sample return cooler 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, M25Lab recommends 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. M25Lab Normal turnaround time is <10 working days. Be sure to insure the samples and equipment to cover damage or loss.

31. Note that all M25Lab equipment is provided for up to 15 days, including transit times, at no additional charge. Rental fees may apply after 15 days per the current price schedule. Call us if your job is delayed, we may be able to waive some or all rental charges.