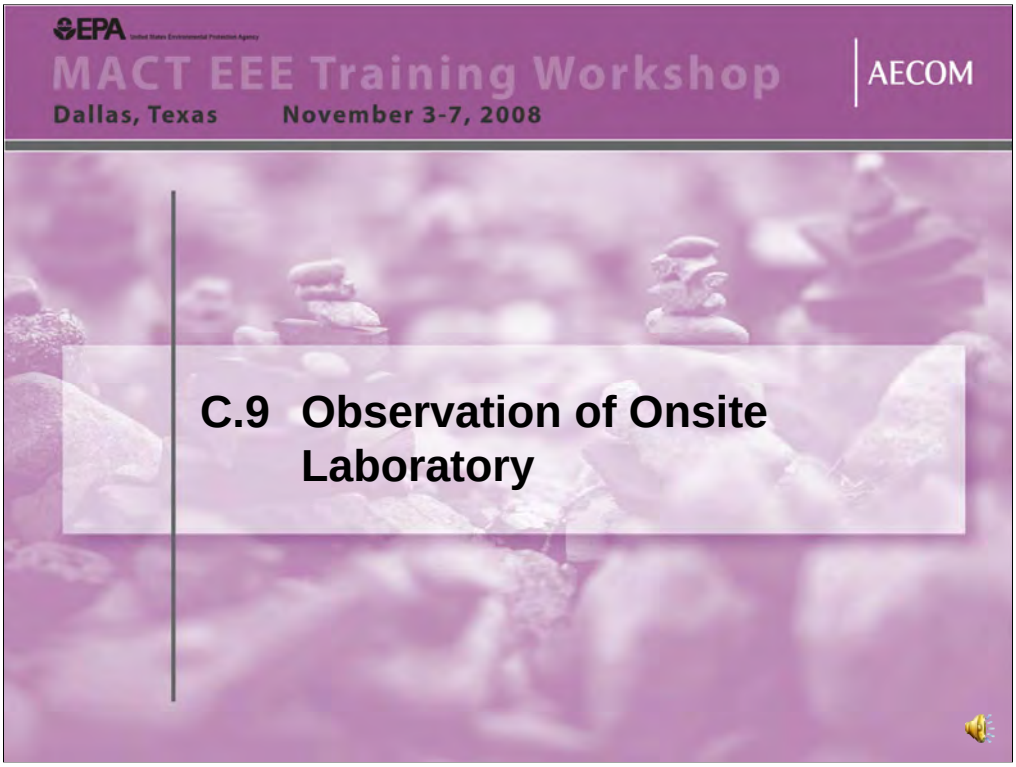


A critical part of a Subpart EEE test program is the onsite lab and observation of key activities for this will be reviewed in this module.

Presentation Overview

- What goes on in the onsite lab
- Another word about safety
- Sample train preparation
- Summary of recovery procedures
- Summary of sample handling activities

This module will first discuss the activities and safety issues concerning the onsite lab. Some of the key activities that will be reviewed are:

What goes on in the onsite lab,

Another word about safety,

Sample train preparation,

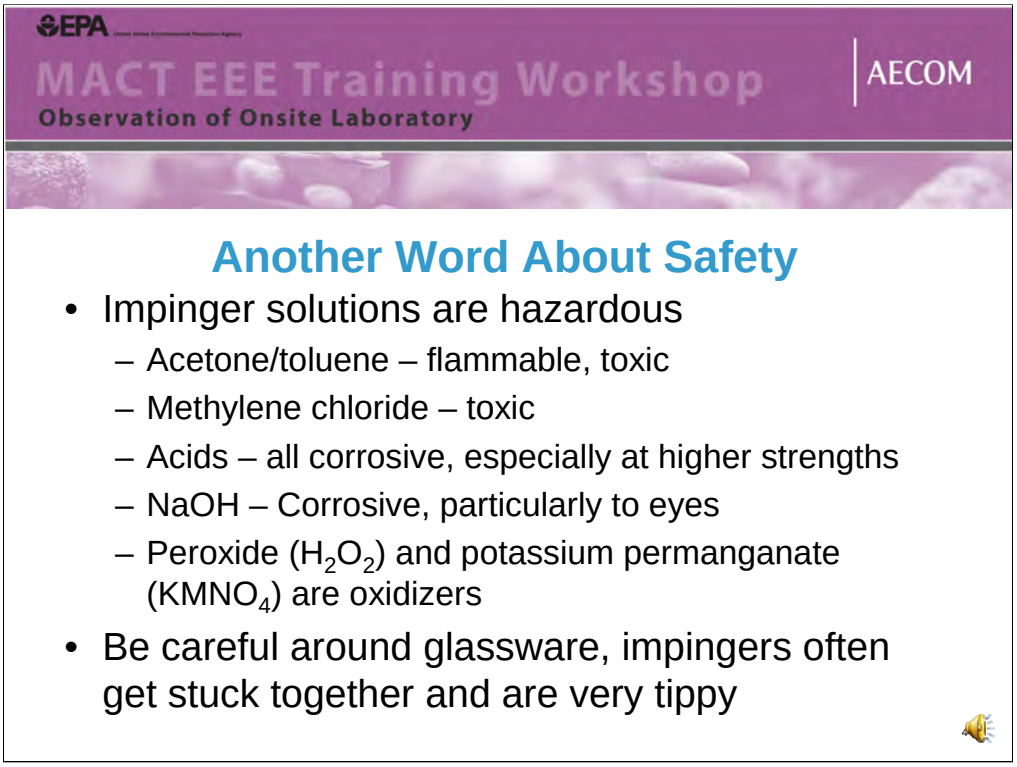
Summary of recovery procedures, and

Summary of sample handling activities

Onsite Lab Activities

- Coordinate stack sampling with process activities
- Maintain accurate records of
  - Run start, stop times,
  - Spiking and
  - Process conditions
- Sampling train materials, supplies, impinger solutions and preparation and sample recovery kit
- Sampling train recovery and preparation for shipment to offsite lab(s)

The onsite laboratory is a central function for the field program and key activities are coordinated through the staff assigned to this function. It is not unusual for the field team leader to also be leading the lab activities. Through this function, all activities of the field test can be coordinated, including the testing team, the HWC operations, and any specialty activities such as the spiking contractor. The lab manager typically maintains a field log book to document all pertinent test program related information, compiles all field data sheets, prepares and recovers the sampling trains and packages and labels the various sample fractions and containers for shipment of offsite laboratories.



From a safety perspective, there are several things for observers to be aware of in and around the lab. First, impinger solutions are typically hazardous or toxic and care should be taken when around them. And secondly, the glassware itself can sometimes get stuck together and is very tippy so caution is advised when observing lab staff handle and work with these items.

Sample Train Preparation - Particulate Matter, HCl/Cl_2 Train

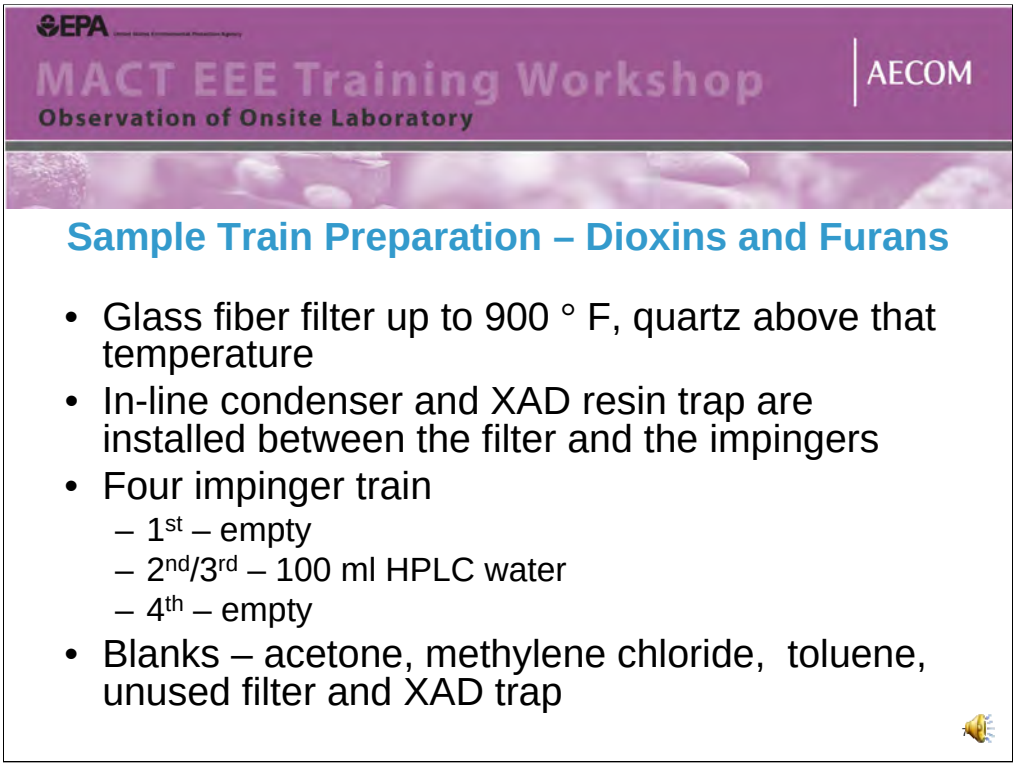
- Glass fiber filter up to 900 ° F, quartz above that temperature
  - Tared
- Five impinger train
  - $1^{st}/2^{nd}$ – 100 ml 0.1 Normal H_2SO_4
  - $3^{rd}/4^{th}$ – 100 ml 0.1 Normal NaOH
  - 5^{th} – empty
- Blanks – Acetone, DI water, H_2SO_4, NaOH

The following slides present summaries of how the primary sampling trains that are used in Subpart EEE CPTs are assembled. This train is a combination EPA Method 5/26A that is used for PM and HCl/Cl2 sampling. This train uses a tared glass fiber or quartz filter with five impingers prepared as indicated. A final impinger will contain dried silica gel to asborb any residual moisture before the gas sample passes through the dry gas meter. Typical blank samples are collected as indicated.

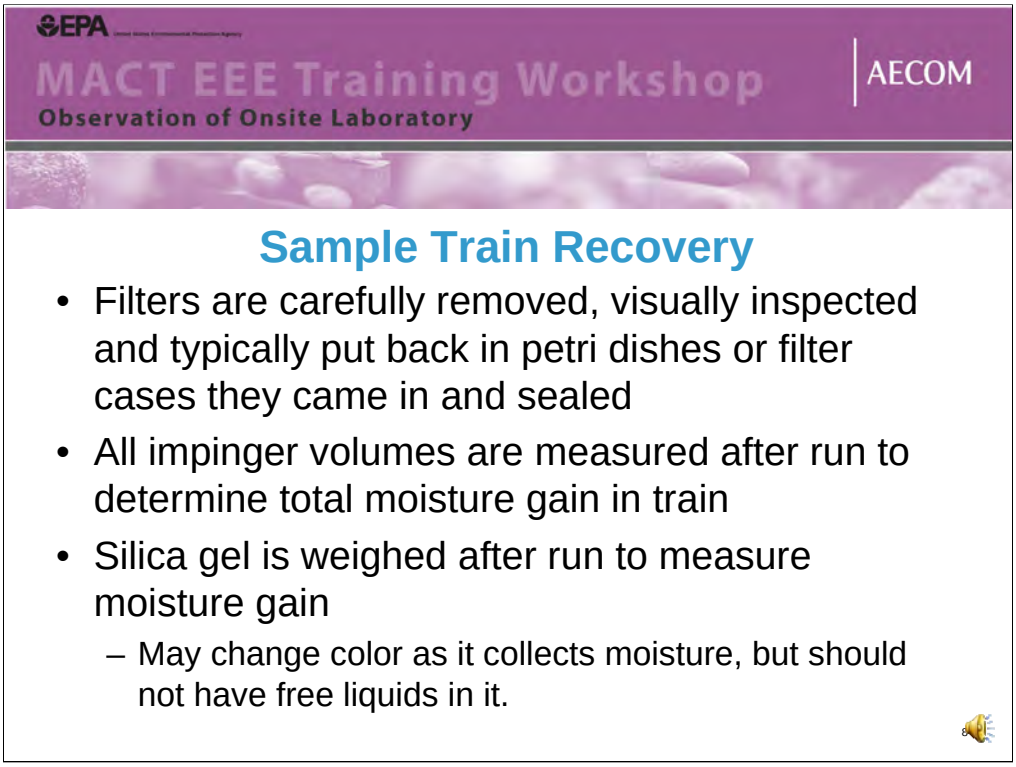
Sample Train Preparation – Method 29 Sampling Train

- Quartz filter
- Five impinger train (may add 6th if high moisture)
  - 1st/2nd – 100 ml 5% HNO_3/10% H_2O_2
  - 3rd – Empty
  - 4th/5th – 10% H_2SO_4/4% $KMNO_4$ (only if Hg is to be sampled for)
- Blanks – HNO_3, HNO_3/H_2O_2, $H_2SO_4/KMNO_4$, DI water, 8 N HCl, one unused filter

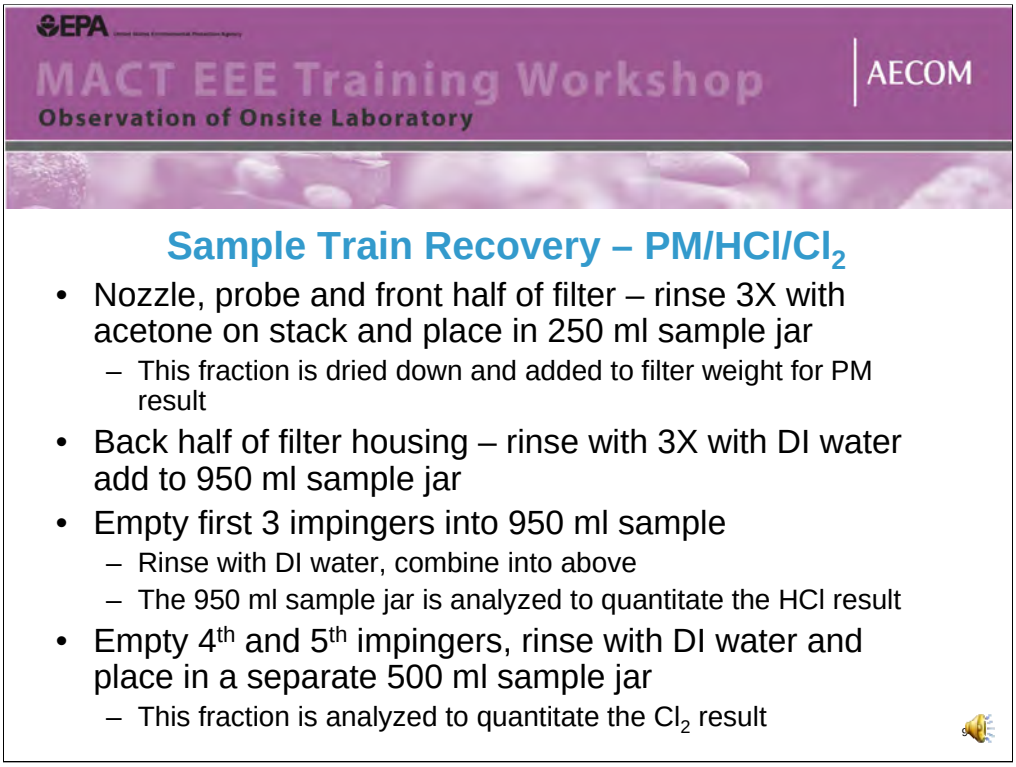
This summarizes the set-up of the multi-metals train in accordance with EPA Method 29 with the additional impingers for mercury sampling. While this train includes a filter, it does not have to be tared as no weight measurement is taken from it. Again, a final impinger is added that is filled with dried silica gel and the blanks are collected as shown on the slide.



The Dioxin and Furan sampling train incorporates a condenser and an XAD resin trap after the filter that has been pre-spiked by the offsite laboratory doing the analysis plus a four impinger set-up followed by the silica gel impinger. In addition to reagent and an unused filter, an unused XAD trap is also analyzed as part of the blank analyses.



The recovery of each isokinetic sampling train at the end of a test run involves several steps that must be done carefully and neatly. Lab cleanliness and organization and the lab manager's careful attention to the details of recovery and handling and labeling of all sample fractions is indicative good field lab practices. And while the opposite of this may not negatively affect results, high blank contamination or unusual results not otherwise explainable may possibly be traced back to the onsite lab. Procedurally, once the sample train has been lowered down from the stack and transferred to the lab, several essential steps are then performed. Filters are carefully removed with clean tweezers from the filter housing, these are then visually inspected and any unusual aspects noted in the field log and then the filters are placed a filter case and sealed and labeled. Then, each impinger in the train is poured out and the final volume recorded on a log sheet. The initial volume for that impinger is subtracted from the final volume and totaled for the entire train. The impinger containing the silica gel is also weighed and the difference between its initial and final weight is added to the other moisture to derive a total moisture content for that train and run.

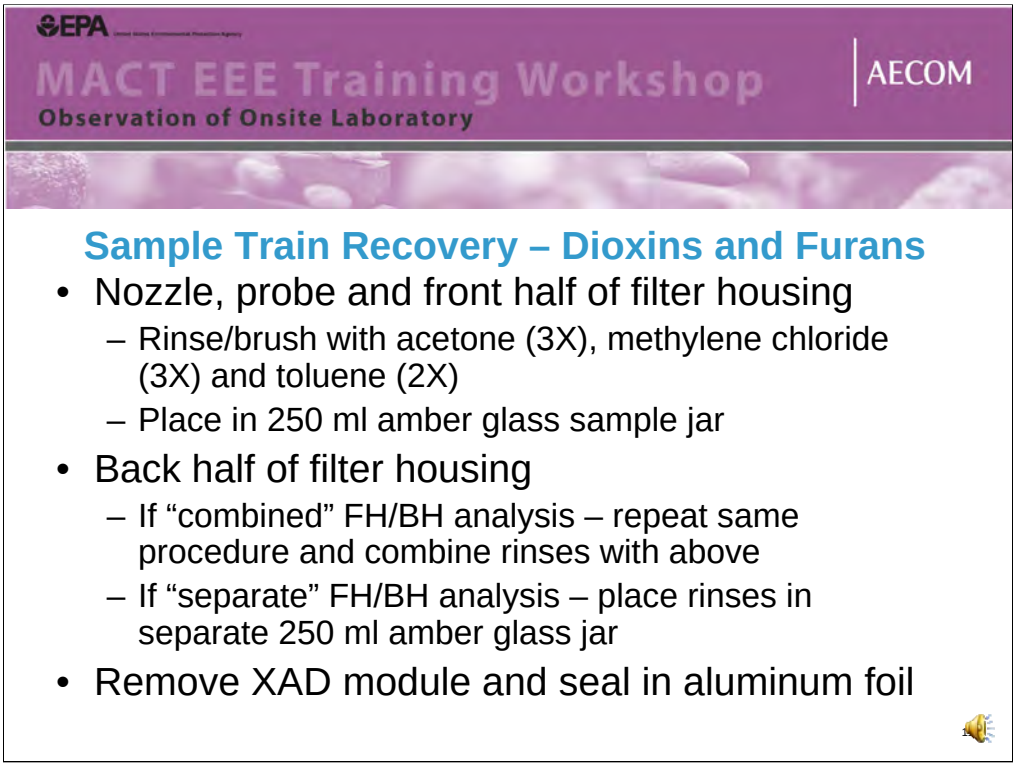


This slide summarizes recovery procedures for the PM/HCl/Cl2 combined sampling train. The nozzle, probe and front half rinse is generally done on the stack. That rinse is dried down and the weight is added to the dried filter for the PM result. In the onsite lab, once the filter is removed, the filter back-half and first 3 impingers are combined after rinsing with DI water and are shipped to the offsite lab for HCl determination. The contents of the 4th and 5th impingers are combined after rinsing with DI water and are packaged and shipped to the offsite lab for Cl2 determination.

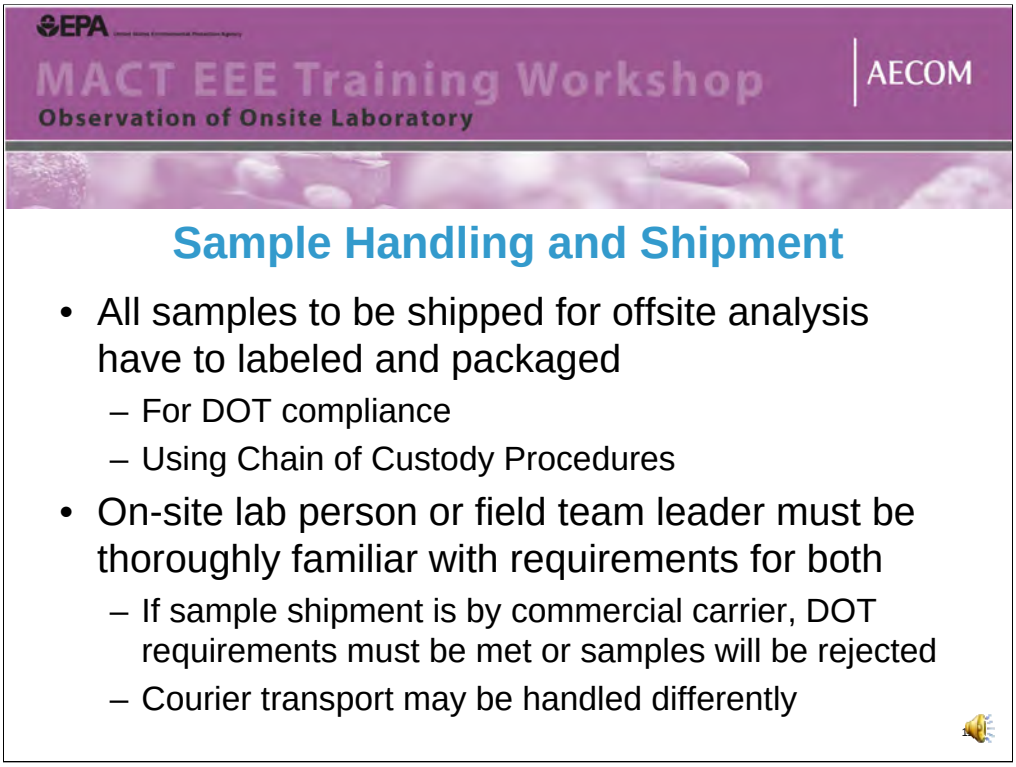
Sample Train Recovery – Method 29 Train

- Nozzle, probe and front half of filter housing
  - Rinse 3X with 0.1 N HNO_3, place in 250 ml amber glass sample jar
- Back of filter housing and first 2 impingers
  - Rinse 3X with 0.1 N HNO_3, place in 950 ml amber glass sample jar
- Empty 3rd impinger and rinse 3X with 0.1 N HNO_3, place in 250 ml amber glass sample jar
- Empty 4th and 5th impingers (for Hg)
  - Rinse with $KMNO_4$ reagent, DI water and the 8 N HCl, if needed, place in 500 ml amber glass sample jar

This slide summarizes the recovery steps for a Method 29 sampling train which includes sampling for mercury. Once again, the recovery of the nozzle, probe and front half of the filter is typically performed on the stack. Back in the onsite lab, several additional separate sample fractions are created in the recovery of the filter back and remaining impingers as shown on this slide. These fractions are separately labeled and shipped along with the filter to the offsite lab for analysis. Results can be presented by sample fraction, separate for front half and back half or as a total train result. Also, the permanganate solution should be made daily during the test program.



The recovery steps for a dioxin and furan sample train vary somewhat depending on whether results are being reported as separate front half and back half (Method 0023 A) or combined (Method 23). Once again, the first recovery step is performed on the nozzle, probe and front half of the filter housing using three different solvents and this if typically done on-stack. Back in the on-site lab, the additional solvent recovery is done and the XAD trap is removed and the ends sealed with aluminum foil. These fractions along with the filter are shipped to the offsite lab for analysis.

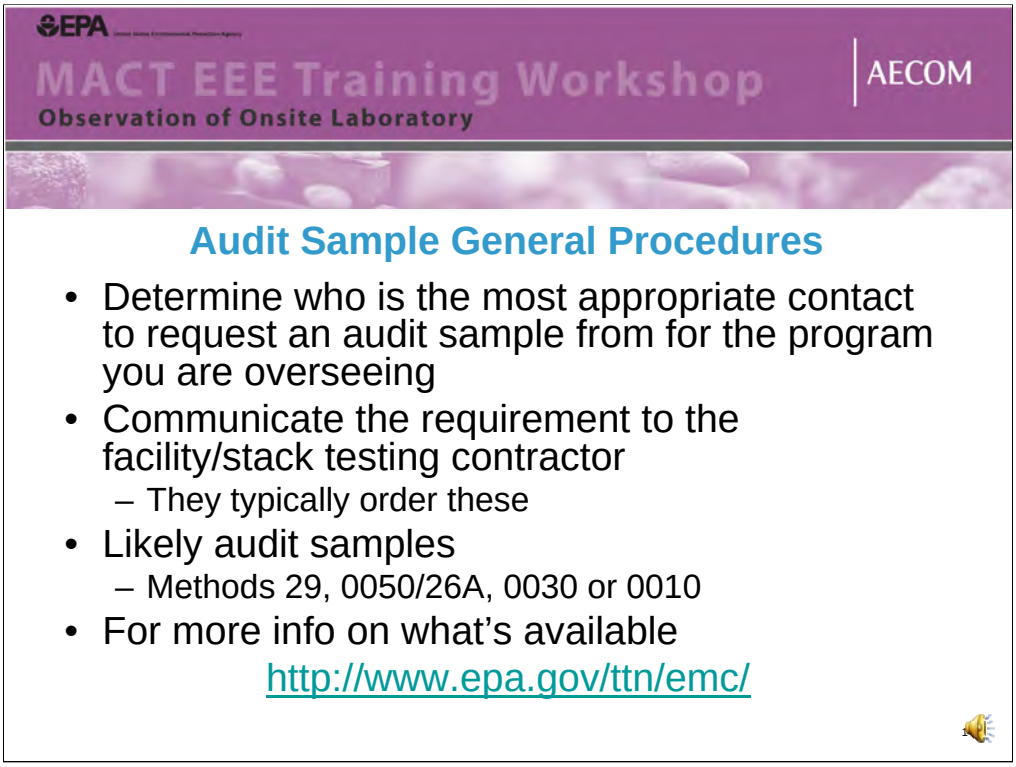


All sample fractions are separately labeled and then packaged in appropriate shipping containers for transportation to the lab. If these are being shipped by a commercial carrier compliance with DOT regulations must be addressed as most of the samples are DOT hazardous materials that must be packaged and labeled accordingly. DOT shipping papers must also accompany the samples along with the appropriate chain of custody documents. Courier transportation may be handled differently, but safe packaged must be assured to avoid any breakage. In addition VOST samples must be shipped cold, so provisions for storage, shipment and receipt at the lab must be made to avoid samples being left on a receiving dock for an extended period of time and allowed to warm up.

Audit Samples

- Purpose is to have a blind sample or samples, typically provided by regulatory agency handled and analyzed as part of the field program samples
- Not always done
- Testing contractor/facility and permitting agency should discuss whether this will be done well before testing so the audit sample(s) can be ordered and delivered to site

Agency audit samples may be included in a CPT at the request of the agency. These should be arranged for in advance and depending on the type of audit sample, the lab manager will likely end up including these samples along with program samples in the shipment to the offsite laboratories.



Agency observers are unlikely to become involved with audit samples other than to observe them as they are handled by the stack testing firm, but this slide provides some general background information.