NC DAQ Source Test Observers Checklist – Particulate Testing
EPA Methods 1 - 5 & 202 (40 CFR 60 Appendix A)
Line by line explanation and references.

This checklist is intended to point out stack testing procedures that can be easily checked to help ensure an accurate and valid test. It is not intended to replace familiarity with the reference test methods. Some items can be filled out prior to the test observation, such as the Facility ID #, source tested, and applicable regulations. Note EPA Method 5 samples filterable particulate, while the addition of EPA Method 202 adds in the condensable particulate portion for a total particulate sample. The sampling train for this method is used in addition to filterable particulate collection using Method 5.

In addition, be certain to review the stack test protocol and the DAQ protocol approval letter prior to going on-site. These documents may indicate changes in methodology or special items you will need to verify during the observation. This information can be essential in answering questions on-site and determining if enough applicable data is being recorded. **Do not reject a test without consulting with the Stationary Source Compliance Branch (SSCB). If you have testing concerns discuss them immediately with the testing company and SSCB.**

Also, this reference document is intended to be used in conjunction with the Methods 1-5 checklist reference document. **Items common to both checklists (most of pages 1 and 3) are explained only in the Methods 1-5 checklist reference document.** This reference documents simply adds guidance for the checklist items from Method 202.

Changes to Page 1 from the Methods 1-5 (only) Observation Checklist

4.1) The impinger/moisture collection setup is very different for M202 versus a straight Method 4. This checklist item now simply informs the observer to refer to page 2 for the M202 requirements.

5.1) M202 is also required to be run concurrently with Methods 2-4.

5.3) The pre-run leak check is optional for a M5 only test. It is mandatory for a combined M5/202 test.

5.4) If the post-run leak check fails the run is voided. This is different from M5 only where the leak rate could be taken into account and the run still be considered valid.

5.9) Clarification is added that these cleanup procedures apply only to the "front half" (Method 5) portion of the sampling train. This is the equipment prior to and including the Method 5 filter. Cleanup for the "back half" (Method 202) portion of the train is very different and described on page 2 (Section 202.7) of the M5/202 checklist.

Method 202 Observation Checklist Guidance (for page 2 of 3 of the M5/202 Checklist)

202.1) *CPM sampling train set-up per method? (also called "back half" of the sampling train; see diagram)*

A. Method 23 Condenser with water bath
B. Dropout Impinger (empty/cutoff stem)
C. Modified Greenburg-Smith (GS) Impinger (empty, open tip)
D. CPM Filter (nonreactive, polymer, etc.)
E. Thermocouple (stainless steel encased, etc. in contact with gas stream?)
F. Ice Bath
G. Modified GS Impinger (100 ml water)
H. Silica Gel Impinger (see 4.3 page 1)
I. Exit Thermocouple (see 4.2 page 1)

The equipment in the sampling train prior to and including the Method 5 filterable particulate filter (nozzle, probe, hotbox, etc.) are discussed in Method 5 and its applicable DAQ checklist. This front part of the sampling train is typically called the filterable or "front half" components. The equipment following the hot box includes the Method 202 condensable particulate sampling train components (or "back half"). A schematic of the sampling train used in this method (M202) is shown in Figure 1 below.

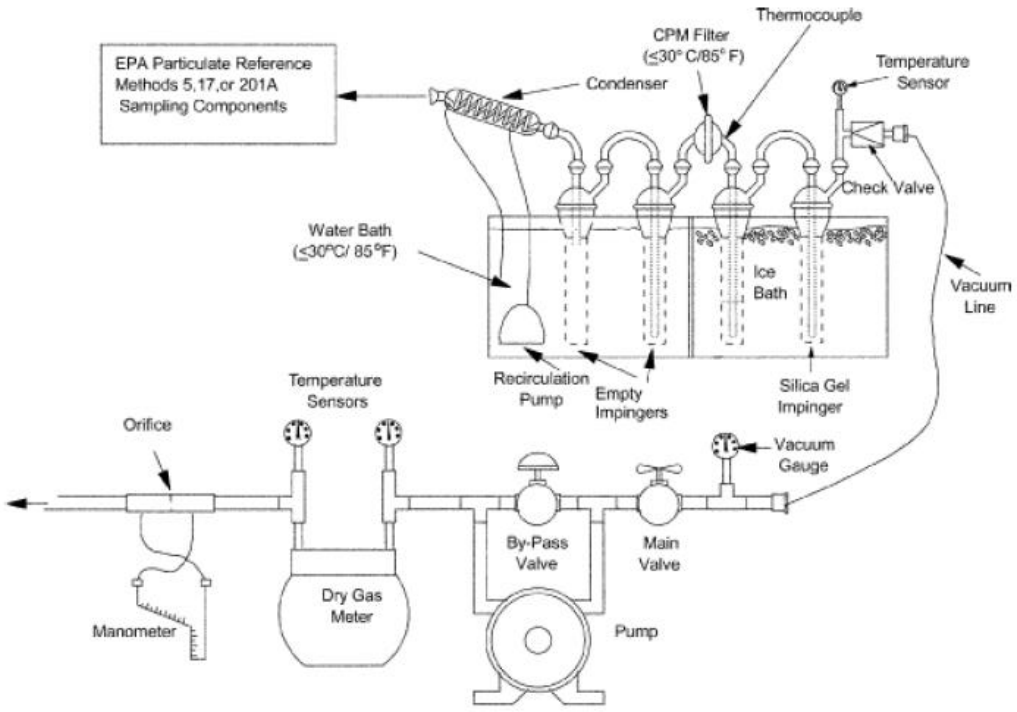


Figure 1 – Schematic of Condensable Particulate Sampling Train (Courtesy of the EPA)

A. *Method 23 Condenser with water bath* – This coil type condenser is the first component after the probe extension from the Method 5 filter. See the design schematic below and the picture in item I. The Method 23 type stack gas condenser is described in Section 2.1.2 of Method 23. The condenser must be capable of cooling the stack gas to less than or equal to 30 °C (85 °F).

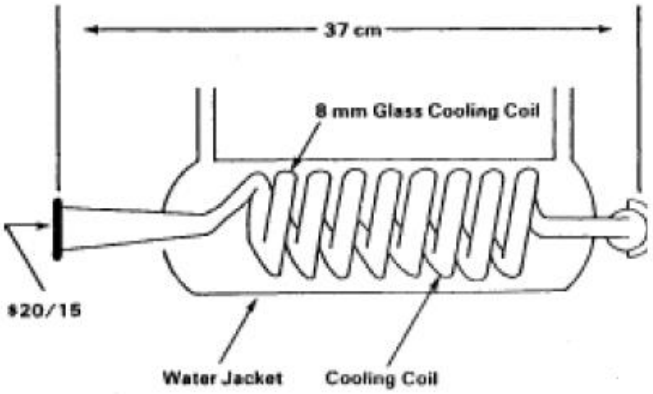


Figure 2 (Method 23 Type Condenser) Courtesy of the EPA

B. *Dropout Impinger (empty/cutoff stem)* – An empty water dropout impinger (knockout impinger) without bubbler tube follows the condenser (see picture 1 below).

Picture 1 - Dropout Impinger

C. *Modified Greenburg-Smith (GS) Impinger (empty, open tip)* - The water dropout impinger is followed by an empty modified Greenburg Smith impinger (backup impinger) with no taper (see picture 2 below). The water dropout and backup impingers are in an insulated box with a water bath at less than or equal to 30 °C (less than or equal to 85 °F). At the start of the test, the water dropout and backup impingers must be clean, without any water or reagent added. Monitor the moisture condensation in the knockout and backup impingers. If the accumulated water from moisture condensation overwhelms the knockout impinger, i.e., the water level is

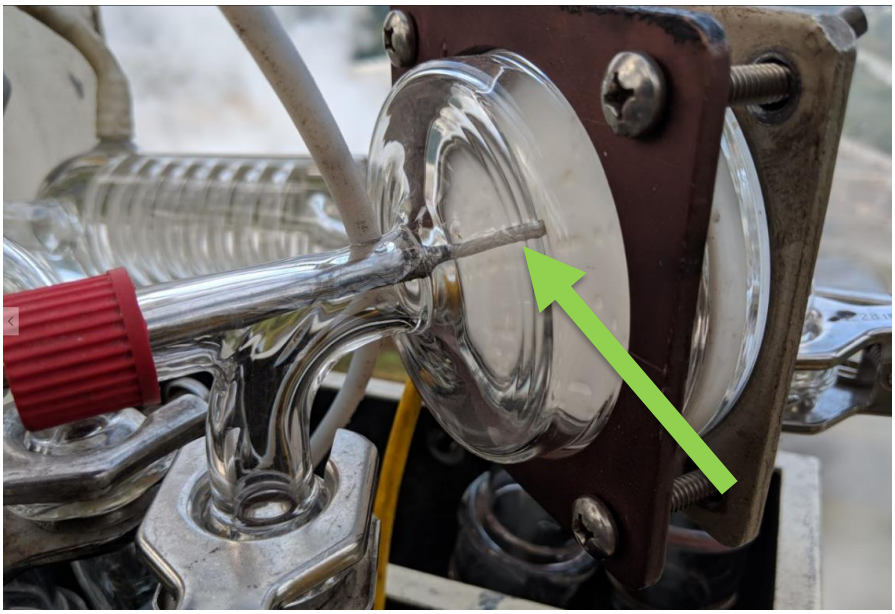
more than approximately one-half the capacity of the knockout impinger, or if water accumulates in the backup impinger sufficient to cover the impinger insert tip, then the tester may interrupt the sampling run, recover and weigh the moisture accumulated in the knockout and backup impinger, reassemble and leak check the sampling train, and resume the sampling run.

Picture 2 - Backup Impinger

D. <u>*CPM Filter*</u> *(nonreactive, polymer, etc.)* – The impinger in item C. above is followed by filter holder that is either glass, stainless steel or fluoropolymer-coated stainless steel. Inside the filter holder should be a CPM Filter meeting these specifications per the sampling method "*You must use a nonreactive, nondisintegrating polymer filter that does not have an organic binder and does not contribute more than 0.5 mg of residual mass to the CPM measurements. The CPM filter must also have an efficiency of at least 99.95 percent (less than 0.05 percent penetration) on 0.3 micrometer dioctyl phthalate particles.*"

Picture 3 – CPM Filter and Holder

E. *Thermocouple (stainless steel encased, etc. in contact with gas stream?)* - At the exit of the CPM filter, is installed a fluoropolymer-coated or stainless steel encased thermocouple that is in contact with the gas stream. A thermocouple "well" is not sufficient for this purpose because the thermocouple must be in contact with the sample gas.

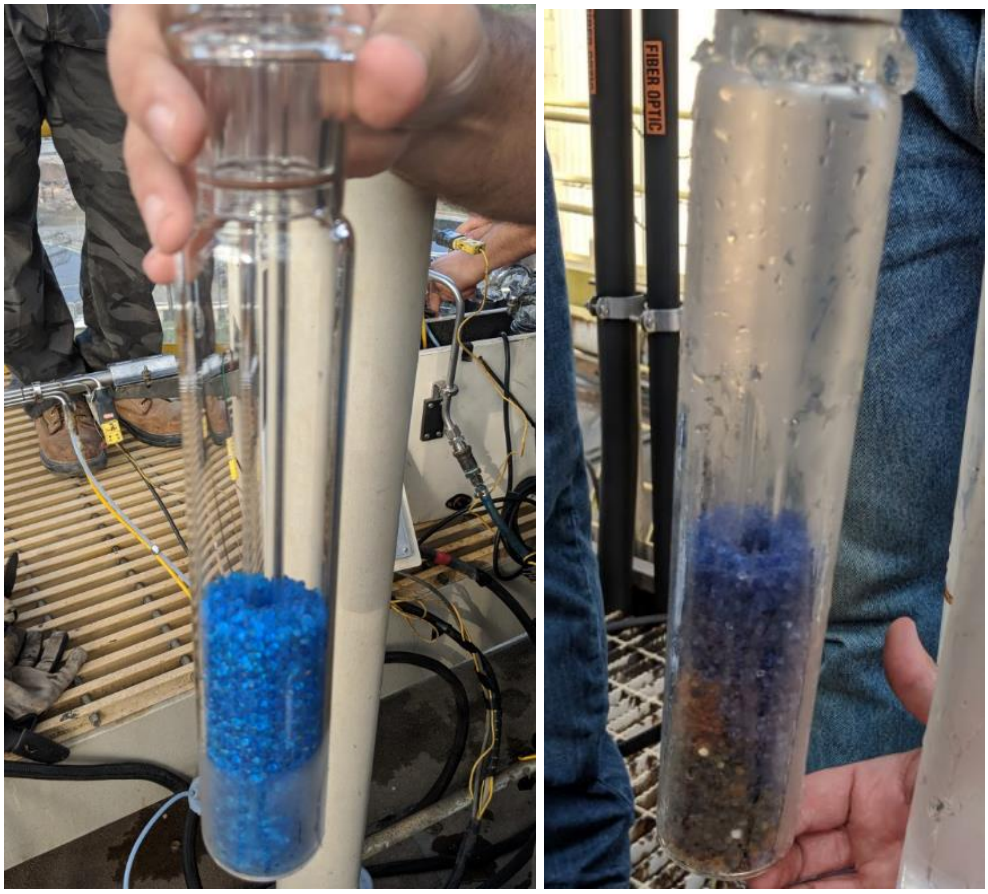


Picture 4 – CPM Thermocouple

F. *Ice Bath* – The impingers in items G. and H. below are placed in an ice bath to ensure collection of the moisture that passes through the CPM filter.

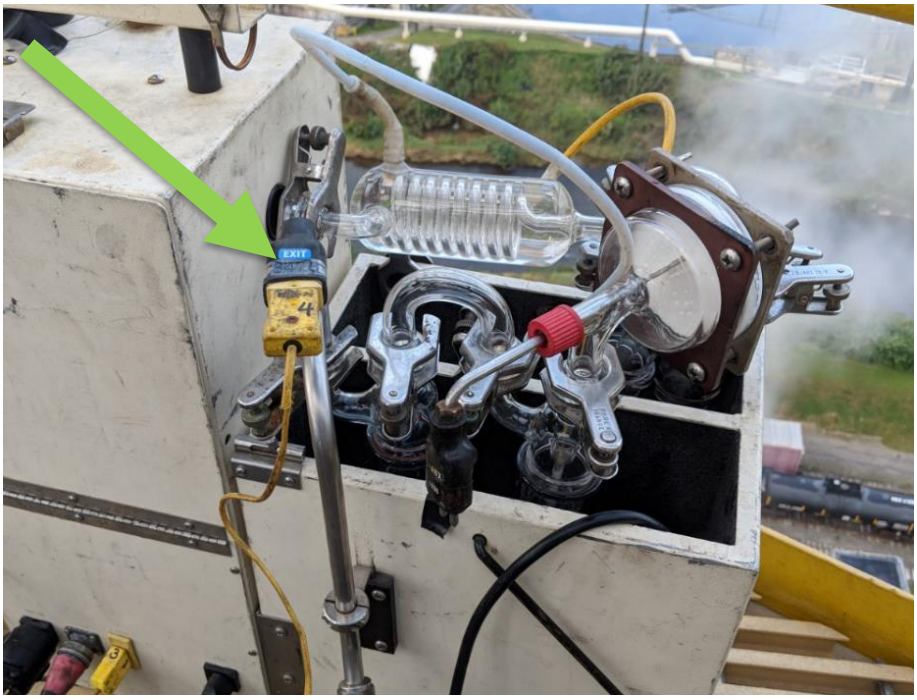
G. *Modified GS Impinger (100 ml water)* - Same impinger as item C. above but with 100 ml of water added.

H. *Silica Gel Impinger* (see 4.3 page 1 of the checklist and the Method 5 checklist help document). The silica gel impinger is shown in picture 5 below.



Picture 5 – Silica Gel Impingers [new (left) and nearly spent (right)]

I. *Exit Thermocouple* (see 4.2 page 1 of the checklist and the Method 5 checklist help document)



Picture 6 – Exit Impinger Thermocouple

202.2) *Glassware properly prepared before test? (soap & water, rinsed using tap water, DI water, acetone, and hexane, then bake at 300 °C for 6 hrs.) Otherwise, a field train proof blank is required (not as common)*

Sampling Train Preparation. A schematic of the sampling train used in this method is shown in Figure 1 above. All glassware that is used to collect and analyze samples must be cleaned prior to the test with soap and water, and rinsed using tap water, deionized water, acetone, and finally, hexane. It is important to completely remove all silicone grease from areas that will be exposed to the hexane rinse during sample recovery. After cleaning, you must bake glassware at 300 °C for six hours prior to beginning tests at each source category sampled at a facility. Prior to each sampling run, the train glassware used to collect condensable PM must be rinsed thoroughly with deionized, ultra-filtered water that that contains 1 ppmw (1 mg/L) residual mass or less. As a less common alternative to baking glassware, a field train proof blank, as specified in Method 202 Section 8.5.4.10, can be performed on the sampling train glassware that is used to collect CPM samples.

202.3) *CPM (or "ambient") filter maintained > 65°F and ≤ 85°F during test run?*

Use the same field data sheet for the filterable particulate method to record the CPM filter temperature readings at each sampling point and when sampling is halted. Maintain the CPM filter greater than 20 °C (greater than 65 °F) but less than or equal to 30 °C (less than or equal to 85 °F) during sample collection. (Note: Maintain the temperature of the CPM filter assembly as close to 30 °C (85 °F) as feasible.) In cold weather, do not allow a supplemental heater (i.e. handwarmers) near the thermocouple that may bias temperature measurements and not reflect the true gas temperature. The test company may take steps to warm the water bath that feeds the condenser to assist maintaining the 65 °F at the CPM filter.

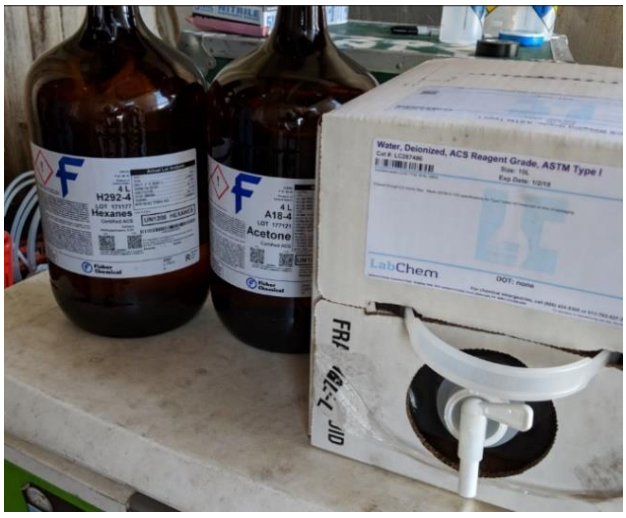
202.4) *Check reagent quality:*

A. Acetone - less than 1.0 ppmw (0.1 mg/100 g) (0.791 mg/l) residue mass

B. Hexane - less than 1.0 ppmw (0.1 mg/100 g)(0.655 mg/l) residue mass

C. Deionized, ultra-filtered water (DIUF) - contains 1 ppmw (1 mg/L) residue mass or less

The test observer should make an effort to observe the reagents on site and reasonably ensure they meet the above specifications.



Picture 6 – Example Cleanup Reagents

202.5) *Check if blanks prepared and completed:*

A. Field train recovery blank completed? (mention that a maximum 2.0 mg correction is allowed)

A Field Train Recovery Blank (FTRB) is used to determine a baseline for contamination of a train that is fully assembled and prepared the same as sampling trains that are used in the test series. However, the FTRB is not inserted into the stack, but recovered in the same manner as a fully sampled train. In addition, the full FTRB train is purged with nitrogen before sample recovery is performed. Essentially, an entire extra sampling train is assembled, purged, recovered, and analyzed for a FTRB. The analysis of the blank sample provides the level of contamination that could be expected to be present in a normal test sample (e.g., glassware, filters, reagents). The results from the recovery of the FTRB are subtracted from the actual sampled train results up to a maximum value of 2.0 milligrams to account for this contamination. This train is typically assembled after the first or second run.

B. Field blanks prepared for DIUF water, acetone, and hexane?

Field reagent blanks are QC control checks of the recovery agents that are taken in the field, directly from each reagent's wash bottle (EPA Method 202 Best Practices Handbook, January 2016). It is recommended that a sample of the water, acetone, and hexane used in the clean-up of a Method 202 train be collected and analyzed for impurities or contamination potential related to the FTRB. The reagent samples should be collected from the original reagent bottle to prevent possible contamination. Essentially, a sample is poured from the reagent bottle directly to a sample container while on site without being used. These samples are analyzed in same manner as test run samples to account for potential impurities in reagents.

Picture 7 – Example Field Blanks

202.6) *Post-run nitrogen purge (can be skipped if no water collected before CPM filter):*
This section describes the pressurized nitrogen purge. A less common setup may include using the sampling system meter box and vacuum pump and is not described here.

A. Purge required and conducted?
Method 202 requires a post-test nitrogen purge if water was collected in the sampling train prior to the CPM filter. This is conducted to remove partially absorbed gases/artifacts, including SO_2 which have the potential to bias the results. The purge is conducted by flowing ultra-high-purity (UHP) nitrogen at 14 lpm for one hour through the entire CPM sampling train (condenser inlet to CPM filter outlet), or from the backup impinger inlet to the CPM filter outlet.
B. H_2O transferred to backup (2nd) impinger?
If purge is not conducted through the entire sampling train from the condenser inlet to the CPM filter outlet, water collected in the condenser and knockout condenser should be quantitatively transferred to the backup (2^{nd}) impinger, where the purge is completed from the inlet of the backup impinger to the CPM filter outlet.
C. Ultra high purity (UHP) nitrogen used?
Ultra-High Purity compressed nitrogen or equivalent is required to purge the sampling train. It is required to have no more than 1 ppmv oxygen, 1 ppmv total hydrocarbons as carbon, and 2 ppmv moisture. The compressed nitrogen must not contribute more than 0.1 mg of residual mass per purge.
D. 14 lpm (liters per minute) for one hour?
The purge is required to be completed for a minimum of one hour at a flow rate of 14 lpm of UHP nitrogen to ensure complete removal of any potential artifacts. A rotameter capable of measuring gas flow up to 20 lpm and with an accuracy to 5% of full scale should be used and monitored during the purge
E. Gas temp maintained > 65 °F and ≤ 85 °F?
Per Method 202 Section 8.5.3.3, During either purge procedure, continue operation of the condenser recirculation pump, and heat or cool the water surrounding the first two impingers to maintain the gas temperature measured at the exit of the CPM filter greater than 20 °C (greater than 65 °F), but less than or equal to 30 °C (less than or equal to 85 °F). Continue the purge under these conditions for at least one hour, checking the rotameter value(s) periodically.
F. Does impinger tip extend below water level during purge? If not was DIUF H_20 added?
If choosing to conduct the purge through the entire sampling system from the condenser inlet to the CPM filter outlet, the short-stem knock-out impinger insert should be replaced with a modified Greenburg Smith Impinger insert. It is crucial that this insert tip length extend below the water level for both methods. If not, a MEASURED amount of degassed, deionized ultra-filtered water that contains 1ppmw (1 mg/L) residual mass or less be added until the impinger tip is at least 1 centimeter below the surface off the water. Any DI added should be recorded to correct the moisture content of the effluent gas stream.

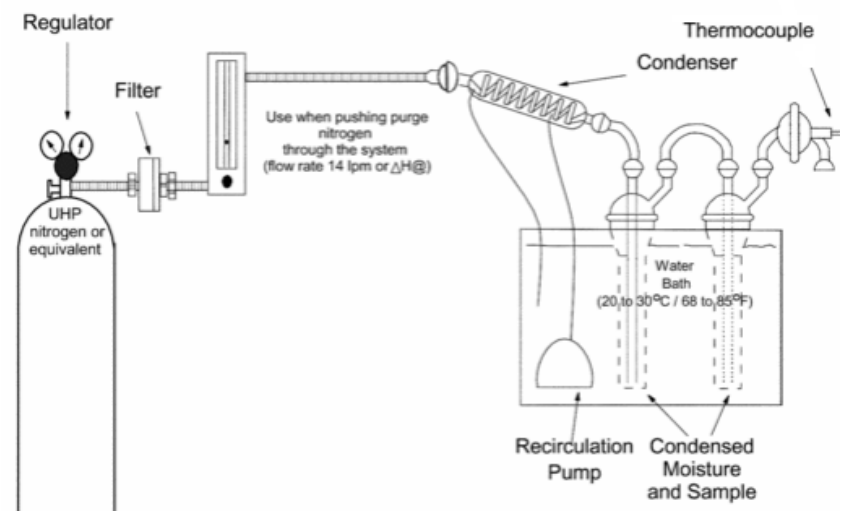


Figure 3 – Nitrogen Purge Setup (courtesy of the EPA)

Picture 8 (Nitrogen Purge Setup – white line is N_2 gas coming from UHP nitrogen tank (green at top); temperature sensor pictured (device with yellow buttons)

202.7) *Cleanup and sample recovery ("back half" - see page one for "front half" cleanup):*
A. Is test team catching all rinses in properly labeled containers?
B. At end of run, was glassware after M5 filter and before CPM filter rinsed as follows: two rinses with DIUF, one acetone rinse, and two rinses with hexane?
Following the Method 5 recovery and M202 nitrogen purge, impinger contents are quantitatively transferred from the dropout and back up impingers (prior to the CPM filter) into a clean, leak-proof container labeled CPM Container #1- Aqueous Liquid Impinger Contents. All sampling train components including the back half of the filterable PM filter bell, the probe extension, condenser, each impinger and connecting glassware, and the front half of the CPM filter are subject to DI water rinses, and subsequently added to CPM Container #1. Liquid level on the container should be marked to ensure no loss of sample between recovery and analysis. Next, the CPM Container #2- Organic Rinses are performed. First all glassware subject to the DI water rinses are rinsed with acetone, then subject to two rinses of hexane, with the acetone and hexane rinses being combined in CPM Container #2- Organic Rinses. Liquid level on the container should be marked as well. Lastly, tweezers and clean disposable gloves are used to remove the CPM filter from the holder and placed into a clean petri dish labeled CPM Container #3- Filter Sample.