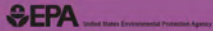


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United States Environmental Protection Agency

# MACT EEE Training Workshop


Dallas, Texas November 3-7, 2008

AECOM

## C.10 Reviewing Test Data



This module discusses what to look for when reviewing test data

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**Presentation Overview**

- Waste analysis data
- Spiking program results
- Process data
- Stack test methods data
- CEM data



The topics shown on this slide will be discussed.

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### What to Look for in Waste Analysis Data

- In general, are the sampling activities representative of what was fed
  - Samples of each major feed
  - Collected at representative intervals
  - Analyzed for key MACT constituents spelled out in CPT
- Review analytical report case narrative for any problems with QA samples
  - Duplicates and spiked duplicates
  - Laboratory control samples



When reviewing the waste feed data, the first major area to look for is to determine whether all the major hazardous waste feeds sampled and analyzed as specified in the CPT Plan. The Test report should include summary tables of all waste streams fed and what the results for each sample are. The analytical methods reported by the lab for each analyte should match what the CPT Plan called for, unless there was a reasonable justification for an alternative. In addition, quality parameters should be reviewed. This would include reviewing the lab narrative and the results of the duplicate, spiked duplicate and laboratory control sample analysis results. Specified holding times should be reviewed as well to assess whether samples were analyzed in a timely fashion. However, for some feed parameters, exceedance of the holding time does not mean the results automatically need be rejected and judgment should be used in evaluating this.

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### How to Evaluate Waste Feed Data


- Calculating mass feed rates from concentration data
  - Converting concentration data to lb/hr

$$\text{lb/hr} = Q_{\text{waste lb/hr}} * C_{\text{ppm or mg/kg}} * \text{kg}/10^6 \text{ mg or million parts}/10^6 \text{ parts}$$
$$\text{lb/hr} = Q_{\text{waste lb/hr}} * C_{\%} * 10^{-2}$$

- Calculate values for each run and each waste
  - Then, can totalize for all waste streams
- This can then be cross-checked with facility calculations



To check waste feed calculations for waste data reported in ug/g, mg/kg or ppm, the feed rate in pounds per hour is multiplied by the concentration and then divided by one million and that will yield constituent feed rate in pounds/hour. Similarly, for waste data reported in percent, like ash content. Multiply the waste feed rate by the concentration in percent and then divide by 100 to get pounds per hour.


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## Spiking Program Results

- Confirm spiking rates
  - Check actuals from Spiking Report against what was in the CPT
- For POHC
  - If native in waste, use analytical result and calculate lb/hr fed as on previous page
  - If pure material, use Certificate of Analysis (COA) for purity



To check the spiking rates, the Spiking Report should include a summary of what was actually fed during the program, which can in some cases, be different from what was planned. Feed rates for POHCs or other spiked constituents that are native in waste are calculation as previously discussed, if it is a solution or pure material, use the certificate of analysis provided by the supplier of the material.


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### Spiking Program Results - Continued

- For inorganic spiking constituents, calculate the MACT component using the solution strength and molecular weight
- For example, what is the lb/hr feed rate of chromium in a 1.5% potassium dichromate solution being fed at 5 lb/hr of solution



For inorganic spiking constituents like metals, the solution strength and molecular weight of the constituent must be used to calculate the pounds/hour of the constituent.

### Example Calculations

- Overall feed rate of potassium dichromate  
 $5 \text{ lb/hr} \times 0.015 = 0.075 \text{ lb/hr}$  potassium dichromate
- Chemical formula of potassium dichromate  
 $\text{K}_2\text{Cr}_2\text{O}_7$   
Molecular weights:  $\text{K} = 39$ ,  $\text{Cr} = 52$ ,  $\text{O} = 16$   
MW of  $\text{K}_2\text{Cr}_2\text{O}_7 = 294$
- Feedrate of Chromium =  $(104/294) \times 0.075$   
 $= 0.026 \text{ lb/hr}$



For a 1.5 % solution of potassium dichromate that is fed at 5 pounds per hour, that yields a net chromium feedrate as shown on this slide.



### Calculating Thermal Feedrates and Mass Weighted Thermal Feedrates


- Thermal feed rate of a single stream:  
 $\text{lb/hr of waste} * \text{Btu of waste/lb} = \text{Btu/hr}$
- Mass weighted thermal feedrate =  
 $(Q_{\text{waste1}}/Q_{\text{total}}) * \text{Btu/lb}_{\text{waste1}} +$   
 $(Q_{\text{waste2}}/Q_{\text{total}}) * \text{Btu/lb}_{\text{waste2}}, \text{ etc.}$

Where:

$$Q_{\text{waste1}} + Q_{\text{waste2}} = Q_{\text{total}} \text{ (lb/hr)}$$



Determining thermal firing rates for single feed streams and mass weighted thermal feed rates for multiple feed streams are summarized on this slide.

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### Calculating OPLs From Process Data

- MACT requires establishing OPL's from a several different types of process data
  - Average of minimum or maximum hourly rolling average
  - Average of test run average
  - Hourly rolling average



When working with the process data, it is essential to get this data in a spreadsheet format, such as Excel. Once in excel, the individual one minute data points can be selected over the run time and the mathematical functions in the program can be easily used to calculate OPLs.

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## Calculating Rolling Averages

- Continuous process data by definition means data collected at least every 15 seconds
- This continuous process data is used to generate a 1 minute data point, which is generally stored by the data system
- 60 individual 1-minute data points are used to calculate an hourly average
- For an hourly rolling average (HRA), the oldest 1 minute data point drops off and the newest is added to the 60 data points needed for the HRA
- For a 12 hour rolling average 720 minutes of consecutive data must be averaged, the rolling average is calculated as above



For process data to be considered continuous, it must be comprised of individual data collected at a minimum of every 15 seconds. The process data storage system must then store a 1 minute result taken from these more frequent readings for use on calculating averages. The hourly and 12-hour rolling averages are calculated as described on this slide.


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## Working with Process Data

- Process data is often summarized in CPT Report as a hard copy in an appendix, but
- Data is typically “dumped” from process data history or archiving system electronically into Excel or equivalent
- Calculations are performed in the spreadsheets
- Important to select run time ranges for calculations



This slide merely includes a couple of other comments on working with process data from a CPT.

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### Reviewing Stack Test Field Data

- Isokinetics should be 90-110% for all runs
- VOST meter boxes should pass pre and post-calibrations
- All meter boxes, thermocouples, hot boxes should have current (annual) calibrations
- Check completeness of field data sheets, question blanks or discrepancies
- Spot check field data inputs (delta H's, delta P's, dry gas meter and stack temperatures) into spreadsheets
- Can check other field data as well – reported moistures, minimum sample volumes, O<sub>2</sub> and CO<sub>2</sub>



From a stack testing perspective, there are a number of key overall issues a reviewer should look for. First, all isokinetic test runs should show be between 90 and 110% isokinetic. Results outside that range indicate that the stack was either under or over sampled. For VOST trains, the meter box must pass a pre-test calibration and a post-test calibration and those results should be included in the Test Report. All train related equipment needs to have current calibrations which is annual for meter boxes, thermocouples and hot boxes. Next, the reviewer can look over field data sheets to look for incomplete entries and discrepancies between what is on a field data sheet and what is reported in summary tables. Most stack testing firm use spreadsheets to transfer field data into and do the actual emissions calculations. Test reports should include copies of the spreadsheets so that various entries from the actual field data sheet can be spot checked for errors. Finally, other field data used in emissions calculations can be verified from the field data sheets such as sample gas volume, reported moisture and fixed gas content.

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## Reviewing the Analytical Data

- If certain lab certifications are required, make sure labs have them
  - Labs should definitely have experience with waste and stack sample matrices on both preparation and analytical finish
- Review the lab report case narratives for any reported issues
- Review lab data quality indicators
  - Spike recoveries
  - Duplicates
  - Laboratory control samples
  - Blanks



In reviewing analytical data, a first step is to make sure the labs used have any needed certifications for the methods they have been asked to perform. In addition, the lab should be specifically experienced in performing the analyses being requested as well. Using a lab that specializes in wastewater analyses only for analyzing stack samples, is not advisable, for example. Besides reviewing the actual results to make sure the data summarized in the test report matches the results in the actual analytical report, the actual analytical report should be provided with a case narrative and QA/QC results so that these can be reviewed as well. Sample hold times should be reviewed to make sure they were analyzed in a timely manner and QA/QC results should fall within the ranges specified in the QAPP portion of the CPT Plan.

## Working with Particulate Data

- Should have results for the net filter weight and for the front-half rinse – usually in mg dry weight
  - Weights should be done to 0.5 mg
- Should also have blank weight for filter and acetone blank
  - Method 5 does have a blank correction procedure, but it is not required to be used
  - Blank results should be reproducible between weights
- Use blank corrected or uncorrected total weight to calculate emission rate corrected to 7% O<sub>2</sub>



When reviewing PM data, there should be a dry weight for both the filter and for the front half acetone rinse. Balances should be able to read down to 0.5 mg. The results should also include blank values as well. PM results may or may not be blank corrected, but the emission rates needs to be corrected to 7% O<sub>2</sub>.


### Working with HCl/Cl<sub>2</sub> Analytical Data

- Analysis will come from two separate fractions of the sample train
  - HCl from the first three impingers
  - Cl<sub>2</sub> from the 4<sup>th</sup> and 5<sup>th</sup> impinger
- The analysis method is the same in both cases, but the impinger solutions and calculation from the raw analyses are different
- Use reported results, typically in  $\mu\text{g}$  directly in the calculation of ppmv, corrected to 7% O<sub>2</sub> or to lb/MMBtu for a thermal based standard



When reviewing HCl/Cl<sub>2</sub> results, note that the HCl result is calculated from the chloride content result of the first three impingers and the Cl<sub>2</sub> content from the 4<sup>th</sup> and 5<sup>th</sup> impingers. Typically a  $\mu\text{g}$  value is reported and the calculation is performed to determine a ppmv result, which is either corrected to 7% O<sub>2</sub> or a lb/hr emissions rate is then divided by the thermal firing in Btu/hr to determine a thermal feed rate.




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## HCl/Cl<sub>2</sub> Analytical Data QA/QC

- For Method 26A,
  - a matrix spike and a matrix spike duplicate should be analyzed with every batch, the front half and back half separately.
  - Results are all within the calibration curve,
  - MS/MSD display recoveries between 75-125%,
- The lab should present the pH of the last impingers. If they fall below 9, the results are probably biased low for chlorine.



Analytical QA/QC for HCl/Cl<sub>2</sub> analysis should include a matrix spike and a matrix spike duplicate from the front half and back half separately with recoveries between 75-125% and all results should be within the lab's calibration curve. Finally, the lab should check and report the pH of the last two impingers and if it is < 9, the results are probably biased low.

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**Working with Metals Analytical Data – except Mercury**

- Metals, except mercury are often reported as separate results
  - Front half – filter, probe rinse, front half of filter housing
  - Back half – back half of filter housing and impingers
- These must be summed for total metal catch, typically in  $\mu\text{g}$  to calculate emission concentration in  $\mu\text{g}/\text{dscm}$  corrected to 7%  $\text{O}_2$  or as  $\text{lb}/\text{MMBtu}$
- Data used in emission rate calculation can be blank corrected following EPA M 29 procedures
- Emission calculations can be performed with lab data that is either blank corrected or uncorrected



Metals (except mercury) results can be reported as combined or separate results for front and back half fractions. Mercury is reported a separate value as it is determined by a different analytical method. The individual fraction results must then be summed for a total  $\mu\text{g}$  catch and then calculations are performed to convert it to either a concentration value corrected to 7%  $\text{O}_2$  or to a thermal emission rate. Method 29 allows blank corrections and facilities have the option of utilizing the procedure or not.

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## Metals Analytical Data QA/QC

- For Method 29,
  - Blank train samples include a particulate filter and all other reagents and are they analyzed exactly the same way as the sample? Are they "clean"
  - Are matrix spikes and/or post digestion spikes included that display recoveries between 75-125%?
  - Does the lab record the quality of the permanganate as received.
  - The single most factor that biases mercury results low is the expiring of the potassium permanganate before the end of the test, and nobody catches it. Sulfur dioxide is a big culprit here.



Metals analytical QA/QC involves several different aspects. Blank train samples should be collected and analyzed along with the actual program samples. Matrix spikes and post digestion spikes should be between 75 and 125%. The quality of the permanganate is crucial to assure quality mercury results. Method 29 requires that this solution be made fresh daily.


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**Working with Metals Analytical Data – Mercury**

- Mercury will generally be reported as separate fraction results
  - Filter and front half rinse
  - Back half rinse plus impingers 1 through 3
  - Impinger 4
  - Impinger 5
  - KMNO<sub>4</sub> rinse from impinger 5
- Individual catches are totaled in  $\mu\text{g}$  and use to calculate emission rate in  $\mu\text{g}/\text{dscm}$  corrected to 7% O<sub>2</sub> or in lb/MMBtu
- As with other metals, M29 provides a blank correction procedure that can be followed. Emission calculations can be performed with corrected or uncorrected lab data 

Mercury results are reported as separate fractions and are then totaled and converted to emissions concentrations or thermal emission rates using the same calculations as are used for the other metals.

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### Working with Dioxin and Furan Analytical Data

- Depending on how the sampling was designed, results will be in pg as either
  - A single value for combined front half and back half
  - Or two separate values for front half and back half
- Seven dioxin isomers, 10 furan isomers, all tetra (4) through octa (8) chlorinated
- Each isomer has its own “toxicity factor”
- Main QA/QC issues are recoveries of surrogates and isotope dilution standards



Dioxin and furan analytical results are typically reported in picograms and can either represent separate or combined front half and back half data. The analytical report should include results for seven dioxin isomers, 10 furan isomers and totals for all tetra- through octa- chlorinated dioxin and furan congener group. EPA has developed a toxicity weighting approach based on toxicological studies and each isomer has its own toxicity factor. The catch of each isomer is converted to a concentration in micrograms per cubic meter, then is multiplied by its toxicity factor to yield a weighted stack concentration for that isomer. The weighted dioxin and furan isomer concentrations are then summed to yield a toxic equivalent concentration (or TEQ) to compare to a numerical standard. Primary QA/QC issues to look for are the surrogate and isotopic dilution standard recoveries which are 70 to 130% for the surrogates and 25 or 40 to 130% for the isotopic standards.

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### D/F Toxicity Factors

Isomer	TEF
2,3,7,8-TCDD	1.000
1,2,3,7,8-PeCDD	0.500
1,2,3,4,7,8-HxCDD	0.100
1,2,3,6,7,8-HxCDD	0.100
1,2,3,7,8,9-HxCDD	0.100
1,2,3,4,6,7,8-HpCDD	0.010
OCDD	0.001

Source: EPA 1989 Toxicity Factors

Isomer	TEF
2,3,7,8-TCDF	0.100
1,2,3,7,8-PeCDF	0.050
2,3,4,7,8-PeCDF	0.500
1,2,3,4,7,8-HxCDF	0.100
1,2,3,6,7,8-HxCDF	0.100
2,3,4,6,7,8-HxCDF	0.100
1,2,3,7,8,9-HxCDF	0.100
1,2,3,4,6,7,8-HpCDF	0.010
1,2,3,4,7,8,9-HpCDF	0.010
OCDF	0.001



EPA developed the dioxin and furan toxicity equivalency factors shown above in 1989 and these are used to calculate the D/F TEQ emission rates under Subpart EEE.