



This module summarizes relevant quality assurance and control aspects of conducting a CPT. This information is typically organized in what is call a Quality Assurance Project Plan which can either be a stand alone document or combined along with other information contained the CPT Plan.



This module will discuss why QAPP's are important, and step-by-step discussion for reviewing QAPP's and some final thoughts on QAPP's.



First, the overall goals of a QAPP are to have a mutually agreed to protocol that defines what data is to be collected, what the objectives and purpose of collecting that data are and therefore what level of accuracy and precision the results of that data collection should meet. Collection of data that is not needed to meet the objectives of the test program (i.e., show compliance with standards and establish OPLs) should be carefully considered before insisting on their inclusion in any test plan.



There are several places in the project where important QA/QC procedures need to be identified. Job set-up issues can include making sure necessary arrangements are made with laboratories, the facility and the testing contractor so that overall project QA/QC requirements are met. This will include making sure labs in particular, know what pre-spiking and sampling media are needed for the project and this is prepared in a timely manner. All field equipment to be used must be properly cleaned, calibrations need to be current and appropriate reagents and gases need to be ordered for the field work. There are also the essential QA/QC activities that occur during the test itself and then finally, there are post test quality requirements for certain test equipment and the laboratory analyses that must be followed.



In large part, this module is based on the US EPA Region 6 Guidance cited in this slide as this document represents a very useful reference for this work. Each element of that guidance will be reviewed in this module.



Elements 1 and 2 of the Guidance outline the title page, signature requirements and table of contents to be included in the QAPP.



Element 3 describes the general project description topics that should be addressed in the QAPP or CPT Plan. The topics are:

Brief project description with reference to CPT;

Concise statement of CPT purpose and objectives;

HWC description and wastes burned;

HWC diagram with all sampling and monitoring points;

Sampling and analysis program summary including sampling matrices and parameters, sampling methods, frequency and # of samples, analytical methods, and field measurements and monitoring methods;

Relationship of results to objectives;

Identification of critical and noncritical measurements;

CPT schedule; and

Identification of special requirements (personnel, equipment, reporting).

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- Are QA requirements sufficient to support CPT objectives?
- List of specific types of QC samples to be used to evaluate precision and accuracy (P&A) and required frequency
- Table of P&A criteria for all field and laboratory results
- Completeness goals for individual and overall CPT results
- Discussion of representativeness and comparability of CPT data
- Discussion of required MDLs and ability of proposed analytical methods to meet MDLs

All sampling and analysis activities associated with the CPT should have QA/QC objectives associated with the collection, handling (i.e., chain of custody) and analysis of results. This will include a summary of what samples will be analyzed to assess precision and accuracy of the results and what blanks will be collected and analyzed as well. Completeness goals for a CPT should be 100%. In addition, a discussion of how detection limits will be determined for specific methods should be included.

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General S Pr	Samplii ogram	ng and Overvi	Analytical ew						
Parameter	MACT Emission Limits	Sampling Method	Analytical Method						
DRE (on POHC)	99.99%	EPA Method 0010	EPA Method 8270C						
Particulate	34 mg/dscm	EPA Method 0050	EPA Method 5 Gravimetric						
HCI/CI ₂	77 ppmv (as HCI)	EPA Method 0050	EPA Method 9057 Ion Chromatography						
LVM Metals (As, Be, Cr)	97µg/dscm	EPA Method 29	EPA Method 6020 ICP-MS						
SVM Metals (Cd, Pb)	240 µg/dscm	EPA Method 29	EPA Method 6020 ICP-MS						
Mercury	130 µg/dscm	EPA Method 29	EPA Method 6020 ICP-MS						
PCDDs/PCDFs	0.40 ng TEQ/dscm	EPA Method 0023	EPA Method 8290 HRGC / HRMS						
Carbon Monoxide	100 ppmv	EPA Method 10	EPA Method 10 NDIR						
Total Hydrocarbons	10 ppmv	EPA Method 25A	EPA Method 25A FID						
Flow, Fixed Gases and Moisture	N/A	EPA Method 2, 3A and 4	N/A						
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While this table may differ from CPT to CPT depending on what subcategory the HWC falls into, what standards need to be tested for and whether MTEC or data in lieu of approaches will be used. A summary table of the performance parameters to be assessed, the relevant standards and sampling and analytical methods should be included in the QAPP.



In addition to the summary table suggested in the previous slide, a table or tables should be included that provide a detailed description of all sampling activities, including location, type of sample, methods for collection and transfer to samples containers, sample size and number of both program and QC samples to be collected. It should be clear what methods and procedures are to be used.



Element 7.1 of the Guidance includes some general and specific questions to consider in reviewing the QAPP. In general, QC activities should at a minimum meet the specific method requirements.

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	Sample Matrix and Sampling Method	Analytical Parameter	ers	Analytical Method	Lab (a)	Total Runs	Total Sa Blanks / Dups	Audit	Lab QC	Total		
	Spiked POHC -	РОНС	(b)	EPA M 8270C	VISTA	1	0	0	0	1		
	Stack Flue Gas EPA M 0050 BIF - Multimetals EPA M 0023 EPA M 3 EPA M 3 EPA M 10 EPA M 25A (a) Vista = Vista Analytical ENSR = ENSR A 17 700	Particulate and HCI and Cl ₂ Metals PCDDs/PCDFs Semi-Volatile POHC O ₂ and CO ₂ CO THC Laboratories, El Dorado	(c) (b) Hills, C	EPA M 5 EPA M 9057 EPA M 60207000 EPA M 8290 EPA M 8270C EPA M 3A EPA M 10 EPA M 25A	ENSR MAX MAX VISTA VISTA ENSR Fac Fac	3 3 3 3 3 3 3 3 3 3	1 1 1 1 1 0 0 0 0	0 0 1 1 1 1 0 0 0	1 1 1 1 1 0 0 0 0	5 5 6 6 3 3 3 3		
	ENSR = ENSR Air Toxics Laboratory, Westford, MA MAX = Maxxam Analytical Services, Burlington, Ontario, CANADA (b) POHC: Hexachtoroethane. (c) Metals include : As, Be, Cd, Cr, Pb, Hg.											Æ

A table like this should be included summarizing all sample types, sampling method, analytical parameters to be determined, analytical method to be used, who is responsible foor sample analysis and the number of field and QC samples associated with each measurement.



Pre-spiking of sample media (e.g., tenax or XAD resins) for VOC, SVOC and D/F analysis should be included as appropriate. Recovery ranges and acceptance criteria should be discussed in the QAPP as well.

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Element 7.1.2 Reagent Blanks

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The CPT QAPP should address the following:

- Reagent blanks for all field sample recovery efforts
- Reagent blanks for all laboratory sample prep and recovery efforts
- Requirement that solvents and reagents be from the same lot as those associated with the field samples
- Reagent blanks must be analyzed by the same analytical method(s) as the samples
- Any special reagents used in the CPT and the need for associated reagent blanks.

Reagent blanks are an important QC step in a well designed CPT as if there are questions on the results, the analyses of these samples can reveal whether there were high levels of the same constituent in the reagent.

The CPT QAPP should address:

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Reagent blanks for all field sample recovery efforts;

Reagent blanks for all laboratory sample prep and recovery efforts;

Requirement that solvents and reagents be from the same lot as those associated with the field samples;

Reagent blanks must be analyzed by the same analytical method(s) as the samples; and

Any special reagents used in the CPT and the need for associated reagent blanks.

ACCOM APP Review **Element 7.1.3 – Field Blanks** The CPT QAPP should address the following: • Collection of field blanks for all field sampling methods, including frequency • Requirement that media, solvents and reagents be from the same lot as those associated with the field samples • Field blanks must be analyzed by the same analytical method(s) as the samples • Any special reagents used in the CPT and the need for associated field blanks

Field blanks are another QC component that is performed as part of the actual testing program in order to evaluate whether there are any contaminants in the area where the test program is being conducted that could affect results quality. Generally, for each isokinetic sampling method train used in a program, a separate full sampling train is assembled, with appropriate media and reagents. The blank train is then recovered and the sample fractions sent off for analysis along with program samples.

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	Example QA	Table for S	SVOCs						
Quality Parameter	Method Determination	Frequency	Target Criteria						
Calibration	Five-level calibration curve; continuing calibration standard	At least once; at the beginning of day; continuing calibration once every 12 h and at end of day	<30% RSD of avg RRF; within 30% of a RRF from calibration						
Accuracy (calibration)	Analysis of calibration check	After every initial calibration	70% - 130% of theoretical value						
Accuracy (surrogates)	Isotopically-labeled compound spiked into samples prior to sampling and/or analysis	Every SVOC sample	50% - 150% recovery						
Precision (surrogates)	Same as for accuracy-surrogates pool results for each SVOC component	Every SVOC sample	<40% RPD of surrogate recovery. If more than 3 determinations- RSD < 35%						
Blanks	Method blank for each SVOC component	One per batch of samples	Blank value < 2x DL. If greater, DL is changed to 1.5x blank level						
	Field blank	Once per test	Evaluated on a case by case basis						

This is an example QA table for the analysis of semi-volatile organic compounds using EPA Method 0010 and analytical method 8270C



Element 7.1.4 discusses the role that trip blanks can play in evaluating whether program samples have been contaminated in transit to and from the lab. These are more commonly used for organic sampling activities, both VOC and SVOC and they can be in the form of VOA vials filled with distilled water, filters or media tubes or traps. All of these trip blanks would be shipped from the lab to the field program location and back to the lab and remain sealed the entire time.

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Element 7.2 – Waste Feed and Process Sampling Procedures

The following information should be included in this section:

- The sampling design should address production of a representative sample
- Specific written procedures for sampling waste feed and process samples should be included or referenced
- Detailed procedures for generating QC samples should be provided
- Explanation of any QC samples required for nonstandard media
- Instructions for collecting grab samples, and for preparing composite samples if needed
- Clear instructions for handling situations that may occur if waste feed or process samples are too heterogeneous or viscous to flow
- Any special instructions for analysis of these samples

Element 7.2 deals with assuring proper sampling procedures are used for waste and process stream sampling. The main issues to understand for this is the variability and heterogeneity of the wastes or process streams to be sampled. Sampling homogeneous liquid streams generally involves collecting a grab sample periodically during each run and then compositing that to yield a single composite per run for each individual waste. If the waste is believed to be somewhat variable, the frequency of the individual grabs can be increased so that resultant variability will be represented in the composite. For bulk solids, that for example are to be fed on a conveyor belt, sample collection technique is important to assure collection of a "representative" sample. This is particularly true where the bulk solid is heterogeneous. Drummed of solids, should generally be sampled from several depths in the container.

The following information should be included in this section:

The sampling design should address production of a representative sample;

Specific written procedures for sampling waste feed and process samples should be included or referenced;

Detailed procedures for generating QC samples should be provided;

Explanation of any QC samples required for nonstandard media;

Instructions for collecting grab samples, and for preparing composite samples if needed;

Clear instructions for handling situations that may occur if waste feed or process samples are too heterogeneous or viscous to flow; and

Any special instructions for analysis of these samples

AECOM AECOM CAPP Review Element 7.3 – Stack Gas Sampling Procedures This section of the QAPP should: • Provide adequate information to support the proposed emission

Include applicable analytes and MDLs in the description of sampling

- and analysis methods
- Assign analytes to the correct sampling and analysis methodologies
 Include detailed discussions of deviations from or modifications to cited methods
- Provide detailed SOPs for activities (field or laboratory) not covered by referenced methods.
- · Include examples or descriptions of record keeping forms
- Specify procedures for validating the analytical data

Element 7.3 discusses some of the important requirements that should be included in the QAPP regarding the stack sampling program. Detailed discussion should be provided regarding the methods used to show compliance with the emissions standards along with any method modifications that are planned. Where activities are not covered by the actual method, detailed procedures should be included.

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Element 7.3.1. discusses the various aspects of assuring that correct stack sampling locations have been selected and that proper flow measurements are made. The actual methods are found in Appendix A to 40 CFR 60. The methods listed in this slide are: method 1, method 2A, method 2B, method 2C, method 2D and method 2E.



Element 7.3.2 addresses measurement of oxygen and carbon dioxide and carbon monoxide. Oxygen and carbon dioxide are important to the calculation of the molecular weight of the flue gas. In addition, the these gases are also used for correction calculations. There are several methods that can be used for oxygen and carbon dioxide. Two very common methods are the ORSAT and Fyrite methods discussed in Method 3. This method requires the collection of a flue gas sample in a tedlar bag, the contents of which are reacted with specific solutions to yield the gas concentrations. These methods do not require a lot of equipment and are simple to set up. Method 3A is an instrument analyzer which essentially requires a full CEM sampling system with the necessary calibration gases.



Element 7.3.3 discusses the sampling for VOCs by EPA Method 0030, called the "VOST" method. This method is set up to collect samples in small paired tubes. Field, lab and trip blanks should be considered in using this method to evaluate possible contamination. In addition, each tube is only capable of absorbing so much analyte before "break through" occurs and the analyte passes through, which biases the sample results low. The CPT Plan should discuss how breakthrough will be handled.

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QAPP Review	Quality Parameter	Method Determination	Frequency	Target Criteria	
	Blanks - sample integrity and field contamination	Field Blanks, 1 pair of traps	One pair per sampling day	Less than lowest standard	1
	Blanks - verify no contamination from storage / shipment	Trip Blanks, 1 pair of traps	One pair per shipment	Less than lowest standard	
	Blanks - verify no lab contamination and system control	Lab Blanks, 1 pair of traps	Daily, before analysis of samples and in-between high-level samples	Less than lowest standard	
	Spiked Blank-Verify acceptable recovery of chlorbenzene	Spike Blanks, 3 pairs	Once for sample program	75% -125% mean RRF	
	Initial Calibration - GC/MS	3-5 stds bracketing DRE level	Prior to sample analysis	Variability of Avg RRF <= 20% RSD	
	Continuing Calibration	Midlevel standard	Prior to sample analysis, then every 12 hr, or after sample set	RRF within ±25% of initial calib (RRF)	
	Consistency in Chromatography	Monitor internal standard; retention time and area	Every sample, standard and blank	Retention time within ±30 sec of last calibration check; area within 65% to 135% from last daily calib. check	
	Precision and accuracy	Replicate analysis of 3 traps spiked with a standard independ. of calibration standards at the expected level of 99.99% DRE	Demonstrated prior to sample analysis	75%-125% recovery; ±25% RSD	
	Continuing accuracy check	Spike each sample with surrogate POHC	Every sample	Within 3 standard deviations of the initial accuracy found during the precision and accuracy determination	
	Verification of VOST System accuracy	Analysis of samples from EPA audit cylinder, if provided	Once per trial burn	Within 50% - 150% of certified concentration	
	VOST Condensate: Precision and accuracy	Surrogate POHCs spiked	All condensate samples	Recovery between 50% - 150%; RSD of all recoveries < 35%	
	Breakthrough Determination	Separate analysis of front and back traps	At least first pair from each run Unnecessary for blanks	Quantity on TX/C trap must be < 30% of amount on TX trap; does not apply when < 75 ng on TX/C trap	~
	RSD = relative standard RRF = relative response	deviation factor		•	2

This table summarizes relevant QA/QC requirements for the VOST method.



Element 7.3.4 summarizes key QA/QC aspects when using the Method 5/0010 sampling train which is an isokinetic train used for determining the concentration of semivolatile compounds in flue gas.

AECOM AECOM CAPP Review Element 7.3.4.1 – SVOCs and Dioxins and Furans Verify the following: • Appropriate sampling methodology has been determined • Sample preparation procedure proposed by the laboratory is described in detail and is appropriate for both groups of organic compounds

- Appropriate analytical instrument calibration procedures have been specified for two different analyses
- MDLs are available for all proposed analytes, or the procedure for determining the MDLs has been specified
- Appropriate field and laboratory QC samples have been addressed

SVOCs can be collected in the same or an essentially identical sampling train. Similar QA requirements apply, however different pre-spiking compounds will be used in the XAD modules.

The following should be verified for this element :

Appropriate sampling methodology has been determined;

Sample preparation procedure proposed by the laboratory is described in detail and is appropriate for both groups of organic compounds;

Appropriate analytical instrument calibration procedures have been specified for two different analyses;

MDLs are available for all proposed analytes, or the procedure for determining the MDLs has been specified; and

Appropriate field and laboratory QC samples have been addressed

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The following should be verified for this element:

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Appropriate sampling analytical methodology have been determined for these components;

Appropriate analytical instrument calibration procedures have been specified;

Calculation procedures that will produce final results in the correct reporting units have been specified; and

Appropriate field and laboratory QC samples have been addressed.



CPT Plans should generally include an evaluation of planned POHC feedrates and expected DRE to project emission rates for purposes of determining how to perform the VOST or SVOC sampling to assure that results will not be too high to detect them analytically. Fortunately, many HWC's have conducted previous DRE testing and have experience with appropriate spiking rates to prevent this from occurring.



There are several approaches for reporting detection limits the are used by laboratories. MDLs are the more common limit utilized by laboratories and a discussion of these are derived should be included CPT Plan. In addition, most laboratories today use a "reporting limit" for reporting non-detects which is generally somewhat higher than a MDL.



Element 7.4 summarizes information that needs to be collected from a process monitoring perspective. Much of this is already covered under CMS requirements the was discussed in module B.3.



Element 7.5 discusses requirements for CEMs which has also been previously covered in Module B.6.



Element 8 of the Guidance discusses aspects of the CPT that deal with sample shipment and handling from the point just after initial collection to laboratory receipt. Most importantly, all different samples to be collected as part of the program should have their preservation requirements and sample hold time stipulated in the QAPP as it is crucial for samples to be processed in a sufficiently timely manner to assure valid results. Hold times can be as short as several days and as long as several months and this varies with the sample matrix and analyte. In addition to holding time, strict accountability for sample handling is managed through the chain of custody which follows the samples from point of creation through to the analytical report. An example is shown on the next slide.

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Г	Site of Program :		Sample Date :	Project Location	Laboratory	Lab BOA / WO#'s	
ŀ	Type of Program :		Date Shipped :				
	Project No. :		Cooler No. :				
	ENSR Office :	WESTFORD	DOT Box No. :				
Ļ	ENSR Contact :	Doug Roeck	Signature :	Airbill No. :			
<u>-</u>	tem Sample ID	Matrix	Description	Parameters	Laboratory	Instructions	
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		(1) Please return t	his form with analytical resu	its.			
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This is an example Chain of Custody form. Note the level of detail that is included to make sure that each sample is uniquely identified by number and the form provides all information needed to assure the correct analysis is performed by the designated lab.



This slide discusses some additional aspects of sampling and analytical documentation and recordkeeping



Element 9 discusses the detail that should be provided in the QAPP. In general, the reviewer should verify that:

All analytical procedures are summarized in tabular form and should:

Include all analyses planned for each matrix for the trial burn;

List all sample preparation, cleanup, and analysis methods for each matrix and analytical parameter; and

Provide complete method references, including version.

Detailed SOPs for each analysis are referenced in this section and provided in an appendix.

The SOPs are of adequate detail and are consistent with the summary table and the referenced methods.



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Generally, only a summary discussion is provided for this topic since all equipment used in a CPT must be fully operational and properly calibrated in order to be used, otherwise, the CPT cannot proceed. It is typical for stack testing and spiking firms to have equipment calibration records available for review at the time of the field program.



These equations are typically included in the QAPP and are standard for all CPTs. The regulations require a minimum of three valid test runs per test condition be conducted and any test data needed to establish conformance with an emissions standard or to establish an OPL must be based on a complete set of data.



Element 14 addresses some procedures that should be included in the QAPP to address internal audits and corrective action.

This section of the QAPP should address the following:

Expected scope, frequency, and procedures for internal performance and systems audits;

The facility's readiness to accommodate external validation and assessment activities;

Mechanism and individuals responsible for triggering, initiating, developing, implementing, and documenting corrective actions during field, laboratory, and data validation and assessment activities;

A statement that all corrective actions likely to affect data quality will be brought to the permit writer's attention before they are implemented; and

Content and frequency of QA reports and identification of preparers and recipients.



The requirements for both CPT Plans and their associated QAPPs are overlapping in a number of areas. The best way to reduce the chances for errors and inconsistencies in this is to present the information once and cross reference it elsewhere. Well thought out and well written QAPPs are an essential part of a well run CPT or any complex compliance test and help to minimize the chance for problems or errors to occur.