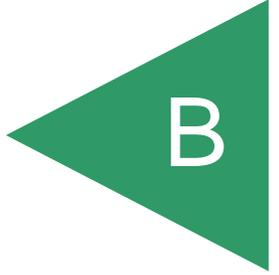


APPENDIX





EMSL Analytical, Inc.

Quality Program Summary

Based on Rev. 16 of the Quality Management System (QMS) Manual



The quality program at EMSL is built on a commitment to quality and continued improvement. This program is a primary part of our every day work; developed, utilized, and maintained by all the dedicated staff at EMSL.

Introduction

This program outline presents a comprehensive overview of the quality assurance program. It provides the reader with a summary of EMSL laboratory policies and procedures as they relate to the technical aspects of the corporate quality objectives.

This program follows quality guidelines as documented by ISO/IEC 17025:2005, the American Industrial Hygiene Association Laboratory Accreditation Program (AIHA-LAP), the EPA's National Voluntary Laboratory Approval Program (NVLAP), The NELAC Institute (TNI) and other applicable state and federal regulatory agencies.

This QA program is designed to ensure that the highest level of quality professional services and technical excellence is provided to our customers. This is accomplished by the implementation of program policies including:

- Development of company standard quality control programs
- Standardization of reporting formats
- Review of regional laboratory QC performance
- Providing technical training for all staff levels
- Achieving traceability of data
- Performance of quality audits
- Participation in applicable accreditation programs
- Participation in third party proficiency testing programs

The objectives of these program policies ensure the quality, accuracy and integrity of our analytical data.

The quality assurance objectives, policies and procedures are formally documented in the EMSL Quality Assurance Manual and program-specific Modules. A summary of this manual is presented on the following pages.

Topics covered are:

- | | |
|---|--|
| 1. Organization Structure | 10. Lab Conditions |
| 2. Document Control | 11. Equipment Calibration |
| 3. Purchasing | 12. Measurement Traceability |
| 4. Complaint Handling | 13. Sample Handling |
| 5. Corrective and Preventive Action | 14. Data Quality Programs |
| 6. Control of Records | 15. Ethics |
| 7. Internal and External Audits | 16. Customer Communication |
| 8. Management Reviews | 17. Notice of Performance |
| 9. Personnel and Training | 18. Estimate of Analytical Uncertainty |



EMSL Quality Policy Statement

EMSL is committed to providing a high standard of service and producing dependable, accurate and technically defensible test results in order to best serve our customers. EMSL will avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgment, or operational integrity. Our experienced and qualified technical personnel are committed to providing data of the highest quality achievable.

The senior management of EMSL Analytical, Inc. is committed to adopting the quality standards utilized by the various accrediting authorities – (e.g. NVLAP, AIHA-LAP, A2LA, state authorities) and those requirements documented in the ISO/IEC 17025 and TNI standards. The major goal (and focus) of the laboratory and its personnel will be toward constant improvement in the quality management system which has been designed with the purpose of ensuring consistent operations leading to quality data.

The senior management staff of EMSL acknowledges and accepts the responsibility for the overall quality of the data produced by the laboratory and makes a commitment toward continual improvement of the final product & the management system. In doing so, management provides the laboratory manager and the Quality Assurance Department with full authority to accomplish this end. Management is committed to providing all of the resources necessary to provide high quality analytical data.

All personnel concerned with testing within the laboratory must familiarize themselves with the quality documentation and implement the policies and procedures addressed in this manual.

Commitment to ISO Standard

- Starting with corporate management and extending to regional and local laboratory management, EMSL is committed to ensuring that the standards documented in ISO/IEC 17025:2005 (or the most recent revision of the 17025 standard) are upheld in all aspects of the company affairs

By way of authority, it is corporate management whom implements, maintains and monitors compliance.

This statement is issued under the authority of company President, Peter Frasca, Ph.D.



1. Organization Structure

The corporate headquarters of EMSL Analytical, Inc. operates out of the Cinnaminson, NJ location. The corporate management oversees the laboratory operations located there, as well as the branch laboratory locations. Corporate headquarters are responsible for the management of all company activities.

EMSL's approximately 30 branch laboratories (as well as the laboratory located in Cinnaminson, NJ) perform the company's analytical services. They report to the corporate headquarters on quality control, productivity, staffing and market issues. Each branch laboratory holds specific accreditations relevant to market requirements and the scope of their analytical work. A copy of each branch lab's qualifications is always available from the EMSL Analytical website.

The Quality Program for EMSL Analytical, Inc. is established and maintained by the corporate Quality Assurance Department, and implemented at the lab level by local branch lab managers and quality control personnel. All changes to corporately issued procedures, processes and policies must be approved by the QA Department and/or executive management of EMSL Analytical, Inc. Changes are controlled to ensure that affected personnel are notified and that the changes do not impact EMSL's compliance with outside regulations and requirements.

Due to this multi-laboratory structure, the EMSL Quality Program is in a constant state of review by outside assessors from multiple agencies. With outside assessments of the quality program occurring on average at least once per month, the program has been thoroughly vetted and is always in a state of continual improvement.

2. Document Control

In order to prepare and distribute documents in an organized fashion and ensure that the most up-to-date documents are available to the end user, procedures for initiation, preparation, review, approval and distribution of controlled copies have been established. EMSL's document control program is a coordinated effort involving both technical review and custodial control. Laboratories are to use only approved, controlled and current documents for all calibrations, analyses, final reports, and other activities performed in this laboratory. Most documents are controlled at the corporate level and may not be altered by branch laboratories without permission of corporate management. This ensures consistent operations across all EMSL branch laboratories and minimizes the risk for local inconsistencies to jeopardize the EMSL quality program as a whole. The document control system that has been established has proven to be effective, efficient and sustainable.

3. Purchasing

Quality results begin with ensuring that supplies and services purchased by EMSL meet necessary quality specifications. Vendors utilized by EMSL are selected based on the establishment of their own quality programs, their reputation in the industry, as well as our past experience when relevant. Supplies and services are only ordered from approved vendors, and any non-conforming product received from these vendors is isolated and reported to both laboratory management and purchasing department. Vendors are evaluated on a regular basis to ensure that products and services provided meet EMSL expectations and vendors are contacted for corrective action if products are found to be unacceptable.



4. Use of Customer Feedback

Customer feedback, both positive and negative, provides consistent input to the evaluation of the effectiveness of EMSL's quality program. Customer feedback is solicited through the use of customer surveys, as well as during routine communications with our customers. This data feeds directly into our continual improvement processes such as corrective and preventive actions and management reviews. Feedback is considered a crucial aspect of improving our quality program.

5. Corrective and Preventive Actions

The heart of EMSL's continual improvement process is the use of corrective and preventive actions to identify areas for improvement. Inputs to the corrective and preventive action programs come from all other quality systems implemented in the lab including, but not limited to: Audit findings (internal and external), complaints, QC results, customer and employee suggestions. The corrective action process includes an evaluation of a problem, a root cause analysis, and then the selection of corrective actions necessary to prevent recurrence. Preventive actions are improvements based on prospective problems that may arise in order to ensure they never occur. In both cases, follow-up actions ensure the actions taken have proven effective. EMSL also reviews corrective actions for trends which may indicate the need for further root cause analysis to improve systems across the entire EMSL enterprise. Corrective and preventive action records are reviewed by corporate management during annual management reviews.

6. Control of Records

EMSL recognizes the importance of maintaining accurate records in a manner that prevents degradation and allows for timely retrieval, if necessary. Policies and procedures for controlling records have been established by EMSL for implementation at the lab level. These policies include general requirements for how changes to records must be handled, confidentiality of records, and how records are to be stored. Each branch laboratory is responsible for how these policies are implemented in their laboratory based on the size, scope and volume of records generated. Electronic records are backed-up according to established procedures to ensure that they are retrievable in case of a computer error and where offsite storage of archived records is necessary storage is contracted with established record management services such as Iron Mountain. Tape backups of many crucial electronic records are also stored offsite to prevent loss in case of an unexpected disaster at the Cinnaminson, NJ location. EMSL has also established a standard 5-year record retention schedule, although some types of analyses require longer retention times as a function of accreditation requirements or through customer agreement.

It is understood that confidentiality and proprietary rights must be respected throughout the performance of services for any customer or for those that may include national security concerns. Information will not be given to those for whom it is not intended and the proprietary rights of our customer will be protected. Data reports and/or other related information will not be given out to any person or agency other than the customer unless we have received prior approval from the customer.



7. Internal and External Audits

EMSL's quality processes and procedures are continually being audited. One of the benefits of having multiple labs is that each is accredited independently and, therefore, each lab audit contains a review of the quality system. Between all branch labs, the EMSL Quality System is being assessed by a 3rd party approximately once per month. Non-conformities noted in these audits are then evaluated and where necessary quality system improvements are implemented for the company as a whole. As a result, the program is continually being improved and over time has become more efficient and effective. All external audit findings are directed to the corporate Quality Assurance Department and, therefore, systematic defects are more easily identified and corrected.

In addition to external assessments of the quality program, EMSL also has established policies and procedures for conducting annual internal audits of each laboratory. Internal audits use checklists similar to those used by outside agencies but with additional information on how to audit EMSL specific systems. Findings from internal audits go through the same corrective action process as external findings. Internal Audit findings and responses are forwarded to the corporate QA report and reviewed by corporate management as part of the annual management review process.

8. Management Reviews

The executive management team conducts annual management reviews for each branch laboratory for the previous year. Input to the management reviews include information from monthly and quarterly quality reports, results from internal and external audits, corrective and preventive actions implemented at each lab, results from lab participation in proficiency testing programs and inter-lab round robin exchanges, summaries of customer feedback, and input on resources and staff training changes. From this input, a management review report is generated which documents the executive management's findings on the suitability of policies and procedures currently in place and makes recommendations on actions that need to be completed by the branch laboratory or corporate management. Follow-up is conducted on these recommendations for improvement to ensure they have been carried out.

9. Personnel and Training

The EMSL Quality Program has established minimum requirements for all technical personnel hired by the laboratory. In addition, all analysts must complete an EMSL training program in order to perform any analysis independently. EMSL provides in-house training pertinent to all areas of analysis. Laboratory managers are responsible for ensuring that appropriate training is provided to every analyst and that they are completely qualified to perform analysis including demonstrating competency with the methods used.

In addition to initial training for lab personnel, ongoing evaluations of competency are conducted at least annually (some areas require 6 month evaluations) for each analyst. Where it is determined that additional training may be beneficial this will be conducted immediately. These evaluations will include review of routine QC, trends in analyst performance, performance in proficiency testing and/or blind sample rounds, and other data. This ongoing evaluation process ensures that the performance of EMSL analysts meets expectations at all times.



10. Lab Conditions/Environmental Monitoring

EMSL has established processes and procedures for ensuring that lab spaces are free from contamination which could jeopardize the health of its employees, customers, or affect the quality of results. In addition to general good housekeeping routines such as bench and hood cleaning with approved cleaners, quarterly monitoring is performed in the lab areas to ensure that there is no contamination above acceptable levels. In addition, blanks are run alongside most analyses which may be affected by cross-contamination. Whenever contamination is detected or suspected, corrective actions will be implemented and documented before re-evaluating to ensure the corrections were effective.

In addition to contamination, lab areas will be monitored for temperature, humidity, etc. where such conditions may have an affect on method performance or stability of samples. Refrigerators, freezers and incubators are monitored according to defined criteria to ensure that they are performing as expected. These checks are maintained in laboratory logs and referenced in lab records as appropriate.

In some more specialized areas, additional precautionary design features may be in place such as UV lighting, additional access controls, and continual monitoring systems.

11. Equipment Calibration

EMSL has established calibration programs for instrumentation which may affect the quality of results. These calibration programs define the frequency, parameters and acceptance criteria for equipment calibrations and calibration verifications. Standards used in in-house calibrations and routine calibration verifications are all traceable to NIST and come from approved providers. Where external calibrations are performed, these are performed by approved calibration services accredited to ISO 17025:2005 when available, and calibration reports must comply with the requirements of that standard. Equipment found to fail performance criteria must be either repaired and re-calibrated or taken out of service.

12. Measurement Traceability

EMSL processes and procedures are established to ensure measurement traceability to recognized standards whenever such standards are available. NIST traceable standards are mandatory for EMSL laboratories whenever available. Where no NIST-traceable standard exists, alternative standards may be considered but shall come from sources reviewed and approved by EMSL management (e.g., non-NIST sources such as ATCC, or where no external source is available, consensus standards such as graded proficiency testing samples). All calibrations shall be performed using traceable standards so that equipment performance is traceable to NIST or other approved source. Instruments shall be identified in lab records for each analysis along with standards used in calibration to ensure that final measurement results are traceable to the original standards.

13. Sample Handling

Chain of Custody

In order to ensure the integrity of any sample, records of its custody must be maintained throughout the sample collection in the field, acknowledgement of receipt, acceptance by the laboratory, and analysis.



EMSL does not perform sampling for customers, nor is it present at the time of sampling, and as a result cannot be responsible for issuing a chain of custody at the time of sampling. However, the laboratory can advise customers regarding sampling requirements (sampling materials, recommended sampling volumes, packaging, instructions for shipping, etc.) and chain-of-custody, and recommends that customers use published EMSL chain-of-custody forms whenever possible.

Once the sample is accepted for analysis by the laboratory, the EMSL “Internal Chain of Custody” is used to document the handling of the samples throughout the analytical process.

Sample Acceptance Criteria

In addition to acknowledgement of the receipt of samples, samples must also be accepted for analysis. Prior to accepting samples, the person preparing the samples for analysis inspects them to determine if they conform to laboratory acceptance criteria. If they do not, or if this person has any question as to the validity of the sample, the laboratory manager or an analyst trained to analyze such samples will determine whether the questionable circumstance is sufficient to cause rejection. Rejections of samples are to be followed up by immediate notification to the customer with an explanation. The sample will be returned upon request.

Log In

Log in of samples is normally done by the administrative coordinator, but may be done by any other employee familiar with the process. Information is entered for samples received into the Laboratory Information Management system (LIMS). LIMS is a computer laboratory information management system which serves to track all samples from receipt through the analysis, reporting, and billing processes. Samples are tagged with a project number label at this time to ensure that they cannot be separated from others in the project.

Archival and Disposal of Samples

Once the analysis is complete and the analysis worksheet is signed, the analyst stores the sample in the appropriate storage area as defined in relevant SOPs. All storage containers are to be stored in a safe manner for the period indicated for that category of waste and in accordance with regulatory requirements for sample retention.

Samples which are not completely consumed in analysis are retained as detailed in the EMSL QAM area-specific modules.

14. Data Control Programs

The ultimate purpose of the EMSL Quality Program is to ensure that “Final Test Reports,” which are EMSL’s final product, are reliable and defensible within the confines of the scope of work. Data is checked through a number of interrelated programs throughout the process from receipt to final approval.

Checks on lab and analyst proficiency are conducted using the following tools:

- External proficiency testing programs
- Round Robin programs



- Blind sample analysis
- Routine QC samples for evaluating accuracy and precision against established control and acceptance limits
- Analysis of data over time using control charts

Any out-of-specification result in a graded proficiency test or round robin will be evaluated and corrective action performed as appropriate.

Specific quality control requirements are established in the QAM and SOPs. These requirements are established by corporate management and the QA department and implemented by lab managers. In addition to quality samples such as blanks and verifications run at the time of samples to ensure accuracy of a particular run, inter- and intra- analyst reanalysis is performed where appropriate to verify accuracy and precision of each analyst. QC data is charted over time against established warning and acceptance limits in order to identify potentially significant trends which may lead to preventive actions. Whenever QC results are out-of-specification, this shall be evaluated by the analyst and lab management. Corrective action is taken where necessary.

In addition, for each project there is a continuous data review process culminating in a final review of the final report, usually by a second independent individual. At each stage, the information on the chain of custody and internal chain of custody is reviewed for errors and any errors are corrected. The final report review includes a thorough review of both completeness and accuracy. A check on any QC analysis performed in association with the samples is executed to ensure that all QC passed or that the final report is adequately notated. If any errors or suspected errors are identified, this will be discussed with the analyst prior to release of the final report.

15. Ethics

One of the objectives of the quality assurance program is to ensure the staff of EMSL is provided information regarding ethics as they pertain to corporate policy. The goals of the EMSL ethics policy and ongoing training program are:

- For each staff member to understand the responsibility to provide true and accurate information.
- The understanding of the consequences of unethical conduct.
- Provide direction to employees regarding prevention of unethical conduct.
- Define right and wrong (as it is job related.)
- Ensuring that all employees are free of undue pressures.

16. Customer Communications

Clear, continuous and open communication between the laboratory and the customer is one of the keys to maintaining a successful, quality operation. Communication should be established prior to the start of any work. Information must be clearly understood between laboratory management and the customer. EMSL provides quality assurance information and technical support to the customer to assure continued quality service. The support and information provided in relation to the work performed includes:

- Field sampling guides



- Availability of pertinent QC records
- Access to the Quality Assurance Department for technical assistance
- Security of data (confidentiality)
- Reasonable access to the relevant areas of the laboratory for the witnessing of analysis

If a major deficiency in policy or procedure is identified which directly affects customer results, the customer will be notified immediately of the problem.

17. Notice of Performance

The laboratory manager shall provide the customer with information as it relates to the performance of the analysis and turnaround time. The laboratory shall notify the customer if:

- Analysis cannot be performed on time
- Integrity of the sample has been jeopardized (either by the laboratory or the customer)
- A discrepancy in the analysis has been found during QC analysis.

18. Estimate of Analytical Uncertainty

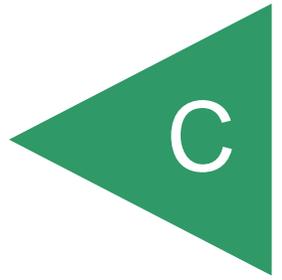
When requested by the customer or required by regulatory agency, an estimate of analytical uncertainty may be provided with customer results. Generally, the uncertainty will be reported at a confidence interval of 95%. It should be noted that this is only analytical uncertainty, and does not cover those contributors to uncertainty that may arise from sample collection activities.



Revision History:

Revision	Rev. Date	Changes	Rev. Author
5	1/16/2014	<ul style="list-style-type: none">• Name of document changed to EMSLQMSOutline.• Content changed to reflect updates which had occurred between revisions 9 – 16 of the Quality Manual.	P. Dragasakis
4.1	4/16/2009	<ul style="list-style-type: none">• Added EMSL logo in header and title.	
4	4/22/2008	<ul style="list-style-type: none">• Numerous grammatical and typographical corrections.Added “Commitment to ISO” language. Added table with sample retention standard times.	

APPENDIX



Title : **EPA Method TO-4A: Determination of Pesticides and PCBs in Ambient Air by Gas Chromatography / Electron Capture Detector.**

Document No. : SOP-M456
Revision No. : 1.1
Supersedes : 1.0

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Revision 1.1 changes are noted in bold italicized typeface and preceded by a "►" marker.

APPROVED FOR RELEASE BY:


MANAGEMENT

10/03/13
DATE


QA DEPARTMENT

10-03-13
DATE

1. METHOD IDENTIFICATION

- 1.1. Determination of Pesticides and PCBs in Ambient Air by Gas Chromatography (GC) / Electron Capture Detector (ECD).

2. APPLICABLE MATRICES

- 2.1. This method is used to determine the concentration of common pesticides and PCBs in ambient air utilizing filters and sorbent cartridges (PUF) followed by GC analysis using an ECD detector.

3. DETECTION LIMITS

- 3.1. The estimated quantitation limits (EQLs) for this method are as follows:

<u>Pesticides in Air</u>	<u>PCBs as Aroclors in Air</u>
0.100 µg/sample	1.00 ug/sample

- 3.2. The EQLs will be proportionally higher for sample extracts which require dilution or cleanup.

4. SCOPE AND APPLICATION

- 4.1. EPA Method TO-4A describes a sampling and analysis procedure for common Pesticides and PCBs involving the use of a combination of quartz filter and polyurethane foam (PUF) cartridge with subsequent analysis by gas chromatography with Electron Capture Detector (ECD) and is based on EPA Method 8081A/B. and 8082 The following compounds may be determined by this method:

- 4.2. The following compounds are routinely determined by this method.

aldrin	endosulfan I	
α-BHC	endosulfan II	
β-BHC	endosulfan sulfate	
γ-BHC (lindane)	endrin	
δ-BHC	endrin aldehyde	
α-chlordane	endrin ketone	
γ-chlordane	heptachlor	
heptachlor epoxide	hexachlorobenzene	
4,4'-DDD	methoxychlor	
4,4'-DDE	dieldrin	
4,4'-DDT		
Aroclor-1016	Aroclor-1242	Aroclor-1260
Aroclor-1221	Aroclor-1248	Aroclor-1262
Aroclor-1232	Aroclor-1254	Aroclor-1268

- 4.3. The following compounds may also be determined by this method:

2,4'-DDE (o,p'-DDE)	chlorobenzilate
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2,4'-DDD (o,p'-DDD)	hexachlorocyclopentadiene
2,4'-DDT (o,p'-DDT)	mirex,
4-4'-dichlorobenzophenone (4,4'-DCBP)	kepone
cis-nonachlor	trans-nonachlor
diallate	oxychlorodane

- 4.4. Upon client request, additional target analytes may be added to this analysis. However, it needs to be demonstrated that any added compounds lend themselves to determination, either by regulatory reference or validation studies.
- 4.5. This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph (GC) and skilled in the interpretation of gas chromatograms.

5. METHOD SUMMARY

- 5.1. Filters and sorbent cartridges (containing PUF) are cleaned in solvents and dried. The filters and cartridges are stored in screw-capped jars wrapped in aluminum foil (or otherwise protected from light) before careful installation on the sampler.
- 5.2. The amount of air sampled through the filter and sorbent cartridge is recorded, and the filter and cartridge are placed in an appropriately labeled container and shipped along with blank filter and sorbent cartridges to the analytical laboratory for analysis.
- 5.3. The filters and sorbent cartridge are extracted by Soxhlet extraction with diethyl ether/hexane (10%v/v). Final extract volume is 10 mL. The PUF extracts are then analyzed by Gas Chromatography coupled with an electron capture detector (ECD).
- 5.3.1. A variety of cleanup procedures may be applied to the extracts, depending on the nature of the target analytes and the matrix interferences.

Prior to performing this procedure, the following sample preparation technique may be performed on each sample.

<u>Type of Sample Preparation</u>	<u>EPA Method No.</u>	<u>SOP No.</u>
Soxhlet Extraction	EPA 3540C	SOP-M203
Gel Permeation Clean-up	EPA 3640A	SOP-M233
Other Clean-ups	EPA 3600C (M)	SOP-M234

6. DEFINITIONS

- 6.1. Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
- 6.2. Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

- 6.3. ►Batch: Environmental samples, which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours, ***unless a client specific Quality Assurance Project Plan (QAPP) guidance provides for a lesser time period or a method specific Standard Operating Procedure (SOP) establishes a different time period, not to exceed 24 hours.*** An analytical batch is composed of prepared environmental samples which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples..
- 6.4. Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
- 6.5. Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.
- 6.6. Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.
- 6.7. Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.
- 6.8. Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.
- 6.9. Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
- 6.10. Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.
- 6.11. Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
- 6.12. Limit of Detection (LOD): A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility.
- 6.13. Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.
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- 6.14. **Matrix Spike (spiked sample or fortified sample):** A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
 - 6.15. **Matrix Spike Duplicate (spiked sample or fortified sample duplicate):** A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
 - 6.16. **Method Blank:** A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
 - 6.17. **Method Detection Limit:** The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
 - 6.18. **Precision:** The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
 - 6.19. **Preservation:** Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.
 - 6.20. **Pure Reagent Water:** Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.
 - 6.21. **Quality Assurance:** An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.
 - 6.22. **Quality Control:** The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.
 - 6.23. **Quantitation Limits:** Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence.
 - 6.24. **Raw Data:** Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.
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- 6.25. Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.
- 6.26. Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.
- 6.27. Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.
- 6.28. Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

7. INTERFERENCES

- 7.1. Contamination by carryover can occur whenever high and low concentration level samples are analyzed sequentially. Suspected high level samples should be diluted and then analyzed at the end of the sequence to prevent carryover contamination. In addition, sample syringes should be thoroughly rinsed with solvent between sample injections. Additional cleanup by silica gel column may be required.
 - 7.2. Interference can also occur when "dirty" samples leave residue in the injector or analytical column. To minimize this effect, a guard column should be used and cut frequently or replaced. Also, the analytical column can be "baked" after such samples.
 - 7.3. Solvents, reagents, glassware, and other sample processing equipment may yield discrete contaminants. This can lead to spurious peaks and/or an elevated baseline, resulting in possible misinterpretation of chromatograms.
 - 7.4. Endrin and 4,4'-DDT are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated with high boiling residue from sample injection or when the injector contains metal fittings.
 - 7.4.1. Endrin and 4,4'-DDT breakdown to endrin aldehyde, endrin ketone, 4,4'-DDD, or 4,4'-DDE.
 - 7.4.2. When such breakdown is observed, the corrective action may include, but is not limited to 1) cleaning and deactivating the injection port, 2) replacing the injection port liner, or 3) clipping the guard column.
 - 7.5. Waxes, lipids, and other high molecular weight materials can be removed by EPA Method 3640A, Gel-Permeation Cleanup.
 - 7.6. Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides.
 - 7.6.1. Certain co-eluting organophosphorus pesticides may be eliminated by the pesticide option of EPA Method 3640A.
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- 7.6.2. Co-eluting chlorophenols may be eliminated by EPA Methods 3630C (Silica Gel Cleanup), 3620C (Florisil Cleanup), or 3610B (Alumina Cleanup).
- 7.7. Polychlorinated biphenyls (PCBs) may interfere with the analysis of the organochlorine pesticides. The problem may be most severe for the analysis of multiple-component analytes such as chlordane and toxaphene. If PCBs are known or expected to occur in samples, consult EPA Methods 3620C and 3630C to separate the pesticides from the PCBs
- 7.8. Glassware must be scrupulously cleaned. The glassware should then be drained dry and heated in a muffle furnace at 550° C for 8 hours.
- 7.9. Plastics contain significant amounts of leachable phthalate esters and must not be used during any stage of analytic processing.
- 7.10. The following provides information regarding possible target analyte losses/interferences during analytic processing:
 - 7.10.1. During sample transport and analysis, heat, ozone, NO₂, and ultraviolet (UV) light may cause sample degradation. Incandescent or UV-shielded fluorescent lighting in the laboratory should be used during analysis.

8. SAFETY

- 8.1. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current version of Calscience's Health, Safety, and Respiratory Protection Manual. In general, safety glasses and laboratory coats are required to be worn in all designated laboratory areas. Protective gloves shall be worn when handling chemicals.
- 8.2. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.
- 8.3. All the Pesticide compounds covered by this method have been tentatively classified as known or suspected human carcinogens: Primary standards of these toxic compounds must be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when analysts handle high concentrations of these compounds.

9. EQUIPMENT AND SUPPLIES

- 9.1. Gas Chromatograph: Agilent 6890N Gas Chromatograph, Agilent 7890A Gas Chromatograph, or equivalent configured with the following components.
 - 9.1.1. Autoinjector, Agilent 7683 Series, Agilent 7683B Series, or equivalent.
 - 9.2. Instrument Software
 - 9.2.1. Requires a PC based data system or equivalent.
 - 9.2.2. Agilent Environmental MSD ChemStation Version E.02 or equivalent.
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9.3. Instrument Maintenance and Troubleshooting

9.3.1. Refer to the current revision of SOP-T066 for instrument maintenance and troubleshooting.

9.3.2. Additional information can be found in the user manual or operating guide for the specific instrument.

9.4. Primary Detection Channel

9.4.1. Detector: Electron capture detector (ECD).

9.4.2. Analytical Column: 30-m × 0.32-mm ID, 0.50- μ m film thickness, narrow-bore, capillary, silicone coated fused-silica, Restek Rtx[®]-CLPesticides or equivalent.

9.5. Confirmation Detection Channel

9.5.1. Detector: Electron capture detector (ECD).

9.5.2. Analytical Column: 30-m × 0.32-mm ID, 0.25- μ m film thickness, narrow-bore, capillary, silicone coated fused-silica, Restek Rtx[®]-CLPesticides2 or equivalent.

9.6. Guard Column: 5-m × 0.32-mm ID, intermediate-polarity deactivated, uncoated fused-silica, Restek IP Deactivated Guard Column or equivalent.

9.7. Carrier Gas: Nitrogen, N₂, high purity (99.998%), compressed, Praxair 4.8 grade or equivalent.

9.8. Makeup Gas: Nitrogen, N₂, compressed, Praxair 4.8 grade or equivalent.

9.9. Syringes, 10- μ L, 25- μ L, 50- μ L, 100- μ L, 250- μ L, and 500- μ L, gastight, Cemented Needle (N) termination, Hamilton 1700 Series or equivalent with NIST Traceable Certificate or equivalent documentation.

9.10. Storage vials, 15-mm × 45-mm (4-mL capacity), screw top, clear glass, with Teflon-lined screw caps and septa, disposable.

9.11. Autoinjector vials, 12-mm × 32-mm (2-mL capacity), crimp top, clear glass, with aluminum crimp caps and Teflon-lined septa, disposable.

9.12. Vial inserts, 300- μ L, clear glass, with conical bottom and spring.

9.13. Balance, analytical, calibrated, capable of weighing to the nearest 0.1 mg.

9.14. Refer to the specific SOPs of the preparatory methods for additional equipment and supplies.

9.15. Quartz fiber filter: 102 mm binderless quartz microfiber filter.

9.16. Polyurethane foam (PUF) plugs: 3-inch thick sheet rock polyurethane type. The PUF cylinders should be slightly larger in diameter than the internal diameter of the cartridge.

9.17. ► **Concentrator/Drying system: Horizon Technology model DryVap or equivalent.**

10. REAGENTS AND STANDARDS

10.1. Reagents

- 10.1.1. Methylene chloride, CH₂Cl₂, pesticide grade or equivalent.
- 10.1.2. Diethyl Ether, C₄H₁₀O, pesticide grade or equivalent.
- 10.1.3. Hexane, C₆H₁₄, pesticide grade or equivalent.
- 10.1.4. Acetone, CH₃COCH₃, clear colorless liquid, pesticide grade or equivalent.
- 10.1.5. All reagents must be inspected, verified and documented prior to use.

10.2. Standards

Pesticides:

- 10.2.1. Pre-certified stock standard solutions, each in sealed glass ampules, containing various concentrations of single-component and multi-component pesticide target analytes, and 200 ppm of each surrogate are used to prepare calibration and check standards.

10.2.1.1. Prepare each working standard solution by diluting the appropriate volumes of the stock standards to the specified volumes with hexane.

10.2.1.2. The routine single-component pesticide working standards are prepared as follows:

Routine Analyte	Initial		Final	
	Conc. (ppm)	Volume (μL)	Conc. (ppm)	Volume (mL)
OC pesticides	2000	8.0	4.0	4.0
hexachlorobenzene	100	160		
surrogates	200	160	8.0	

Routine Analyte	Initial		Final	
	Conc. (ppm)	Volume (μL)	Conc. (ppm)	Volume (mL)
OC pesticides	200	80	4.0	4.0
hexachlorobenzene	100	160		
surrogates	200	160	8.0	

10.2.1.3. The non-routine single-component pesticide working standards are prepared as follows:

Non-Routine Analyte	Initial		Final	
	Conc. (ppm)	Volume (µL)	Conc. (ppm)	Volume (mL)
2,4'-DDD	100	160	2.0	8.0
2,4'-DDE	100		2.0	
2,4'-DDT	100		2.0	
mirex	100		2.0	
cis-nonachlor	100		2.0	
trans-nonachlor	100		2.0	
oxychlorane	100		2.0	
4,4'-DCBP	500		160	
kepone	1000	8.0	2.0	4.0
chlorobenzilate	1000	8.0	2.0	4.0
diallate	1000		2.0	
hexachlorocyclopentadiene	1000		2.0	
surrogates	200		80	

Non-Routine Analyte	Initial		Final	
	Conc. (ppm)	Volume (µL)	Conc. (ppm)	Volume (mL)
2,4'-DDD	100	160	2.0	8.0
2,4'-DDE	100		2.0	
2,4'-DDT	100		2.0	
mirex	100		2.0	
cis-nonachlor	100		2.0	
trans-nonachlor	100		2.0	
oxychlorane	100		2.0	
4,4'-DCBP	500		160	
kepone	100	80	2.0	4.0
chlorobenzilate	100	80	2.0	4.0
diallate	100		2.0	
hexachlorocyclopentadiene	100		2.0	
surrogates	200		80	

10.2.2. Pre-certified stock standard solution, in sealed glass ampule, containing 200 ppm each of decachlorobiphenyl (DCB) and 2,4,5,6-tetrachloro-m-xylene (TMX) is used to prepare surrogate working standard.

10.2.2.1. Prepare the 2.0-ppm surrogate working standard solution by diluting 10 mL of the 200-ppm surrogate stock standard to 1.0 L with acetone or other acetone miscible solvent.

10.2.3. Pre-certified stock standard solutions, each in sealed glass ampules, containing various concentrations of target analytes are used to prepare spike working standards.

10.2.3.1. Prepare each 1.0-ppm spike working standard solution by diluting the appropriate volumes of the stock standards to the specified volumes with acetone or other acetone miscible solvent.

10.2.3.2. The 1.0-ppm spike working standards are prepared as follows:

Routine Analyte	Initial		Final	
	Conc. (ppm)	Volume (mL)	Conc. (ppm)	Volume (mL)
OC pesticides	200	1.0	1.0	200
hexachlorobenzene	1000	0.2		

Non-Routine Analyte	Initial		Final	
	Conc. (ppm)	Volume (μL)	Conc. (ppm)	Volume (mL)
2,4'-DDD	100	100	1.0	10
2,4'-DDE	100		1.0	
2,4'-DDT	100		1.0	
mirex	100		1.0	
cis-nonachlor	100		1.0	
trans-nonachlor	100		1.0	
oxychlorodane	100		1.0	
4,4'-DCBP	500		100	
kepone	100	100	1.0	10
chlorobenzilate	100	100	1.0	10
diallate	100		1.0	
hexachlorocyclopentadiene	100		1.0	

10.2.4. Degradation test stock standard solution containing 500 ppm each of 4,4'-DDT and endrin in methanol.

10.2.4.1. Prepare the 500-ppb degradation test working standard solution by diluting 10 μL of the 500-ppm degradation test stock standard to 10 mL with hexane.

10.2.4.2. Prepare the 50-ppb degradation test working standard solution by diluting 4.0 mL of the 500-ppb degradation test working standard to 40 mL with hexane.

10.2.4.3. Inject 1.0 μL of the 50-ppb degradation test working standard for degradation test.

10.2.5. Calibration standard solutions containing various concentrations of target analytes and surrogates in hexane.

10.2.5.1. Dilute the appropriate volumes of the working and stock standards to the specified volumes with hexane for initial calibration.

10.2.5.2. Use the following calibration levels as guidance to prepare the routine single-component pesticide calibration standards.

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A1	S	A1 + S	A1 + S	A1 + S
10	20	4.0 + 8.0	10	4.0
20	40	4.0 + 8.0	20	4.0
40	80	4.0 + 8.0	40	4.0
60	120	4.0 + 8.0	60	4.0
80	160	4.0 + 8.0	80	4.0

Note: A1 = Routine Single-Component Analyte; S = Surrogate

10.2.5.3. Use the following calibration levels as guidance to prepare the non-routine single-component pesticide calibration standards.

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A2	A3	A2 + A3	A2 + A3	A2 + A3
10	50	2.0 + 10	40	8.0
20	100	2.0 + 10	80	8.0
40	200	2.0 + 10	160	8.0
60	300	2.0 + 10	240	8.0
80	400	2.0 + 10	320	8.0

Note: A2 = Non-Routine Single-Component Analyte; A3 = 4,4'-DCBP

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A4	S	A4 + S	A4 + S	C
10	20	2.0 + 4.0	20	4.0
20	40	2.0 + 4.0	40	4.0
40	80	2.0 + 4.0	80	4.0
60	120	2.0 + 4.0	120	4.0
80	160	2.0 + 4.0	160	4.0

Note: A4 = Kepone, Chlorobenzilate, Diallylate, or Hexachlorocyclopentadiene
S = Surrogate

10.2.5.4. The midpoint standards are also used as the continuing calibration verification solutions.

10.2.6. Initial calibration verification (ICV) solutions containing the appropriate concentrations of each target analyte and surrogate in hexane. The ICV solution must be of a source differing from that used for the initial multi-point calibration. If it is of the same source, then it must be of different lot.

10.2.6.1. Dilute the appropriate volumes of the second source working and stock standards to the specified volumes with hexane for initial calibration verification.

10.2.6.2. Use the following calibration level as guidance to prepare the routine single-component pesticide ICV solution.

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A1	S	A1 + S	A1 + S	A1 + S
40	80	4.0 + 8.0	40	4.0

Note: A1 = Routine Single-Component Analyte; S = Surrogate

10.2.6.3. Use the following calibration levels as guidance to prepare the non-routine single-component pesticide ICV solutions.

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A2	A3	A2 + A3	A2 + A3	A2 + A3
40	200	2.0 + 10	160	8.0

Note: A2 = Non-Routine Single-Component Analyte; A3 = 4,4'-DCBP

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A4	S	A4 + S	A4 + S	C
40	80	2.0 + 4.0	80	4.0

Note: A4 = Kepone, Chlorobenzilate, Diallate, or Hexachlorocyclopentadiene
S = Surrogate

10.2.7. Continuing calibration verification (CCV) solutions containing the appropriate concentrations of each target analyte and surrogate in hexane. The CCV solution is of a source same as that used for the initial multi-point calibration.

10.2.7.1. Dilute the appropriate volumes of the working and stock standards to the specified volumes with hexane for continuing calibration verification.

10.2.7.2. Use the following calibration level as guidance to prepare the routine single-component pesticide CCV solution.

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A1	S	A1 + S	A1 + S	A1 + S
40	80	4.0 + 8.0	400	40

Note: A1 = Routine Single-Component Analyte; S = Surrogate

10.2.7.3. Use the following calibration levels as guidance to prepare the non-routine single-component pesticide CCV solutions.

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A2	A3	A2 + A3	A2 + A3	A2 + A3
40	200	2.0 + 10	160	8.0

Note: A2 = Non-Routine Single-Component Analyte; A3 = 4,4'-DCBP

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A4	S	A4 + S	A4 + S	C
40	80	2.0 + 4.0	80	4.0

Note: A4 = Kepone, Chlorobenzilate, Diallate, or Hexachlorocyclopentadiene
S = Surrogate

10.2.8. Surrogate working standard solution containing 2.0 ppm each of deca-chlorobiphenyl (DCB) and 2,4,5,6-tetrachloro-m-xylene (TMX) in acetone or other acetone miscible solvent.

10.2.8.1. Add 500 µL of the 2.0-ppm surrogate working standard to each sample including each quality control (QC) check sample and method blank prior to solvent extraction.

10.2.9. Spike working standard solutions containing various concentrations of target analytes in acetone or other acetone miscible solvent. The spike standard solution must be of a source differing from that used for the initial multi-point calibration. If it is of the same source, then it must be of different lot.

10.2.9.1. Use the 1.0-ppm spike working standard solutions as the single-component pesticide spike working standard solutions. The spike standards are used to prepare QC check samples such as matrix spikes (MS/MSDs) and laboratory control samples (LCS/LCSDs).

10.2.9.2. Add 500 µL of the single-component pesticide spike working standard to each MS/MSD and LCS/LCSD sample prior to solvent extraction.

10.2.10. All working standards must be replaced after six months (unless specified otherwise) or sooner if routine QC or comparisons with check standards indicates a problem.

10.2.10.1. Store all working standards under dark and refrigerated condition.

10.2.11. All stock standards must be inspected and documented in the Chemicals and Supplies Verification Logbook prior to use.

- 10.2.11.1. Check all opened stock standards frequently for signs of degradation or evaporation.

PCBs:

- 10.2.12. Pre-certified stock standard solutions, each in sealed glass ampules, containing 100/1000 ppm of each target analyte, and 200 ppm of each surrogate are used to prepare calibration and check standards.

- 10.2.12.1. Prepare each working standard solution by diluting the appropriate volumes of the stock standards to the specified volumes with hexane.

- 10.2.12.2. The 20-ppm working standards are prepared as follows:

Analyte	Initial		Final	
	Conc. (ppm)	Volume (µL)	Conc. (ppm)	Volume (mL)
Aroclor 1016	100	800	20	4.0
Aroclor 1260	100	800		
surrogates	200	80	4.0	

Analyte	Initial		Final	
	Conc. (ppm)	Volume (µL)	Conc. (ppm)	Volume (mL)
Aroclor 1016	1000	80	20	4.0
Aroclor 1260	1000	80		
surrogates	200	80	4.0	

Analyte	Initial		Final	
	Conc. (ppm)	Volume (µL)	Conc. (ppm)	Volume (mL)
Aroclor 1221	100	800	20	4.0
Aroclor 1254	100	800	20	4.0
Aroclor 1232	100	800	20	4.0
Aroclor 1262	100	800	20	4.0
Aroclor 1248	100	800	20	4.0
Aroclor 1268	100	800	20	4.0
Aroclor 1242	100	800	20	4.0
surrogates	200	80	4.0	4.0

10.2.12.3. The 500-ppb working standard is prepared as follows:

Analyte	Initial		Final	
	Conc. (ppm)	Volume (µL)	Conc. (ppm)	Volume (mL)
Aroclor 1221	100	800	20	4.0
Aroclor 1254	100	800	20	4.0
Aroclor 1232	100	800	20	4.0
Aroclor 1262	100	800	20	4.0
Aroclor 1248	100	800	20	4.0
Aroclor 1268	100	800	20	4.0

10.2.13. Pre-certified stock standard solution, in sealed glass ampule, containing 200 ppm each of decachlorobiphenyl (DCB) and 2,4,5,6-tetrachloro-m-xylene (TMX) is used to prepare surrogate working standard.

10.2.13.1. Prepare the 2.0-ppm surrogate working standard solution by diluting 10 mL of the 200-ppm surrogate stock standard to 1.0 L with acetone or other acetone miscible solvent.

10.2.14. Pre-certified stock standard solutions, each in sealed glass ampules, containing 100/1000 ppm of each target analyte are used to prepare spike working standards.

10.2.14.1. Prepare each 10-ppm spike working standard solution by diluting the appropriate volumes of the stock standards to the specified volumes with acetone or other acetone miscible solvent.

10.2.14.2. The 10-ppm spike working standards are prepared as follows:

Analyte	Initial		Final	
	Conc. (ppm)	Volume (mL)	Conc. (ppm)	Volume (mL)
Aroclor 1016	1000	2.0	10	200
Aroclor 1260	1000	2.0		
Aroclor 1221	100	1.0	10	10
Aroclor 1254	100	1.0	10	10
Aroclor 1232	100	1.0	10	10
Aroclor 1262	100	1.0	10	10
Aroclor 1248	100	1.0	10	10
Aroclor 1268	100	1.0	10	10
Aroclor 1242	100	1.0	10	10

10.2.15. Calibration standard solution containing various concentrations of target analytes and surrogates in hexane.

10.2.15.1. Dilute the appropriate volumes of the 20-ppm working standards to the specified volumes with hexane for initial calibration.

10.2.15.2. Use the following calibration levels as guidance to prepare the calibration standards.

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A	S	A + S	A + S	A + S
100	20	20 + 4.0	20	4.0
250	50	20 + 4.0	50	4.0
500	100	20 + 4.0	1000	40
750	150	20 + 4.0	150	4.0
2000	400	20 + 4.0	400	4.0

Note: A = Aroclor; S = Surrogate

10.2.15.3. The midpoint standard is also used as the continuing calibration verification solution.

10.2.16. Initial calibration verification (ICV) solution containing 500 ppb of each target analyte and 100 ppb of each surrogate in hexane. The ICV solution must be of a source differing from that used for the initial five-point calibration. If it is of the same source, then it must be of different lot.

10.2.16.1. Dilute 100 µL of the second source 20-ppm working standard to 4.0 mL with hexane for initial calibration verification.

10.2.16.2. Use the following calibration level as guidance to prepare the ICV solution.

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A	S	A + S	A + S	A + S
500	100	20 + 4.0	100	4.0

Note: A = Aroclor; S = Surrogate

10.2.17. Continuing calibration verification (CCV) solution containing 500 ppb of each target analyte and 100 ppb of each surrogate in hexane. The CCV solution is of a source same as that used for the initial five-point calibration.

10.2.17.1. Dilute 1000 µL of the 20-ppm working standard to 40 mL with hexane for continuing calibration verification.

10.2.17.2. Use the following calibration level as guidance to prepare the CCV solution.

Calibration Level (ppb)		Initial		Final
		Concentration (ppm)	Volume (µL)	Volume (mL)
A	S	A + S	A + S	A + S
500	100	20 + 4.0	1000	40

Note: A = Aroclor; S = Surrogate

- 10.2.18. Surrogate working standard solution containing 2.0 ppm each of deca-chlorobiphenyl (DCB) and 2,4,5,6-tetrachloro-m-xylene (TMX) in acetone or other acetone miscible solvent.
- 10.2.18.1. Add 500 µL of the 2.0-ppm surrogate working standard to each sample including each quality control (QC) check sample and method blank prior to solvent extraction.
- 10.2.18.2. Add 500 µL of the 2.0-ppm surrogate working standard to each mobility-procedure extract including each mobility-procedure extract designated as QC check sample and method blank prior to solvent extraction.
- 10.2.19. Spike working standard solution containing 10 ppm of each target analyte in acetone or other acetone miscible solvent. The spike standard solution must be of a source differing from that used for the initial five-point calibration. If it is of the same source, then it must be of different lot.
- 10.2.19.1. Use the 10-ppm spike working standard solution containing only Aroclor 1016 and Aroclor 1260 if samples are not expected to contain any Aroclor. Use the 10-ppm spike working standard solution containing the specific Aroclor(s) if samples are expected to contain these Aroclor(s).
- 10.2.19.2. The spike standards are used to prepare QC check samples such as matrix spikes (MS/MSDs) and laboratory control samples (LCS/LCSDs).
- 10.2.19.3. Add 200 µL of the spike working standard containing only Aroclor 1016 and Aroclor 1260 to each MS/MSD and LCS/LCSD sample prior to solvent extraction.
- 10.2.19.4. Per client request or project specific data quality objectives (DQOs), add 200 µL of the spike working standard containing the specific Aroclor(s) to each MS/MSD and LCS/LCSD sample prior to solvent extraction.
- 10.2.19.5. Add 200 µL of the spike working standard containing only Aroclor 1016 and Aroclor 1260 to each mobility-procedure extract designated as MS/MSD and LCS/LCSD prior to solvent extraction.
- 10.2.19.6. Per client request or project specific DQOs, add 200 µL of the spike working standard containing the specific Aroclor(s) to each mobility-procedure extract designated as MS/MSD and LCS/LCSD prior to solvent extraction.

10.2.20. All working standards must be replaced after six months (unless specified otherwise) or sooner if routine QC or comparisons with check standards indicates a problem.

10.2.20.1. Store all working standards under dark and refrigerated condition.

10.2.21. All stock standards must be inspected and documented in the Chemicals and Supplies Verification Logbook prior to use.

10.2.21.1. Check all opened stock standards frequently for signs of degradation or evaporation.

11. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

11.1. Deployment of Cartridges for Field Sampling: At least one PUF cartridge assembly and one filter from each batch of approximately twenty should be tested for the compounds of interest to serve as a process blank and certified before the batch is considered for field use. See Appendix A for Certification procedure.

11.2. During each sampling episode, at least one filter/PUF cartridge should be shipped to the field and returned, without drawing air through the sampler, to serve as a field blank.

11.3. Fill out a "chain-of-custody" indicating cartridge number, date of certification, etc. The chain-of-custody must accompany the cartridge to the field and return to the laboratory.

11.4. During the analysis of each batch of samples, at least one solvent process blank (all steps conducted with filter/PUF cartridge included) should be carried through the procedure and analyzed.

11.5. Levels for process, field and solvent blanks should not exceed 100 ng/sample for single components.

11.6. Fill out a "chain-of-custody" indicating cartridge number, date of certification, etc. The chain-of-custody must accompany the cartridge to the field and return to the laboratory.

11.7. Cartridges are placed in their shipping containers and shipped to the field.

11.8. After collection, the chilled (0-6°C) samples are returned in the aluminum shipping container (containing the filter and PUF) to the laboratory for analysis. The "chain-of-custody" should be completed. The samples are logged in the laboratory according to sample location, filter and sorbent cartridge number identification, and total air volume sampled (uncorrected). If the time span between sample collection and laboratory is to exceed 24 hours, refrigerate sample. All samples should be extracted within 7 days. Prior to extraction, the cartridges are spiked with surrogate compounds to monitor matrix effects, breakthrough, etc. Add surrogate compounds to the center of the PUF cartridge, using a micro syringe.

11.9. All extracted samples are then stored under dark and refrigerated (4°C) conditions and must be analyzed within a 40-day period post extraction.

12. QUALITY CONTROL

12.1. Degradation Test

- 12.1.1. Prior to running the calibration standards, the degradation test standard solution must be analyzed and meet the defined acceptance criteria.
- 12.1.2. The following criteria must be demonstrated every 12 hours.
 - 12.1.2.1. The degradation (or percent breakdown) of 4,4'-DDT and endrin shall be $\leq 15\%$ for each compound. The formula for calculating %B is listed in Section 15.14.
- 12.1.3. If these criteria are not met, then the analytical system is deemed unacceptable for sample analysis to begin. Effect corrective actions, rerun the degradation test, perform injector maintenance, and recalibrate.

12.2. Initial Calibration (IC)

- 12.2.1. The initial multi-point calibration must be established prior to the processing of sample extracts.
 - 12.2.1.1. The calibration curve is established with a minimum of five calibration standards, but may contain six or seven calibration standards.
 - 12.2.1.1.1. A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other Aroclor mixtures. Hence, it is not necessary to establish the initial five-point calibration for each of the other Aroclors.
 - 12.2.1.1.2. In situations where only a few Aroclors are of interest for a specific project, it will be necessary to establish the initial five-point calibration for each Aroclor of interest.
 - 12.2.1.2. If the calibration curve is not established for each Aroclor other than Aroclor 1016 and Aroclor 1260, analyze the 500-ppb working standards for pattern recognition.
 - 12.2.1.2.1. The 500-ppb working standards may also be used to determine the single-point calibration factor for each Aroclor if both of the following conditions are met.
 - 12.2.1.2.1.1. The linearity of the detector response is demonstrated using the calibration standards containing only Aroclor 1016 and Aroclor 1260.
 - 12.2.1.2.1.2. The calibration option is linear least squares regression and the regression is forced through zero.

- 12.2.2. The IC is deemed valid if the %RSD for each analyte is $\leq 20\%$.
- 12.2.3. If the %RSD for an analyte is $\leq 20\%$, the response factor (RF) is assumed to be constant over the calibration range, and the average RF may be used for quantitation.
- 12.2.4. If the %RSD criterion for an analyte is not met, apply one of the alternative calibration options.
- 12.2.4.1. The first calibration option is linear least squares regression with equal weighting factor. The IC is deemed valid if the correlation coefficient, r , is ≥ 0.99 .
- 12.2.4.1.1. For some calibrations, it may be appropriate to force the regression through zero. Forcing the curve through zero is not the same as including the origin (0, 0) as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 before the regression is calculated, thereby setting the bias to favor the low end of the calibration range by "pivoting" the function around the origin to find the best fit and resulting in one less degree of freedom. However, forcing the regression through zero must not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of calibration standards.
- 12.2.4.1.2. Rationale for forcing the regression through zero must be documented and approved by the Group Leader.
- 12.2.4.2. The second calibration option is linear least squares regression with inverse of concentration weighting factor. The IC is deemed valid if the coefficient of determination, r^2 , is ≥ 0.99 .
- 12.2.4.2.1. This option allows a better fitting of the points at the lower calibration levels.
- 12.2.4.3. The third calibration option is linear least squares regression with inverse square of concentration weighting factor. The IC is deemed valid if the coefficient of determination, r^2 , is ≥ 0.99 .
- 12.2.4.3.1. This option also allows a better fitting of the points at the lower calibration levels.
- 12.2.4.4. The fourth calibration option is quadratic least squares regression with equal weighting factor. The IC is deemed valid if the coefficient of determination, r^2 , is ≥ 0.99 .
- 12.2.4.4.1. This option requires at least six calibration levels.
- 12.2.5. If these criteria are not met, then the calibration is unacceptable for sample analysis to begin. Effect corrective action and recalibrate.
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12.2.5.1. If the RSD or correlation of any analyte is unacceptable, review the results (e.g., proper identification, area count, response factor, etc.) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards.

12.2.5.2. If the problem appears to be associated with a single calibration standard, then that one standard may be reanalyzed once within the same analytical shift prior to sample analysis to rule out problems due to random chance.

12.2.5.2.1. In some cases, replace the calibration standard may be necessary.

12.2.5.3. If a calibration standard is replaced and/or reanalyzed, recalculate the RSD or correlation, and document the rationale for re-analysis.

12.3. Initial Calibration Verification (ICV)

12.3.1. The initial calibration is deemed valid if the %D for each analyte is $\leq 20\%$.

12.3.1.1. If the calibration option is average response, the %D is the percent difference.

12.3.1.2. If the calibration option is linear or quadratic least squares regression, the %D is the percent drift.

12.3.2. If these criteria are not met, the initial calibration is deemed unacceptable for sample analysis to begin. An unacceptable ICV result indicates either a disagreement between like solutions from separate sources or a change in instrument conditions. Normally, this is caused when at least one of the solutions is no longer intact (representative of the stated concentration). Document the unacceptable result and reanalyze the ICV within 2 hours after the failed ICV. If the ICV criteria remain unacceptable, investigate, effect corrective action, which may include re-preparation of standard solutions or instrument maintenance, and recalibrate.

12.4. Continuing Calibration Verification (CCV)

12.4.1. Following the establishment of a valid initial calibration, a CCV standard must be analyzed daily prior to sample analysis, after every batch of 20 samples or portion thereof within a 12-hour shift, and at the end of sequence.

12.4.2. The initial calibration is deemed valid if the %D for each analyte is $\leq 20\%$.

12.4.2.1. If the calibration option is average response, the %D is the percent difference.

12.4.2.2. If the calibration option is linear or quadratic least squares regression, the %D is the percent drift.

12.4.3. If these criteria are not met, the initial calibration is deemed unacceptable for sample analysis to resume. Document the unacceptable result and

reanalyze the CCV within 2 hours after the failed CCV. If the CCV criteria remain unacceptable, effect corrective action and recalibrate.

12.5. Retention Time Window

12.5.1. Establishment of retention time window width is accomplished by making three injections of CCV standards throughout the course of a 72-hour period. Serial injections over a shorter period of time may result in narrow retention time window width that does not accurately account for variations over several days.

12.5.1.1. Retention time window width is $\pm 3S$ (where S is the standard deviation of the three retention times for that analyte/surrogate) or ± 0.030 minute, whichever is greater.

12.5.1.1.1. For each multi-component analyte (i.e., chlordane and toxaphene), calculate the standard deviation for each one of the five major characteristic peaks.

12.5.2. Establishment of retention time window position is accomplished by using the midpoint calibration standard once per initial calibration, and by using a CCV standard at the beginning of an analytical sequence.

12.5.2.1. When initial calibration is performed, daily retention time window for each analyte/surrogate is the retention time of the analyte/surrogate in the midpoint calibration standard $\pm 3S$ or ± 0.030 minute, whichever is greater.

12.5.2.2. When initial calibration is not performed, daily retention time window for each analyte/surrogate is the retention time of the analyte/surrogate in the CCV standard $\pm 3S$ or ± 0.030 minute, whichever is greater.

12.5.3. Retention time for each analyte/surrogate in the calibration verification standard is verified as follows:

12.5.3.1. When initial calibration is performed, the ICV standard and all CCV standards throughout the course of an analytical sequence within a 12-hour shift must fall within the daily retention time window established by the midpoint calibration standard.

12.5.3.2. When initial calibration is not performed, all succeeding CCV standards throughout the course of an analytical sequence within a 12-hour shift must fall within the daily retention time window established by the first CCV standard.

12.5.3.3. If these criteria are not met, determine the cause of the problem, effect corrective action, and re-establish the retention time window width and/or position, if necessary.

12.6. Event Based Quality Control (MBs and LCS/LCSDs)

12.6.1. Event based quality control consists of QC samples prepared and processed with each preparatory event. This consists of a method blank (MB) and a laboratory control sample and laboratory control sample duplicate (LCS/LCSD).

12.6.2. The acceptance criteria for MBs are as follows:

12.6.2.1. Ideally, the concentrations of target analytes in an MB should be less than the respective reporting limits (RLs). If the concentration of any target analyte exceeds its RL, the source of contamination must be investigated and, if possible, eliminated.

12.6.2.2. If a target analyte is found in the MB, but not in the associated samples, report the sample and MB data without qualification.

12.6.2.3. If a target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified or rejected and the samples re-processed and/or re-analyzed.

12.6.3. The acceptance criteria for LCS/LCSD compounds are as follows:

12.6.3.1. The lower and upper acceptance limits for %REC of each LCS/LCSD compound are 50% and 135%, respectively. The RPD is $\leq 25\%$.

12.6.3.1.1. If historical data is available, the lower and upper acceptance limits for %REC and RPD of each LCS/LCSD compound are based upon the historical average recovery $\pm 3S$ that is updated at least annually.

12.6.3.2. All LCS/LCSD compounds must be within acceptance limits. However, if a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside of control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary. Lower and upper marginal exceedance (ME) limits can be established to determine when corrective action is necessary.

12.6.3.3. ME is defined as being beyond the LCS control limit (3 standard deviations), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.

12.6.3.4. The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte lists with fewer than 11 analytes.

12.6.3.5. The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number of Analytes Allowed in ME of the LCS Control Limit
> 90	5
71 - 90	4
51 - 70	3
31 - 50	2
11 - 30	1
< 11	0

12.6.3.6. Marginal exceedances must be random. If the same analyte exceeds the LCS control limit 2 out of 3 consecutive LCS, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.

12.7. Matrix Based Quality Control (Surrogates and MS/MSDs)

12.7.1. Matrix based quality control consists of QC samples prepared and processed using actual environmental samples. This consists of a matrix spike and matrix spike duplicate (MS/MSD) and surrogates added to each sample.

12.7.2. The acceptance criteria for surrogate compounds are as follows:

12.7.2.1. The lower and upper acceptance limits for %REC of each surrogate compound are 50% and 135%, respectively

12.7.2.1.1. If historical data is available, the lower and upper acceptance limits for %REC of each surrogate compound are based upon the historical average recovery $\pm 3S$ that is updated at least annually.

12.7.2.1.2. For EPA Region 9 requirement, the lower and upper acceptance limits for %REC of each surrogate compound are 60% and 150%, respectively.

12.7.2.2. If the surrogate compound recoveries are acceptable, report the surrogate and sample data without qualification.

12.7.2.3. If one or more surrogate recoveries are not acceptable, evaluation is not necessarily straightforward. The sample itself may produce effects due to factors such as interferences and high analyte concentration or a problem may have occurred during extraction or cleanup. The data alone cannot be used to evaluate the precision and accuracy of individual sample analysis. However, when exercising professional judgment, this data should be used in conjunction with other available QC information.

12.7.2.4. By itself, unacceptable surrogate recoveries do not invalidate sample data. The following must be accomplished if surrogate recoveries are not acceptable.

- 12.7.2.4.1. Check the surrogate standard solutions for degradation and contamination.
 - 12.7.2.4.2. If the nonconformance is due to poor instrument performance or if the above actions fail to reveal the cause of the unacceptable surrogate recoveries, the same extract should be re-analyzed.
 - 12.7.2.4.3. If incorrect procedures or degraded/contaminated standard solutions are determined to have not caused the unacceptable surrogate recoveries, the affected sample(s) must be re-processed and re-analyzed or, if insufficient sample remains, reference made to the associated MB surrogate recoveries and the sample data reported with qualification.
 - 12.7.2.4.3.1. If, upon re-processing and re-analysis, the surrogates remain unacceptable, matrix interference can be cited and reference made to the associated MB surrogate recoveries and the sample data reported with qualification.
 - 12.7.2.4.3.2. If the MB surrogates are unacceptable, all associated sample data must be invalidated and all associated samples re-processed and re-analyzed.
 - 12.7.2.5. Where sample dilution is required, depending on the dilution factor, the surrogate recovery will be low or not detected. This is an expected occurrence and reference should be made to the MB surrogate recovery which must be reported to the client.
 - 12.7.3. The acceptance criteria for MS/MSD compounds are as follows:
 - 12.7.3.1. The lower and upper acceptance limits for %REC of each MS/MSD compound are 50% and 135%, respectively. The RPD is $\leq 25\%$.
 - 12.7.3.1.1. If historical data is available, the lower and upper acceptance limits for %REC and RPD of each MS/MSD compound are based upon the historical average recovery $\pm 3S$ that is updated at least annually.
 - 12.7.3.1.2. For EPA Region 9 requirement, the lower and upper acceptance limits for %REC of each MS/MSD compound are 50% and 135%, respectively. The RPD is $\leq 30\%$.
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- 12.7.3.2. When the %REC and RPD of the MS/MSD compounds are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.
- 12.7.3.3. If the %REC and/or RPD of the MS/MSD compounds are not within the established acceptance limits, the analytical system performance shall be suspect.
- 12.7.4. Unacceptable %REC values are typically caused by matrix effects or poor instrument performance/technique. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.
- 12.8. If the %REC or RPD of the MS/MSD and LCS/LCSD are unacceptable, all associated sample data must be invalidated and all associated samples re-processed and re-analyzed.
- 12.9. Additional information regarding internal quality control checks is provided in SOP-T020.

13. CALIBRATION AND STANDARDIZATION

- 13.1. Analytical Balance
- 13.1.1. Calibrate the analytical balance at 2 mg, 1 g, and 100 g using Class 2 weights as outlined in the current revision of SOP-T043.
- 13.1.2. If control limits are not specified, calibration shall be within $\pm 0.1\%$ or ± 0.5 mg, whichever is greater. If control limits are specified, calibration shall be within the specified limits. If the values are not within these limits, recalibrate the balance.
- 13.2. Chromatograph Degradation Test
- 13.2.1. Prior to initial calibration and the analysis of field or QC sample extracts, the GC system must be shown to be resistant to the breakdown of 4,4'-DDT and endrin. The acceptance criteria for the degradation test are listed in Section 12.1.
- 13.3. Chromatograph Initial Calibration
- 13.3.1. Establish an acceptable multi-point calibration curve. The acceptance criteria for the initial calibration are listed in Section 12.2.
- 13.3.1.1. Because of the sensitivity of the electron capture detector, always clean the injection port and column prior to performing the initial calibration.
- 13.3.1.2. Recalibration is required for the following maintenance procedures.

- 13.3.1.2.1. Change, replace, or reverse the analytical column.
- 13.3.2. After obtaining an acceptable multi-point calibration curve and prior to processing field or QC sample extracts, an ICV standard must be analyzed to verify the initial calibration. The acceptance criteria for the ICV are listed in Section 12.3.
- 13.3.3. The initial multi-point calibration and ICV shall include all anticipated target analytes for the duration of the use of the initial calibration.
- 13.4. Retention Time Window
- 13.4.1. Retention time window width for each analyte/surrogate is generated by running three CCV standards over a 72-hour period. Retention time window width determination shall be performed at method set-up, following column changes, after major instrument maintenance or when a significant retention time shift is suspected.
- 13.4.2. Document the serial number of the analytical column associated with the retention time window study.
- 13.4.3. Record the retention time in minutes for each analyte/surrogate to three decimal places.

14. ► PROCEDURE

14.1. *Extraction Procedure (PCBs)*

- 14.1.1. *The sample receiving department receives samples for analysis, they log-in PUF cartridge / Filter using CEL workorder and unique sample number using the engraved identifying number on the cartridge.*
- 14.1.2. *Remove the sample from the plastic container and then unwrap aluminum foil over the PUF cartridge.*
- 14.1.3. *Set up a soxhlet extraction apparatus by using a 1000 ml flask pour 700 ml of diethyl ether /hexane (10%v/v) add four to five clean boiling stone, attach a soxhlet extractor and Place the PUF cartridge/filter inside the soxhlet apparatus then set the unit on a heating mantle with temperature set between 60-70° C.*
- 14.1.4. *Assemble the soxhlet extraction apparatus for the complete batch of samples include QC (Method blank, Laboratory control sample and Laboratory control sample duplicate).*
- 14.1.5. *Prior to extraction add 0.1 ml of surrogate compounds (TCMX/Decachlorobiphenyl surrogate at 2 ppm) into the PUF cartridge in all laboratory samples, add .04 ml of spike standard compounds (Aroclor 1016/1260 Mix at 10 ppm) into the PUF cartridge for Laboratory control sample and Laboratory control sample duplicate.*
- 14.1.6. *Reflux the sample for 18 hours at a rate of at least 3 cycles per hour, allow cooling and then disassembling the apparatus.*
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14.1.7. Concentrate the extracted sample to 2 ml by using a DryVap Concentrator system from Horizon Technology, transfer the extract by pouring it to glass container on the concentrator unit, rinse the flask and column with about 30ml of 10% diethyl ether/hexane to complete the quantitative transfer. Prior to starting the Concentrator set dry volume to zero to complete the process at 2 ml, heater power to 1 for the temperature set at approximately 70° C then push the start button to start the concentration process.

14.1.8. After concentration is complete at 2 ml transfer the remaining sample to a 40ml glass vial.

14.1.9. All glassware that is used is rinsed with Hexane, washed with hot water containing detergent. Rinse with tap water and several partitions of distilled water. Drain, dry, and heat in a muffle furnace at 400 degree Celsius for 4 hours. After glassware is dry and cool, rinse it with Hexane.

14.2. Extraction Procedure (Pesticides)

14.2.1. The sample receiving department receives samples for analysis, they log-in PUF cartridge / Filter using CEL workorder and unique sample number using the engraved identifying number on the cartridge.

14.2.2. Remove the sample from the plastic container and then unwrap aluminum foil over the PUF cartridge.

14.2.3. Set up a soxhlet extraction apparatus by using a 1000 ml flask pour 700 ml of diethyl ether /hexane (10%v/v) add four to five clean boiling stone, attach a soxhlet extractor and Place the PUF cartridge/filter inside the soxhlet apparatus then set the unit on a heating mantle with temperature set between 60-70 ° C.

14.2.4. Assemble the soxhlet extraction apparatus for the complete batch of samples include QC (Method blank, Laboratory control sample and Laboratory control sample duplicate).

14.2.5. Prior to extraction add 0.10 ml of surrogate compounds (TCMX/Decachlorobiphenyl surrogate at 2 ppm) into the PUF cartridge in all laboratory samples, add 0.10 ml of spike standard compounds (orgeno-chlorine/pesticide at 1 ppm) into the PUF cartridge for Laboratory control sample and Laboratory control sample duplicate.

14.2.6. Reflux the sample for 18 hours at a rate of at least 3 cycles per hour, allow cooling and then disassembling the apparatus.

14.2.7. Concentrate the extracted sample to 2 ml by using a DryVap Concentrator system from Horizon Technology, transfer the extract by pouring it to glass container on the concentrator unit, rinse the flask and column with about 30ml of 10% diethyl ether/hexane to complete the quantitative transfer. Prior to starting the Concentrator set dry volume to 0 to complete the process at 2 ml, heater power to 1 for the

temperature set at approximately 70 degrees Celsius then push the start button to start the concentration process.

14.2.8. After concentration is complete at 2 ml transfer the remaining sample to a 40ml glass vial.

14.2.9. All glassware that is used is rinsed with Hexane, washed with hot water containing detergent. Rinse with tap water and several portions of distilled water. Drain, dry, and heat in a muffle furnace at 400 degree Celsius for 4 hours. After glassware is dry and cool, rinse it with Hexane.

14.3. Instrument Setup

14.3.1. Use the following GC operating conditions as guidance to establish the GC temperature program and flow rate necessary to separate the analytes of interest.

Description	GC Operating Condition
Inlet mode	pulsed splitless
Inlet temperature	220°C
Inlet pressure	23.806 psi
Total flow rate	90.805 mL/min
Carrier gas flow rate	4.905 mL/min
Makeup gas flow rate	30 mL/min
Detector temperature	300°C
Initial temperature	120°C
Temperature program	120°C to 200°C at 45°C/min 200°C to 230°C at 12.5°C/min 230°C to 330°C at 20°C/min
Final temperature	330°C, hold 2 min

14.3.2. Autoinjector is set to inject 1 µL of field or QC sample extract.

14.3.3. An initial oven temperature of ≤ 140–150°C may be necessary to resolve the four BHC isomers. A final oven temperature of 240–270°C may be necessary to elute decachlorobiphenyl.

14.3.4. The use of injector pressure programming will improve the chromatography of late eluting peaks.

14.3.5. Once established, the same operating conditions must be applied for all subsequent standard, sample, and blank analyses.

14.4. Following the establishment of a valid initial calibration, a CCV standard must be analyzed daily prior to sample analysis, after every batch of 20 samples or portion thereof within a 12-hour shift, and at the end of sequence. If the QC and retention time criteria are met, the initial calibration is assumed to be valid and sample analysis may resume. The acceptance criteria are listed in Section 12.4. and Section 12.5.3.

14.4.1. For EPA Region 9 requirement, refer to Section 12.4.1.1. for CCV frequency.

- 14.4.2. If a failed CCV is the first of the day, effect corrective action prior to analyzing any samples.
- 14.4.3. If a failed CCV is not the first of the day, effect corrective action and reanalyze all samples since the last acceptable CCV.
- 14.5. Following extraction by one of the methods specified in Section 5.2., the extracts for the QC and actual environmental samples are received in autoinjector vials. The autoinjector vials are then loaded onto the GC sample tray.
- 14.6. Standard and sample vials are loaded in the following or other logical order:
- 1) Degradation Test
 - 2) Continuing Calibration Verification (CCV)
 - 3) Laboratory Control Sample (LCS)
 - 4) Laboratory Control Sample Duplicate (LCSD)
 - 5) Method Blank (MB)
 - 6) Samples (up to 20 per batch, including QC check samples and MBs)
 - 7) Matrix Spike (MS)
 - 8) Matrix Spike Duplicate (MSD)
 - 9) Ending CCV
- 14.6.1. Item 1: An acceptable degradation test demonstrates that the chromatographic system is not causing breakdown of thermally labile compounds due to active sites (e.g., the injection port is contaminated or contains catalytic active sites). A degradation test meeting the acceptance criteria is required daily prior to sample analysis and every 12 hours thereafter during analysis.
- 14.6.2. Items 2 and 9: A CCV is used to verify the acceptance of the initial multi-point calibration on a continuing basis. An acceptable CCV is required daily prior to sample analysis, after every batch of 20 samples or portion thereof within a 12-hour shift, and at the end of sequence.
- 14.6.2.1. More frequent (e.g., every 10 samples) calibration verification may be useful to minimize the number of sample extract re-analyses that would be required in the event of an unacceptable CCV.
- 14.6.3. Item 3: The LCS is a known matrix which has been spiked with known concentrations of specific target analytes. The purpose of the LCS is to demonstrate that the entire analytical process and systems are in control. The LCS is processed concurrently with the associated samples. In the processing of the LCS, reagents and procedures identical to those for actual samples are used.
- 14.6.3.1. For aqueous samples, the LCS consists of the specified compounds spiked into clean reagent water. For solid and oil samples, the LCS consists of the specified compounds spiked into washed sea sand. For wipe samples, the LCS consists of the specified compounds spiked into unused gauze pad. For filter samples, the LCS consists of the specified compounds spiked into unused filter paper. For tissue samples, the LCS consists of the specified compounds spiked into washed sea sand containing food grade oil. For mobility-procedure extracts,
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the LCS consists of the specified compounds spiked into the mobility-procedure extract designated as LCS.

- 14.6.3.2. One LCS is required every day preparatory methods (i.e., extractions, cleanups, etc.) are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.
 - 14.6.4. Item 4: The LCSD is handled identically to the LCS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the LCS in combination with the LCSD can be used to assess the precision of the analytical process. The measurement is expressed as relative percent difference (RPD). The formula for calculating RPD is listed in Section 15.7.
 - 14.6.5. Item 5: The MB is a known matrix similar to the samples being analyzed which is processed concurrently with the associated samples. In the processing of the MB, reagents and procedures identical to those for actual samples are used (e.g., surrogates, etc.).
 - 14.6.5.1. One MB is required every day preparatory methods (i.e., extractions, cleanups, etc.) are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.
 - 14.6.5.2. When samples that are processed together are analyzed on separate instruments or on separate analytical shifts, the MB associated with those samples must be analyzed on at least one of the instruments. A solvent blank consisting of hexane must be analyzed on all other instruments where the associated samples are analyzed to demonstrate that the instruments are not contributing contaminants to the samples.
 - 14.6.6. Item 6: Up to 20 sample (including QC check sample and method blank) extracts per batch. Complex extracts should be sufficiently diluted or subjected to cleanup procedures to ensure that instrument is not contaminated. Dilution or cleanup of extracts will result in increased reporting limits.
 - 14.6.6.1. All dilutions should keep the responses of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.
 - 14.6.7. Item 7: The MS is the actual sample matrix spiked with known concentrations of specific target analytes. The sample which is spiked for the MS is processed concurrently with the associated samples. In the processing of the MS, reagents and procedures identical to those for actual samples are used.
 - 14.6.7.1. The purpose of the MS is to assess the effect of a sample matrix on the recovery of target analytes (i.e., assess the accuracy of the analytical measurements of the matrix). The measurement is expressed as percent recovery (%REC). The formula for calculating %REC is listed in Section 15.6.
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- 14.6.7.2. One MS is required for every batch of 20 samples per matrix or portion thereof processed concurrently. This approach is considered "closed batch" as opposed to "open batch."
- 14.6.8. Item 8: The MSD is handled identically to the MS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the MS in combination with the MSD can be used to assess the precision of the analytical measurements. The measurement is expressed as relative percent difference (RPD). The formula for calculating RPD is listed in Section 15.7.
- 14.6.9. Solvent blanks may be added elsewhere in the sequence, as necessary (i.e., after suspected high concentration sample extracts), to check for potential carryover or cross-contamination.
- 14.7. Ensure that a sufficient amount of hexane is present in the autoinjector solvent rinse bottles and that a sufficient unused volume exists in the autoinjector waste bottles at the beginning of the sequence.
- 14.8. Edit the sequence in the data system. After all correct sample information is entered, save the sequence. After saving the sequence, record pertinent information in the instrument run logbook or on the sequence table printout.
- 14.9. Initiate the sequence.
- 14.10. Data Interpretation
- 14.10.1. Establish the daily retention time window for each analyte/surrogate (see Section 12.5.2.1. and Section 12.5.2.2.).
- 14.10.1.1. Tentative identification of an analyte/surrogate occurs when a peak from a sample extract falls within the daily retention time window.
- 14.10.1.1.1. For each multi-component analyte (i.e., chlordane and toxaphene), choose a minimum of 5 characteristic peaks that are at least 25% of the height of the largest characteristic peak for the analyte, and determine the retention time window of each characteristic peak.
- 14.10.1.2. Use the succeeding CCV standards analyzed throughout the course of an analytical sequence within a 12-hour shift to evaluate retention time stability (see Section 12.5.3.). If any analyte(s)/surrogate(s) in the CCV standard fall outside of their daily retention time window(s), determine the cause of the problem and effect appropriate corrective action.
- 14.10.1.2.1. If any analyte(s)/surrogate(s) in the single-component pesticide CCV standard fall outside of their daily retention time window(s), then all samples analyzed since the last acceptable CCV should be invalidated, corrective action effected, and the affected samples re-analyzed.
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- 14.10.2. Quantitation of a target analyte is based on a reproducible response of the detector within the calibration range and a direct proportionality of the magnitude of response between peaks in the sample extract and the calibration standards.
- 14.10.2.1. Determine the concentration based on the initial calibration curve.
- 14.10.2.1.1. Calculate the concentration of each single-component pesticide target analyte in a sample extract using the average of the initial RFs and the area of the characteristic peak. The formula for calculating concentration is listed in Section 15.8.
- 14.10.2.1.2. The data system is programmed to perform the calculation of concentration.
- 14.10.2.2. If the instrument response exceeds the calibration range, dilute the extract and reanalyze.
- 14.10.3. Tentative identification of a target analyte occurs when a peak from a sample extract falls within the analyte's retention time window. Confirmation is necessary when the composition of samples is not well characterized. Qualitative confirmation techniques are by second column with dissimilar stationary phase, GC/MS with Selected Ion Monitoring (SIM) or Full Scan mode, or GC data from two different detectors.
- 14.10.4. Second column confirmation is made on a "confirmation" channel configured with a column of dissimilar stationary phase and a second detector. The principle is that the retention time of the target analyte will differ between the primary and confirmation column and, unless the detected compound is the particular target analyte, it will not be observed within both retention time windows.
- 14.10.4.1. Report the result from the primary column.
- 14.10.4.1.1. If one result is significantly higher (e.g., > 40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are observed, examine the baseline parameters established by the instrument data system (or operator) during peak integration. A rising baseline may cause the mis-integration of the peak for the lower result.
- 14.10.4.1.2. If no anomalies are observed, review the chromatographic conditions. If there is no evidence of chromatographic problems, then it may be appropriate to report the lower result.
- 14.10.4.1.3. The data user must be advised of the disparity between the results on the two columns. Under some circumstances, including those involving in monitoring compliance with an action level or
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regulatory limit, further cleanup of the sample or additional analyses may be required when the two results in question span the action level or regulatory limit.

- 14.10.4.2. In cases where a peak is not observed in the confirmation column's retention time window, the analyte is reported as "ND."
 - 14.10.4.3. A calibration curve and retention time window for each analyte/surrogate are also established and maintained for the confirmation channel. The calibration and quality control requirements for the confirmation channel are identical to those of the primary channel.
 - 14.10.5. GC/MS confirmation is more reliable than second column confirmation. In this case, where confirmation is required by project requirements, the sample is re-analyzed on GC/MS. When GC/MS results indicate that a target analyte is not present, the GC result is reported as "ND."
 - 14.10.6. Confirmation is required for all positive results unless the samples meet all of the following requirements:
 - 14.10.6.1. All samples (air, aqueous, solid, oil, or tissue) come from the same source (e.g., same monitoring well). However, samples of the same matrix from the same site but from differing sources (e.g., different monitoring wells) are not exempted.
 - 14.10.6.2. All chemical parameters have been previously analyzed, identified, and confirmed by a second column with dissimilar stationary phase, GC/MS with Selected Ion Monitoring (SIM) or Full Scan mode, or GC data from two different detectors. Documentation of such must be maintained.
 - 14.10.6.3. The resulting chromatograms are relatively simple and do not contain complex or overlapping peaks.
 - 14.10.6.4. Chromatograms are largely unchanged from those for which confirmation was carried out.
 - 14.10.7. Manual integration of peaks shall adhere to the procedures and documentation policies outlined in the current revision of SOP-T023.
 - 14.10.7.1. When the instrument software produces proper integrations, it is highly recommended to use the integrations produced by the instrument software for consistency.
 - 14.10.7.2. When the instrument software does not produce proper integrations (e.g., selecting an improper baseline, missing the correct peak, integrating a co elution, partially integrating a peak, etc.), manual integrations performed by the analyst are necessary.
 - 14.10.7.3. Manual integration should be minimized by properly maintaining the instrument, updating the retention times, and configuring the peak integration parameters.
-

14.11. Recommended Instrument Maintenance

- 14.11.1. Perform the following routine maintenance procedures to improve chromatographic performance.
 - 14.11.1.1. Replace septa after the oven is cooled.
 - 14.11.1.2. Clean and deactivate injector liners.
 - 14.11.1.3. Clean the metallic surfaces of the injection port.
 - 14.11.1.4. Prevent capillary columns from touching the oven walls, and keep oxygen out of capillary columns.
 - 14.11.1.5. Scrub carrier gas to remove traces of water and oxygen, and change the scrubbers regularly.
 - 14.11.2. Perform the following tasks to remedy the column adsorption problem.
 - 14.11.2.1. Inject an 800-ppb single-component pesticide standard solution to prime (or deactivate) the column.
 - 14.11.2.2. Run one or more solvent blanks consisting of hexane until no carryover is observed prior to analyzing any standards or samples.
 - 14.11.3. Perform the following tasks to eliminate the degradation problem.
 - 14.11.3.1. For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter.
 - 14.11.3.2. Break off the first few centimeters (up to 30 cm) of the injection port side of the column.
 - 14.11.3.3. Check the injector temperature and lower it to 205°C, if necessary.
 - 14.11.3.4. Remove the columns and solvent back flush according to the manufacturer's instructions.
 - 14.11.3.5. If all else fail, it may be necessary to deactivate the metal injector body and/or replace the columns.
 - 14.11.4. Perform the following tasks to rinse the analytical column.
 - 14.11.4.1. Depending on the nature of the residues expected, the first rinse might be reagent water, followed by methanol and acetone, with methylene chloride as the final rinse. In some cases, methylene chloride may be the only solvent necessary.
 - 14.11.4.2. After the final rinse, the analytical column should be filled with methylene chloride and remained flooded overnight to allow materials within the stationary phase to migrate into the solvent.
 - 14.11.4.3. The analytical column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen passing through the column.
-

15. CALCULATIONS

15.1. The response factor is calculated as follows:

$$RF = \frac{A_x}{C_x}$$

where: RF = response factor for target analyte being measured.
 A_x = area of the characteristic peak(s) for target analyte being measured.
 C_x = concentration of target analyte being measured in $\mu\text{g/L}$.

15.2. The percent relative standard deviation is calculated as follows:

$$\%RSD = \frac{SD}{RF_{ave}} \times 100$$

where: %RSD = percent relative standard deviation.
SD = standard deviation of the RFs for the target analyte.
 RF_{ave} = mean of the 5, 6, or 7 initial RFs for the target analyte.

15.3. The percent difference of each analyte is calculated as follows:

$$\%D = \frac{|RF_{ave} - RF_{daily}|}{RF_{ave}} \times 100$$

where: %D = percent difference.
 RF_{daily} = daily RF for the target analyte.
 RF_{ave} = mean of the 5, 6, or 7 initial RFs for the target analyte.

15.4. The percent drift of each analyte is calculated as follows:

$$\%D = \frac{|C_{expected} - C_{measured}|}{C_{expected}} \times 100$$

where: %D = percent drift.
 $C_{expected}$ = concentration of target analyte expected.
 $C_{measured}$ = concentration of target analyte measured.

Note: Concentrations must be in equivalent units.

15.5. The recovery of each LCS compound is calculated as follows:

$$\%REC_{LCS} = \frac{C_{recovered}}{C_{added}} \times 100$$

where: % REC_{LCS} = percent recovery of target analyte in LCS (or LCSD).
 $C_{recovered}$ = concentration of target analyte recovered.

C_{added} = concentration of target analyte added.

Note: Concentrations must be in equivalent units.

15.6. The recovery of each MS compound is calculated as follows:

$$\% \text{REC}_{\text{MS}} = \frac{C_{\text{recovered}} - C_{\text{sample}}}{C_{\text{added}}} \times 100$$

where: $\% \text{REC}_{\text{MS}}$ = percent recovery of target analyte in MS (or MSD).
 $C_{\text{recovered}}$ = concentration of target analyte recovered.
 C_{sample} = concentration of target analyte in environmental sample used.
 C_{added} = concentration of target analyte added.

Note: Concentrations must be in equivalent units.

15.7. The relative percent difference is calculated as follows:

$$\text{RPD} = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where: RPD = relative percent difference between two measurements (C_1 and C_2).
 C_1 = concentration of target analyte in measurement 1.
 C_2 = concentration of target analyte in measurement 2.

Note: Concentrations must be in equivalent units.

15.8. The target analyte concentration for a sample extract is calculated as follows:

$$C_{\text{ex}} = \frac{A_x}{\text{RF}_{\text{ave}}}$$

where: C_{ex} = concentration of target analyte in extract in $\mu\text{g/L}$.
 A_x = area of the characteristic peak(s) for target analyte.
 RF_{ave} = mean of the 5, 6, or 7 initial RFs for the target analyte.

15.9. The target analyte concentration for an air sample is calculated as follows:

$$C_A = \frac{C_{\text{ex}} \times V_{\text{ex}} \times D}{V_A}$$

where: C_A = concentration of target analyte in air sample in ng/sample .
 C_{ex} = concentration of target analyte in extract in mg/L .
 V_{ex} = volume of extract in mL .
 V_A = volume of air sampled.
 D = dilution factor, if the sample or extract was diluted prior to analysis.
If no dilution was made, $D = 1$.

15.10. The percent breakdown is calculated as follows:

15.10.1. The percent breakdown of DDT is calculated as follows:

$$\%B_{DDT} = \frac{A_{DDD} + A_{DDE}}{A_{DDT} + A_{DDD} + A_{DDE}} \times 100$$

where: $\%B_{DDT}$ = percent breakdown of DDT.
 A_{DDD} = degradation peak area of DDD.
 A_{DDE} = degradation peak area of DDE.
 A_{DDT} = peak area of DDT.

15.10.2. The percent breakdown of endrin is calculated as follows:

$$\%B_{endrin} = \frac{A_{aldehyde} + A_{ketone}}{A_{endrin} + A_{aldehyde} + A_{ketone}} \times 100$$

where: $\%B_{endrin}$ = percent breakdown of endrin.
 $A_{aldehyde}$ = degradation peak area of aldehyde.
 A_{ketone} = degradation peak area of ketone.
 A_{endrin} = peak area of endrin.

15.11. Refer to the preparatory method(s) for additional calculations.

15.12. All concentrations shall be reported in ng/Sample for air samples.

15.13. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

16. METHOD PERFORMANCE

16.1. A demonstration of analytical capability shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

16.2. Calibration protocols specified in Section 13., "Calibration and Standardization," shall be followed.

16.3. Proficiency test sample results shall be used to evaluate the ability to produce accurate results.

17. POLLUTION PREVENTION

17.1. The toxicity, carcinogenicity and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.

17.2. Exposure to these chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current revision of Calscience's Health, Safety, and Respiratory Protection Manual. In

general, protective eyewear (e.g. safety glasses or goggles), and protective apparel (e.g. lab coats) and gloves are required to be worn when handling chemicals.

- 17.3. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials:
 - 17.3.1. A NIOSH approved air purifying respirator with cartridges appropriate for the chemical handled.
 - 17.3.2. Extended length protective gloves.
 - 17.3.3. Face shield.
 - 17.3.4. Full-length laboratory apron.
- 17.4. Processes that promote vaporization of volatile chemicals should be performed in an area well ventilated to the exterior of the laboratory to prevent contamination to other areas in the laboratory.
- 17.5. When working with large amounts of volatile chemicals, the Coordinator must be cautious of the risk of high levels of volatile displacing the atmospheric air within the work area; therefore causing asphyxiation. Air purification respirators are ineffective in this situation and must not be used. The Coordinator must immediately vacate the area until ventilation has effectively reduced the concentration of volatiles. Alternatively, the Coordinator may utilize a self-contained breathing apparatus or other supplied air system if appropriately trained and approved by the Health and Safety Manager.
- 17.6. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

18. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

- 18.1. The acceptance criteria for LCS/LCSD compounds vary depending upon historical data. The lower and upper acceptance limits for %REC and RPD of each LCS/LCSD compound are based upon the historical average recovery $\pm 3S$. All LCS/LCSD compounds must be within acceptance limits (see Section 12.5.2. for additional information).
 - 18.1.1. If the LCS and/or LCSD %REC is outside of the acceptance limits high, the RPD is within acceptance limits, and all target analytes in the associated samples are not detected, the sample data can be reported without qualification.
 - 18.1.2. The LCSD is only reported when the MS/MSD is unacceptable due to matrix interference effects, or when the LCS/LCSD is used in place of MS/MSD due to insufficient sample quantity.
 - 18.2. Ideally, the concentration of target analytes in an MB should be less than the respective reporting limits (RLs). If the concentration of any target analyte exceeds its RL, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs are as follows:
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- 18.2.1. If a target analyte is found in the MB but not in the associated samples, report the sample and MB data without qualification.
- 18.2.2. If a target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified or rejected and the samples re-extracted and/or re-analyzed.
- 18.3. The acceptance criteria for surrogate spike compound recoveries vary depending upon historical data. The lower and upper acceptance limits for %REC of each surrogate spike compound are based upon the historical average recovery $\pm 3S$.
 - 18.3.1. If the surrogate compound recoveries are acceptable, report the surrogates and sample data without qualification.
 - 18.3.2. If one or more surrogate recoveries are not acceptable, evaluation is not necessarily straightforward. The sample itself may produce effects due to factors such as interferences and high analyte concentration. This data alone cannot be used to evaluate the precision and accuracy of individual sample analyses. However, when exercising professional judgment, this data should be used in conjunction with other available QC information.
 - 18.3.3. By itself, unacceptable surrogate recoveries do not invalidate sample data. The following must be accomplished if surrogate recoveries are not acceptable.
 - 18.3.3.1. Check the internal standard and surrogate spiking solutions for degradation and contamination.
 - 18.3.3.2. If the nonconformance is due to poor instrument performance or if the above actions fail to reveal the cause of the unacceptable surrogate(s) recovery, the same sample or extract should be re-analyzed.
 - 18.3.3.3. If incorrect procedures or degraded/contaminated spiking solutions are determined to have not caused the unacceptable surrogate recoveries, the affected sample(s) must be re-extracted and/or re-analyzed or, if insufficient sample remains, reference made to the associated MB surrogate recoveries and the sample data reported with qualification.
 - 18.3.3.3.1. If, upon re-extraction and re-analysis, the surrogates remain unacceptable, matrix interference can be cited and reference made to the associated MB surrogate recoveries and the sample data reported with qualification.
 - 18.3.3.3.2. If the MB surrogates are unacceptable, all associated sample data must be invalidated and all associated samples re-extracted and re-analyzed.
 - 18.3.4. Where sample dilution is required, depending on the dilution factor, the surrogate recovery will be low or not detected. This is an expected

occurrence and reference should be made to the MB surrogate recovery which must be reported to the client.

- 18.4. The acceptance criteria for MS/MSD compounds vary depending upon historical data. The lower and upper acceptance limits for %REC and RPD of each MS/MSD compound are based upon the historical average recovery $\pm 3S$.
 - 18.4.1. When the %REC and RPD of the MS/MSD compounds are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.
 - 18.4.2. If the %REC and/or RPD of the MS/MSD compounds are not within the established acceptance limits, the analytical system performance shall be suspect.
- 18.5. Matrix effects or poor instrument performance/technique typically causes unacceptable %REC values. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.
- 18.6. Additional information regarding internal quality control checks is provided in SOP-T020.
- 18.7. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

19. CORRECTIVE ACTIONS

- 19.1. If on the basis of internal or external systems or performance audits, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented.
- 19.2. The Operations Manager, Project Manager, Quality Control Manager, Group Leader and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if corrective action will impact the project budget or schedule, the action may directly involve the Laboratory Director.
- 19.3. Corrective actions are generally of two types, immediate and long-term actions.
 - 19.3.1. An **immediate action** is designed to correct or repair nonconforming instruments and measurement systems. The analyst or Group Leader as a result of calibration checks and other QC sample analyses most frequently will identify the need for such an action.
 - 19.3.2. A **long-term action** is designed to eliminate causes of nonconformance. The need for such actions is identified by systems and performance audits. The systematic nonconformance identified during the data generation process and the appropriate corrective measures taken are thoroughly

documented in the Corrective Action Record. Examples of this type of action include:

- 19.3.2.1. Remedial training of staff in technical skills, technique or implementation of operating procedures.
 - 19.3.2.2. Rescheduling of analytical laboratory routine to ensure analysis within holding times.
 - 19.3.2.3. Revision of standard operating procedures.
 - 19.3.2.4. Replacing personnel, as necessary.
- 19.4. For either type of corrective action, the sequential steps that compose a close-loop corrective action system are as follows:
- 19.4.1. Define the problem.
 - 19.4.2. Assign responsibility for investigating the problem.
 - 19.4.3. Investigate and determine the cause of the problem.
 - 19.4.4. Assign and accept responsibility for implementing the corrective action.
 - 19.4.5. Determine effectiveness of the corrective action and implement correction.
 - 19.4.6. Verify that the corrective action has eliminated the problem.
- 19.5. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be properly documented on a Corrective Action Record.

20. CONTINGENCIES FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

- 20.1. Out-of-control data are reviewed and verified by the technical director of the appropriate department. All samples associated with an unacceptable QC set are then subject to reanalysis, depending upon the QC type in question.
- 20.1.1. MS/MSD: Acceptability of the MS/MSD recoveries is subject to the matrix and any anomalies associated with the subject batch. Failure of recoveries of an MS/MSD data set does not constitute an automatic reanalysis of the batch samples. Rather, it is acceptable to defer to the LCS/LCSD recoveries, to determine acceptance of the sample results.
 - 20.1.2. LCS/LCSD: Because they denote whether the analytical system is operating within control, it is imperative that the LCS recoveries obtained are within acceptance criteria. If the recoveries fail for a given reported compound, the technical director confirms the unacceptable result.
 - 20.1.2.1. If the LCS results are verified as acceptable, no corrective action is required.
 - 20.1.2.2. If the LCS result is verified as out-of-control, and the subject compound is to be reported in samples within that analytical batch, the samples reported with that failed compound must be reanalyzed with a valid LCS recovery for the compound.

- 20.1.2.3. If the LCS result is verified as out-of-control, and the subject compound is NOT to be reported in the samples within that analytical batch, the samples are not subject to reanalysis. No corrective action is required for that batch.

21. WASTE MANAGEMENT

- 21.1. The proper disposal of analytical samples and laboratory wastes is not only good laboratory practice, but also regulated by a variety of local, state, and federal laws. In order to remain compliant with these laws, and at the same time keep sample disposal costs at a minimum, the samples and wastes are identified, segregated, and either returned to the client (preferable) or placed into the proper laboratory waste stream.
- 21.2. Unused or remaining soil or liquid samples and all other solid or liquid wastes resulting from our laboratory operations are considered hazardous for disposal purposes.
- 21.3. All laboratory personnel must be aware of the types of chemicals they are using and the appropriate procedures for their disposal.
- 21.4. Each specific laboratory area shall maintain clearly labeled waste containers for small quantity waste collection. These waste containers shall be used for temporary collection of residual sample from aliquotting procedures, contaminated consumables, sample extracts, purged aqueous samples, and other wastes that require disposal as hazardous waste.
- 21.5. To ensure compliance with Federal RCRA regulations, the Hazardous Waste Coordinator collects and disposes of the hazardous waste at each satellite collection point no less than monthly.
- 21.6. In order to maintain accountability for all samples received by Calscience, when a sample is used in its entirety for analysis, the empty container(s) are returned to Sample Control for placement in analytical storage.
- 21.7. Waste management procedures shall adhere to the current revision of SOP-T005, "Disposal of Laboratory Samples and Wastes."

22. REFERENCES

- 22.1. EPA Method TO-4A, Compendium of methods for Toxic Organic Air Pollutants, January 1999, Second Edition.
- 22.2. *Organochlorine Pesticides by Gas Chromatography*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1B, Method 8081B, USEPA, Revision 2, February 2007.
- 22.3. *Polychlorinated Biphenyls (PCBs) by Gas Chromatography*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1B, Method 8082, USEPA, Revision 0, December 1996.
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- 22.4. *Determinative Chromatographic Separations*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1B, Method 8000C, USEPA, Revision 3, March 2003.
- 22.5. *Quality Control*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition; Volume 1, Chapter One, USEPA, Revision 1, July 1992.
- 22.6. *Choosing the Correct Procedure*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Two, USEPA, Revision 4, February 2007.
- 22.7. *Organic Analytes*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Four, USEPA, Revision 4, February 2007.
- 22.8. *Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs)*, SW-846 Method 8081 or 8080, Region 9 Quality Assurance Data Quality Indicator Tables, USEPA, December 1999.

23. ► TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

- 23.1. Appendix A: Procedure for Certification of PUF Cartridge Assembly.
- 23.2. Appendix B: Quantitation Peaks for Single-Component Target Compounds.

24. ► MODIFICATIONS

M456 Section	EPA Method TO-4A Section	Summary of Modification
Section 10.2.8.1	Section 11	Spike surrogate after sample returned to lab, before extraction process.
11.	6, 8, 10, and 11	Sampling procedure is not included.

25. ► REVISION HISTORY

Revision	Description	Author(s)	Effective Date
0.0	SOP creation.	Y. Patel / L. Lem	08/20/12
1.0	Sec 10 PCBs calibration information added.	Y. Patel	05/20/13
	Sec 12 PCBs QC information added.	Y. Patel	
	Appendix A updated.	Y. Patel	
	Appendix C added.	Y. Patel	
1.1	Added Sec 14.1, Extraction procedure for PCB and Sec 14.2, Extraction procedure for pesticides	Y. Patel	10/14/13

Appendix A

Procedure for Certification of PUF Cartridge Assembly

1. Extract one filter and PUF sorbent cartridge by Soxhlet extraction and concentrate using a K-D evaporator or DryVap concentrator for each lot of filters and cartridges sent to the field.
2. Assemble cleaned Soxhlet apparatus. Charge the Soxhlet apparatus with 700 ml of the extraction solvent diethyl ether/hexane (10%v/v) and reflux for 18 hours. Add 350 ml of diethyl ether /hexane (10%v/v) to the Soxhlet apparatus. Reflux the sample for 18 hours at a rate of at least 3 cycles per hour. Let the apparatus cool, disassemble it, and discard the used extraction solvent. Transfer the filter and PUF glass cartridge to the Soxhlet apparatus.
- 3.
4. Assemble a K-D concentrator by attaching a 10-ml concentrator tube to a 500-mL evaporative flask. If prep in DryVap system then tube used is 200 ml glass concentrator glass tube.
5. Transfer the extract by pouring it through a drying column containing about 5 gm of anhydrous granular sodium sulfate and collect the extract in the K-D concentrator or DryVap concentrator glass tube. Rinse the Erlenmeyer flask and column with about 30 ml of 10 % diethyl ether/Hexane to complete the quantitative transfer.
6. Add one or two clean boiling chips and attach a 3-ball Snyder column to the evaporative flask. Prewet the Snyder column by adding about 5 mL of the hexane to the top of the column. Place the K-D apparatus on a hot water bath (~70°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 1 hour. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches approximately 5 mL, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 5 mL of hexane. If using DryVap system set temp control at scale 2, concentrate extract to about 5 ml.
7. Concentrate the extract to 10 mL and analyze using GC/ECD.
8. The acceptance levels of common pesticides must be less than 100 ng for each pair of filter and adsorbent assembly analyzed. Once certified clean, the cartridges can be shipped to the field without being chilled.

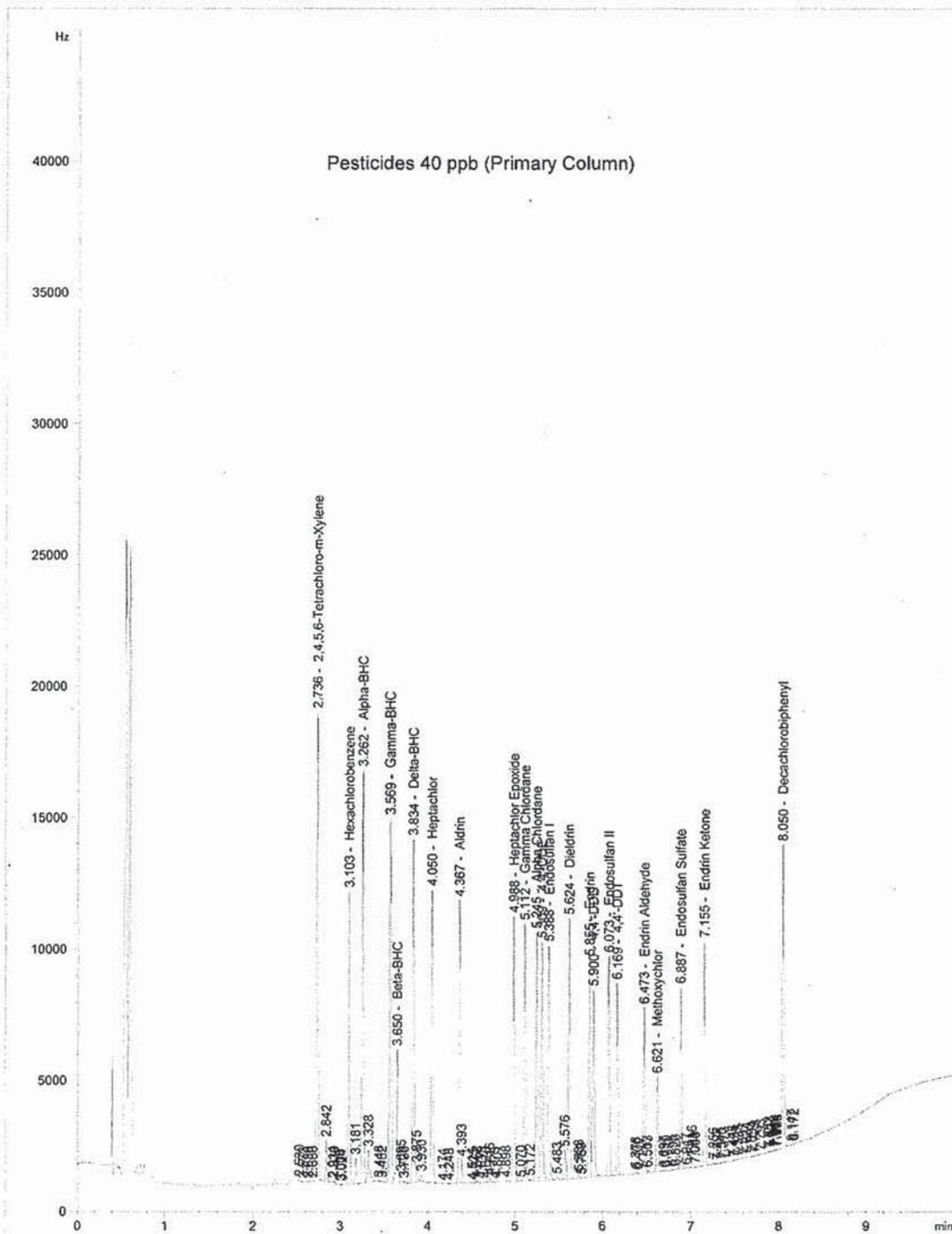
Cartridges are considered clean for up to 30 days from date of certification when sealed in their containers.

Appendix B

QUANTITATION PEAKS FOR SINGLE-COMPONENT TARGET COMPOUNDS

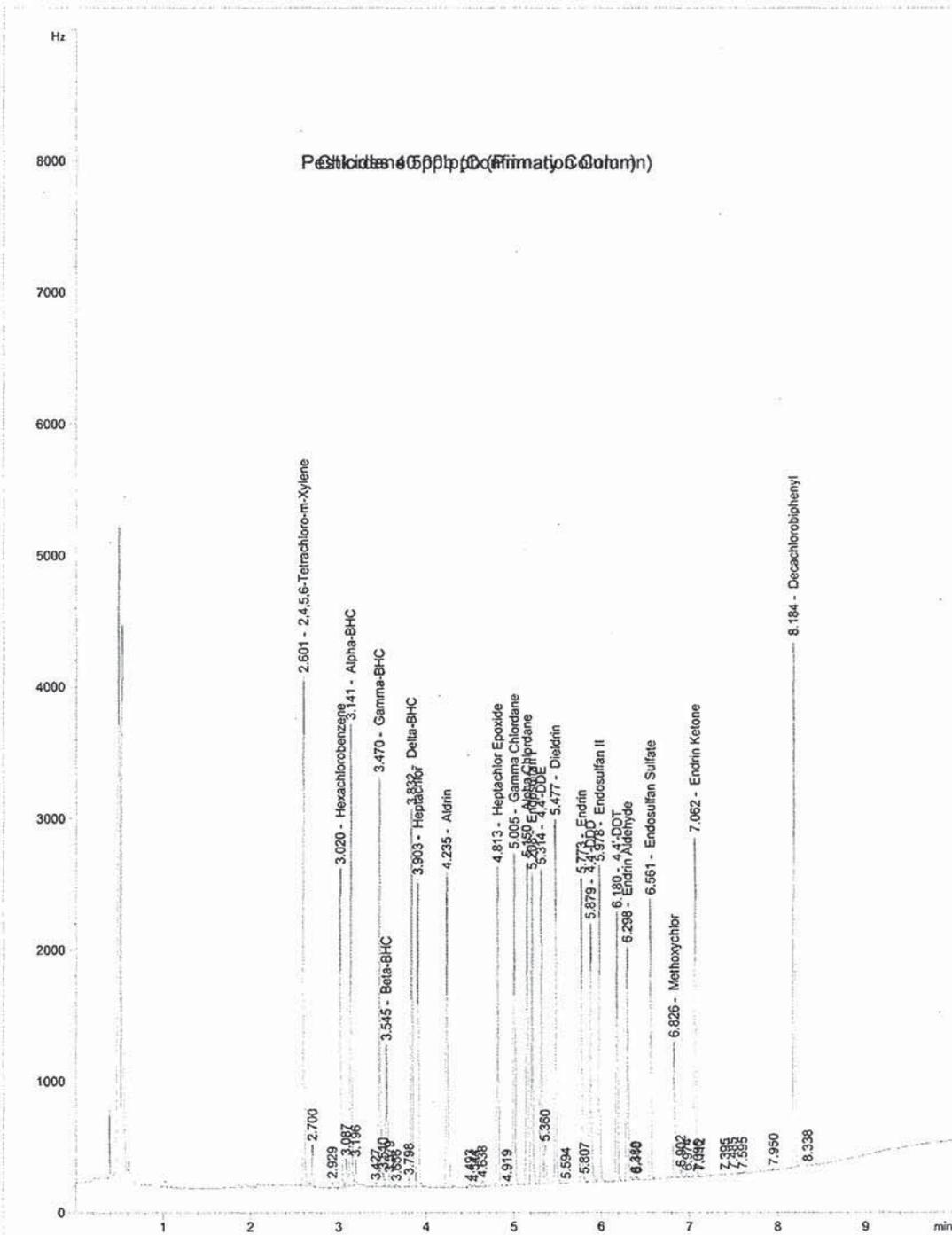
Appendix B

Quantitation Peaks for Single-Component Target Compounds



Appendix B

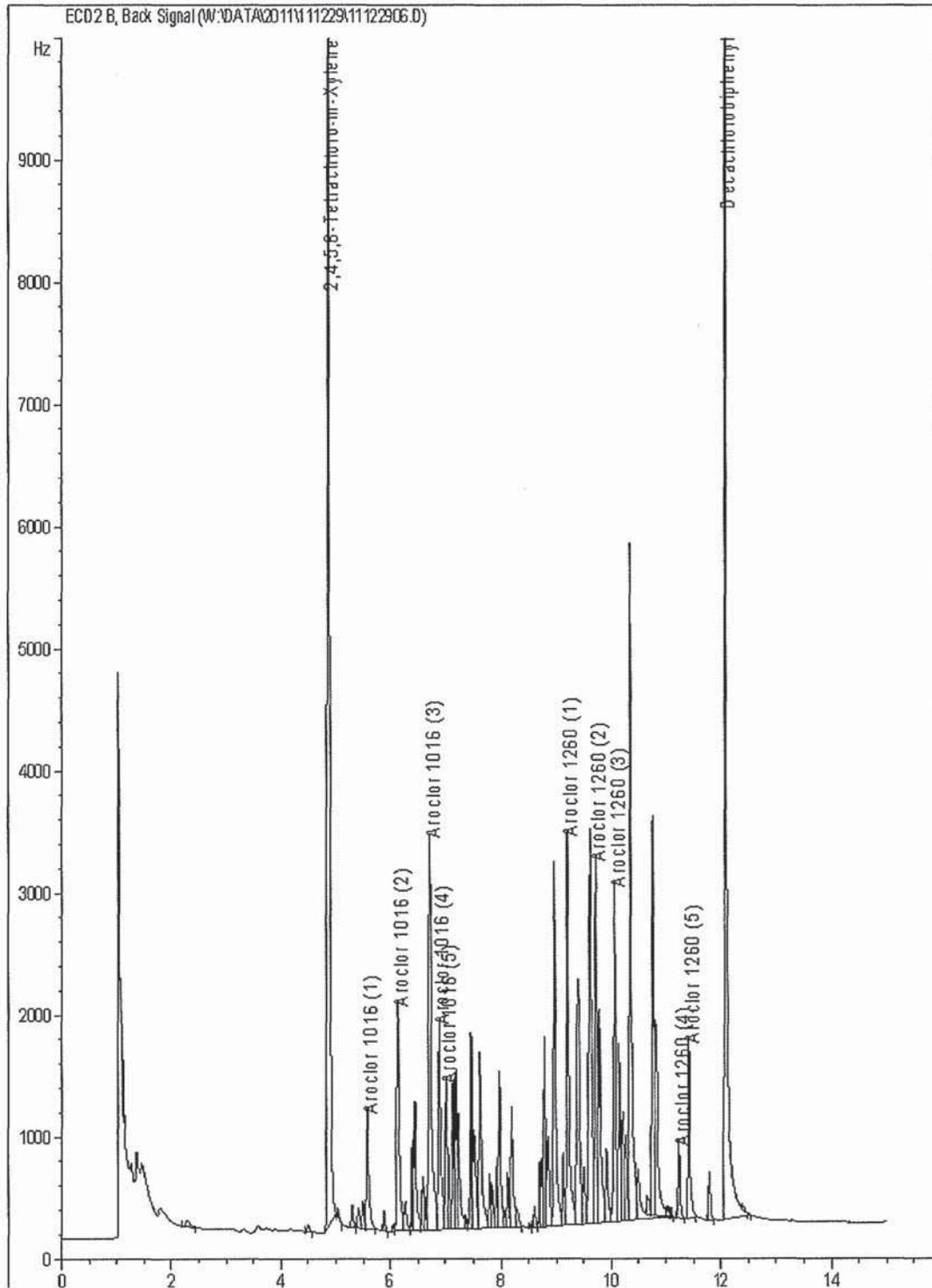
Quantitation Peaks for Single-Component Target Compounds (Cont.)



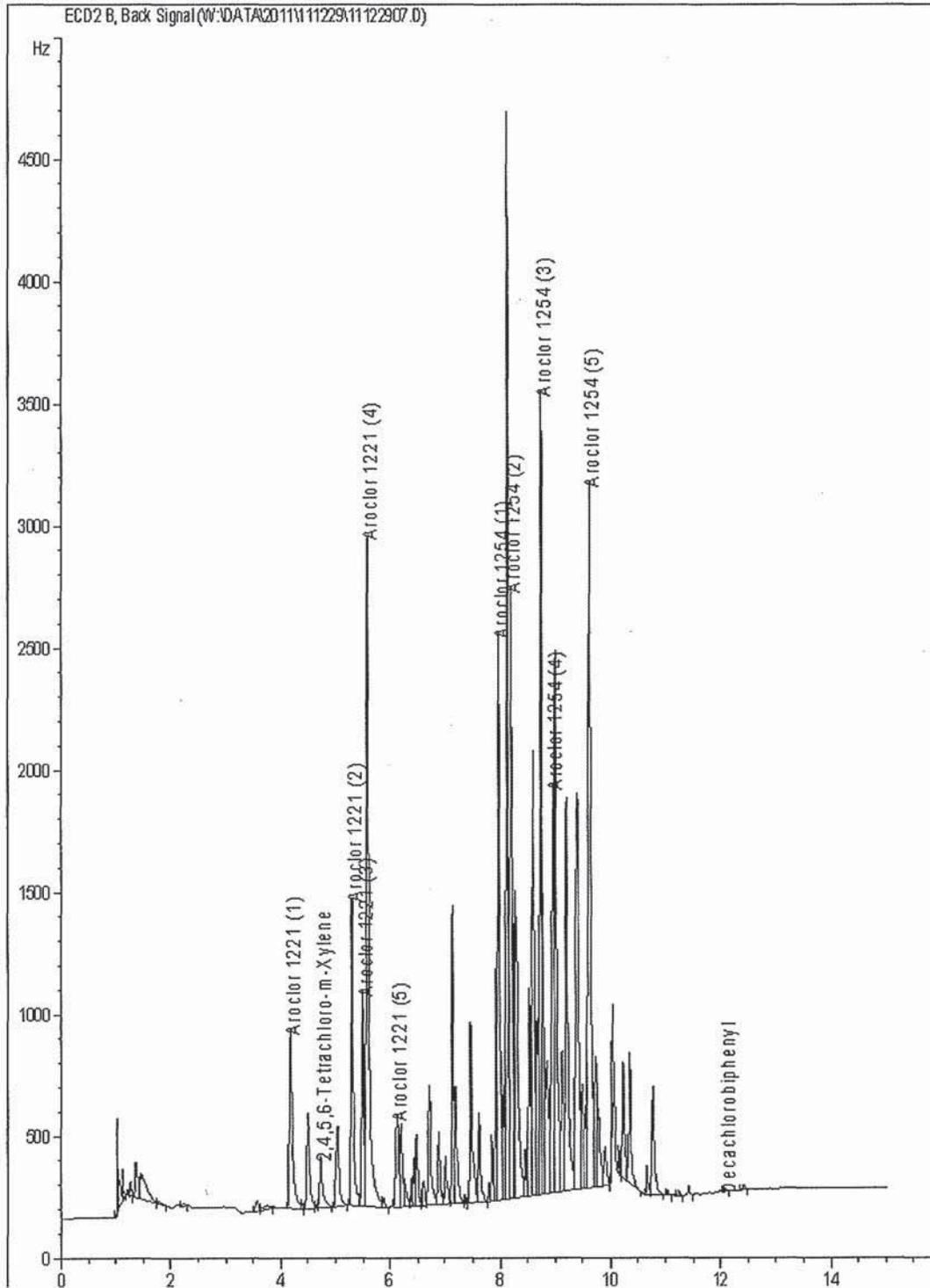
Appendix C

QUANTITATION PEAKS FOR MULTIPLE-COMPONENT TARGET COMPOUNDS

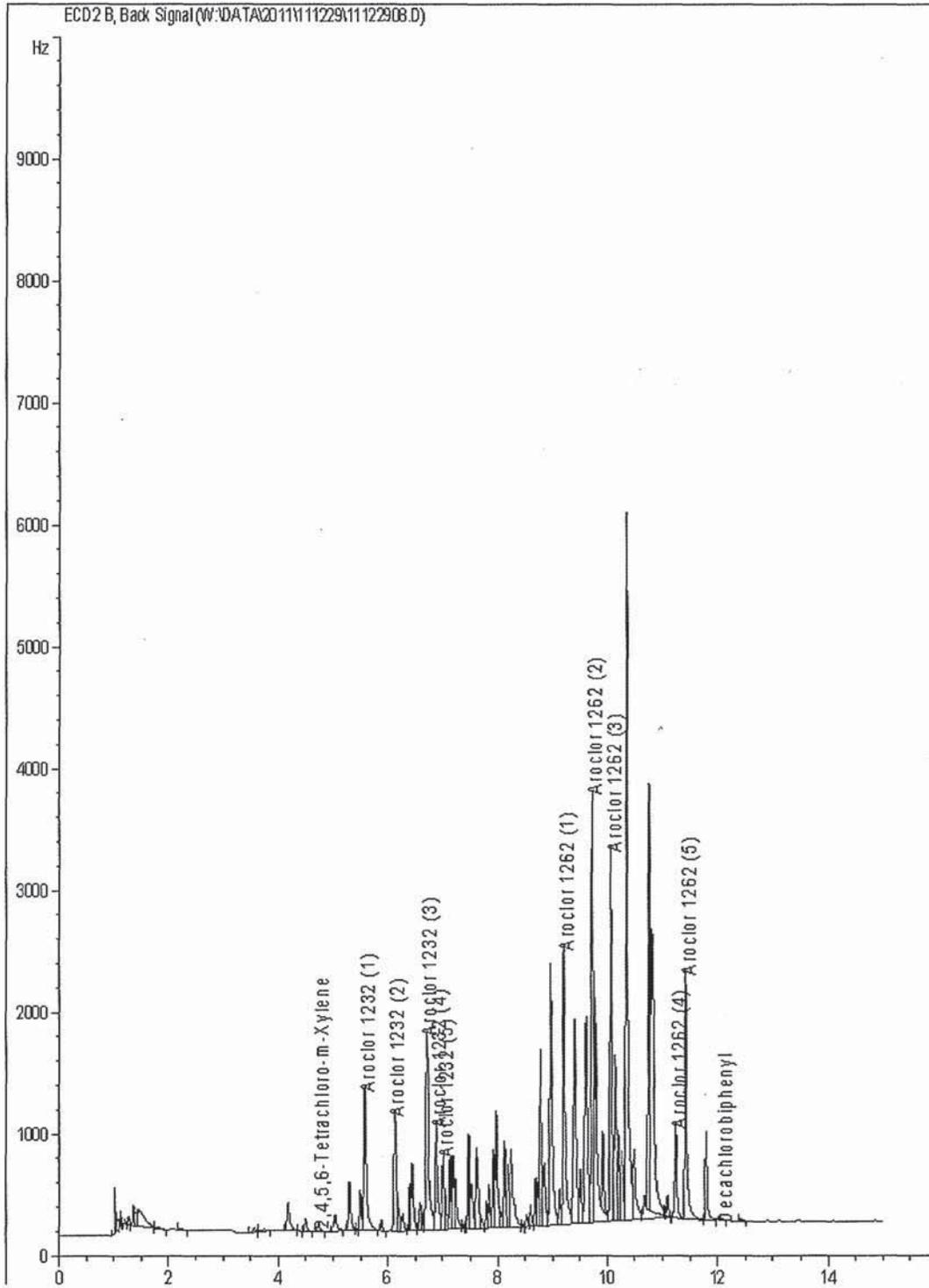
Quantitation Peaks for Aroclor 1016 and Aroclor 1260



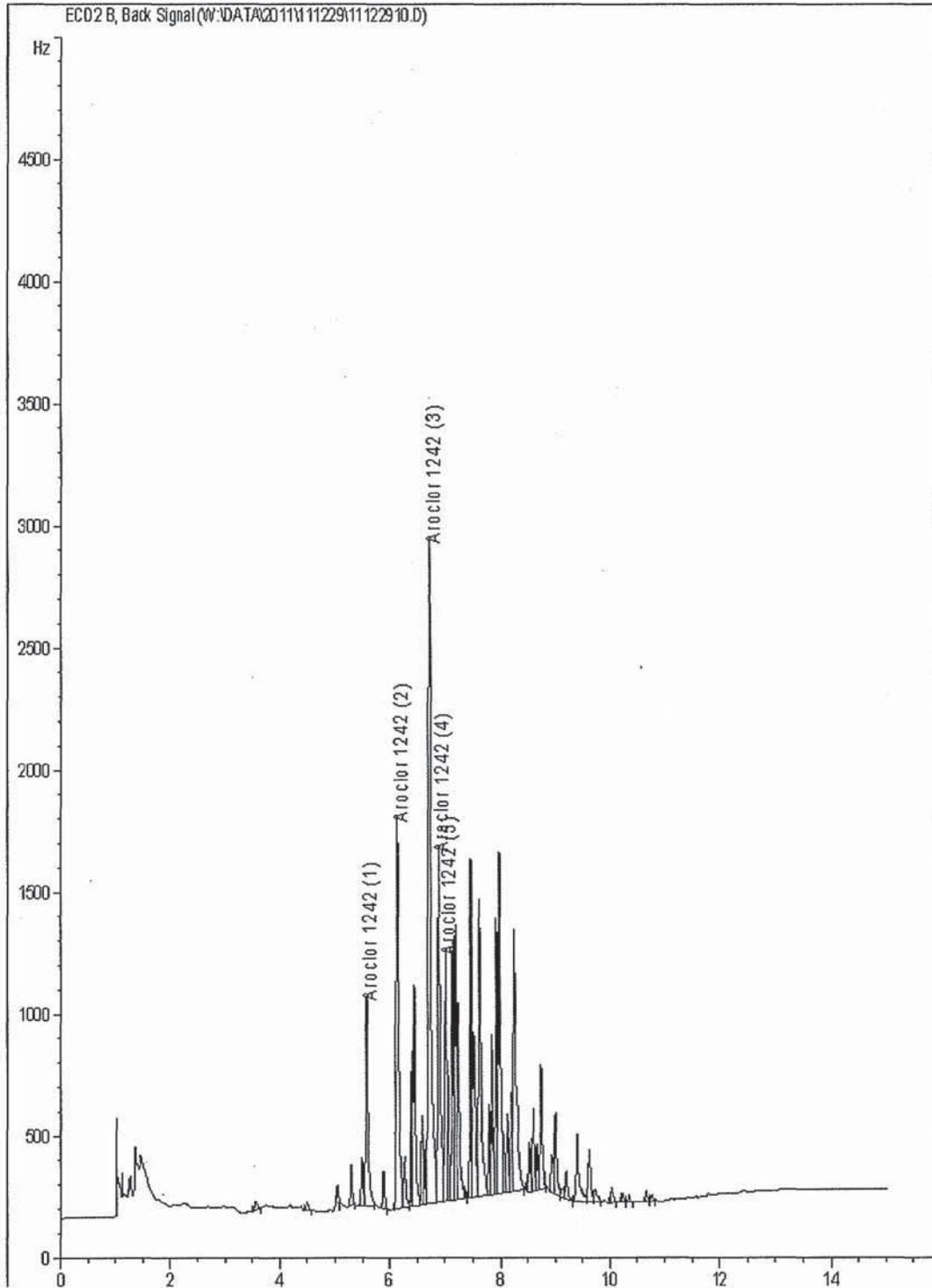
Quantitation Peaks for Aroclor 1221 and Aroclor 1254



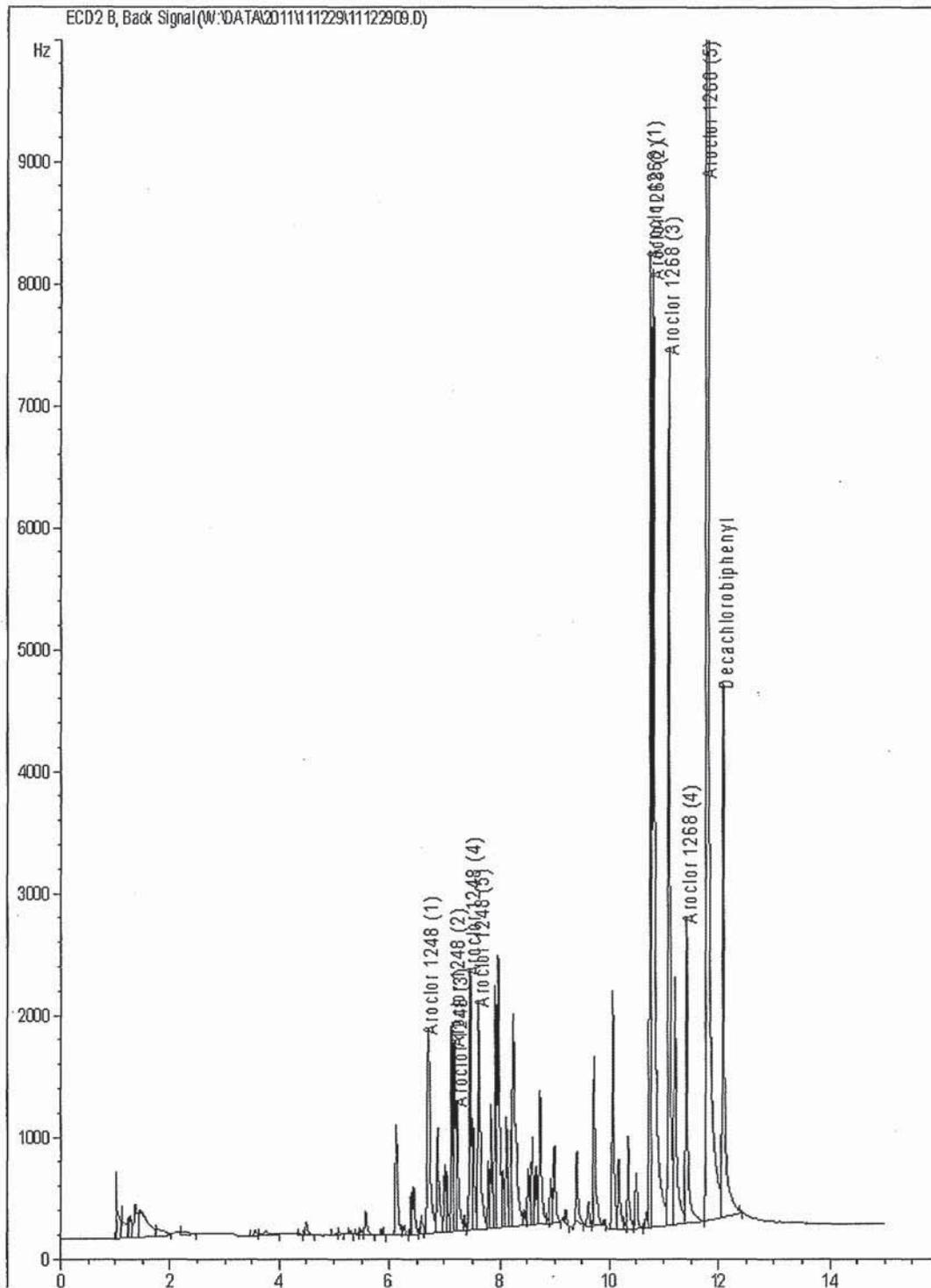
Quantitation Peaks for Aroclor 1232 and Aroclor 1262



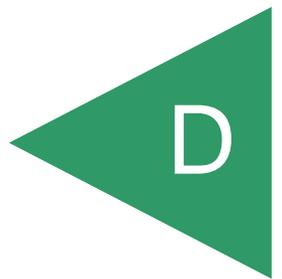
Quantitation Peaks for Aroclor 1242



Quantitation Peaks for Aroclor 1248 and Aroclor 1268



APPENDIX



ANALYTICAL METHODS

Section 5.0

Method: Modified EPA Method TO-11A Aldehydes/Ketones

Eurofins Air Toxics SOP #65 Revision 14 Effective Date: January 19, 2016 Methods Manual Summary

Description: This method involves high-pressure liquid chromatography (HPLC) analysis of aldehydes and ketones in ambient air samples. The sampling media is a 2,4-Dinitrophenylhydrazine (DNPH)-coated (silica) cartridge. Aldehydes and ketones are readily converted to a stable hydrazone derivative. The DNPH cartridges are eluted with acetonitrile using gravity-feed technique. Analysis is performed by reverse phase HPLC with UV detection at 360 nm.

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, Eurofins Air Toxics reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. For the extraction process, the non-standard compound recovery is evaluated in the extracted laboratory control spike. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Eurofins Air Toxics performs modified versions of this method. The method modifications, standard target analyte list, Limits of Quantitation (LOQs), reporting limits (RLs), Quality Control (QC) criteria, and QC summary can be found in the following tables.

Table 1. Summary of Method TO-11A Modifications

Requirement	TO-11A	Eurofins Air Toxics Modification
Initial Calibration Curve (ICAL)	Multi-point using linear regression performed every 6 months	Multi-point using average Response Factor; re-calibration if daily calibration fails, major maintenance, or column change. Linear regression is performed when requested. Initial Calibration (ICAL) is performed at least once per year.
Calibration Model and ICAL criterion	Linear Regression R^2 for curve ≥ 0.999	Average Response Factor (RF); $\%RSD \leq 10\%$ with demonstration of linearity of curve; $R^2 \geq 0.999$
Blank Subtraction	Average blank concentrations calculated. Blank value subtracted from sample result.	One Lab Blank is analyzed per batch; no automatic blank subtraction performed on samples.

Table 2. Method TO-11A Analyte List and QC Criteria (Environmental Field Samples)

Analyte	TO-11A LOQ/RL ^a (µg)	ICAL (%RSD)	ISCV (%R)	CCV (%R)
Acetaldehyde	0.10	≤ 10	± 15	± 10
Acrolein ^b	0.25 ^d	≤ 10	± 15	± 10
Benzaldehyde	0.25	≤ 10	± 15	± 10
Crotonaldehyde	0.25	≤ 10	± 15	± 10
Formaldehyde	0.05	≤ 10	± 15	± 10
Hexanal	0.25	≤ 10	± 15	± 10
Isopentanal	0.25	≤ 10	± 15	± 10
MEK/Butyraldehydes ^c	0.25	≤ 10	± 15	± 10
m,p-Tolualdehyde	0.25	≤ 10	± 15	± 10
o-Tolualdehyde	0.25	≤ 10	± 15	± 10
Pentanal	0.25	≤ 10	± 15	± 10
Propanal	0.25	≤ 10	± 15	± 10
Acetone	0.25	≤ 10	± 15	± 10
Acetophenone*	N/A	≤ 10	± 15	± 10
Isophorone*	N/A	≤ 10	± 15	± 10
Heptaldehyde*	0.25	≤ 10	± 15	± 10
2,5-Dimethylbenzaldehyde*	0.25	≤ 10	± 15	± 10

^a Noted reporting limits are subject to change based on most current MDL study.

^b Because its derivative is not stable, when the target analyte list includes Acrolein the sample will need to be extracted in field. A special order should be placed with the laboratory during the project set-up stage.

^c Methyl Ethyl Ketone and the Butyraldehydes co-elute.

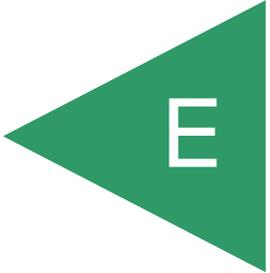
^d Not recommended.

* Special compounds upon request only.

Table 3. Summary of Calibration and QC Procedures for Method TO-11A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
5-Point Initial Calibration Curve (ICAL)	Analyzed in triplicate prior to sample analysis	%RSD \leq 10	Repeat calibration.
Initial Calibration Verification (ICV)	With each ICAL	%R = 85–115%	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis, after a maximum of every 10 injections, and at the end of the analytical batch	Within \pm 10% of the expected value	Check the system and re-analyze the standard. If the criteria cannot be met, re-calibrate the instrument. If the standard is biased low, re-analyze all samples since last acceptable CCV. If biased high and samples are “ND”, re-analysis is not required. “Q”-flag high recoveries.
Instrument (Solvent) Blank Analysis	Following analysis of Standards	Results less than the laboratory RL	Inspect the system and re-analyze the blank.
Laboratory Duplicates - Laboratory Control Spike Duplicate	One per analytical batch	RPD \leq 25%	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate the data.

APPENDIX

A solid green triangle pointing to the left, containing the letter 'E' in white.

E



OPERATIONS MANUAL

*TE-5170V Volumetric Flow Controlled
Total Suspended Particulate
High Volume Air Sampler*

**Tisch Environmental, Inc.
145 South Miami Avenue
Village of Cleves, Ohio 45002**

Toll Free: (877) 263 -7610 (TSP-AND-PM10)

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TE-5170V Volumetric Flow Controlled
Total Suspended Particulate High Volume Air Sampler

Welcome

We are the experts in high volume air sampling, lead sampling, lead samplers, particulate monitoring, particulate emissions, pesticide monitoring, pesticide sampling, total suspended particles, particulate sampler, Federal Reference Method PM-10, Federal Reference Method PM2.5, EPA Method TO-4A, EPA Method TO-9A, EPA Method TO-13A. TEI is a family business located in the Village of Cleves, Ohio. TEI employs skilled personnel who average over 20 years of experience each in the design, manufacture, and support of air pollution monitoring equipment. Our modern well-equipped factory, quality philosophy and experience have made TEI the supplier of choice for air pollution monitoring equipment. Now working on the fourth generation, TEI has state-of-the-art manufacturing capability and is looking into the future needs of today's environmental professionals.

Assistance

If you encounter problems or require detailed explanations, do not hesitate to contact Tisch Environmental offices by e-mail or phone.

Toll Free: (877) 263 -7610 (TSP AND-PM10)

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Introduction

EPA Standards

The following manual will instruct you in the unpacking, assembly, operation, calibration, and use of this product. For information on air sampling principles, procedures and requirements and to ensure compliance with government regulations, refer to Title 40 of the Code of Federal Regulations and consult **Appendix B to Part 50, Reference Method for Determination of Suspended Particulate Matter in the Atmosphere (High Volume Method)** or **Appendix G to Part 50, Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air**. For additional information, contact the local Environmental Protection Agency office serving your area.

Safety Precautions

Before using Tisch Environmental products, always review the corresponding operations manuals and take all necessary safety precautions, especially when working with electricity.

Important Safety Instructions

Read and understand all instructions. Do not dispose of these instructions. Failure to follow all instruction listed in this manual may result in electric shock, fire, and/or personal injury. When using an electrical device, basic precautions must always be followed, including the precautions listed in the safety section of this manual. Never operate this unit in the presence of flammable materials or vapors are present as electrical devices may produce arcs or sparks that can cause fire or explosions. Always disconnect power supply before attempting to service or remove any components. Never immerse electrical parts in water or any other liquid. Always avoid body contact with grounded surfaces when plugging or unplugging this device is wet or dangerous conditions.

Electrical Installation

Installation must be carried out by specialized personal only, and must adhere to all local safety rules. This unit can be used for different power supply versions; before connecting this unit to the power line, always check if the voltage shown on the serial number tag corresponds to the one on your power supply. This product does use grounded plugs and wires. Grounding provides the path of least resistance for electrical currents, thereby reducing the risk of electric shock to users. This system is equipped with electrical cords with internal ground wires and a grounding plug. The plug must be plugged into a matching outlet that is properly installed and grounded in accordance with all local codes and ordinances. Do not modify the plug provided. If plug will not fit outlet, have the proper corresponding outlet installed by a professional, qualified electrician.

Do Not Abuse Cords

In the event that any electrical component of this system needs to be transported, **DO NOT** carry the unit by its power cord or unplug the unit by yanking the cord from the outlet. **Pull the plugs, not the cords**, to reduce risk of damage to unit. Keep all cords away from heat, oil, sharp objects, and moving parts.

Extension Cords

It is always advisable to use the shortest extension cord possible. Grounded units require a three-wire extension cord. As the distance from the supply outlet increases, you must use a heavier gauge extension cord. Using extension cords with inadequately sized wires results in serious changes in voltage, resulting in a loss of power and possible damage to equipment. It is recommended to only use 10-gauge extension cords for this product. Never use cords that exceed one hundred feet. Outdoor extension cords must be marked with the suffix "W-A" (or "W" in Canada) to indicate that it is suitable for outdoor usage. Always ensure that extension cords are properly wired and in good electrical condition. Always replace damaged extension cords immediately, or seek repair from qualified electricians before further use. Remember to protect extension cords from sharp objects, excessive heat, and damp or wet conditions.

Product Description

Introduction

The High Volume Air Sampler (also known as a **lead sampler**) is the recommended instrument for sampling large volumes of air for the collection of TSP (Total Suspended Particulate). The TE-5170V TSP VFC sampler consists of a TE-5001 Anodized aluminum shelter, TE-5070 Aluminum Blower Motor Assembly, TE-5003V 8"x10" Stainless Steel Filter Holder with pressure tap, TE-5009 Continuous flow/pressure recorder, TE-10557 TSP Volumetric Flow Controller, TE-5007 Mechanical Timer, TE-5012 Elapsed Time Indicator, TE-5030 Manometer, TE-10618 male stagnation tube fitting.

Applications

- Ambient air monitoring to determine mass concentration of suspended particulate levels relative to air quality standards. The result is reported in micrograms per cubic meter.
- Impact of a specific source on ambient levels of suspended particulates by incorporating a "wind-direction-activation" modification which permits the sampler to operate only when conditions are such that a source-receptor relationship exists.

Calibration Requirements

TE-5170V TSP VFC High Volume Air Sampler should be calibrated at the site:

- Upon installation
- After routine maintenance
- Once every quarter (three months)
- After 360 sampling hours

Calibration Kits

The TE-5028 is the instrument used to calibrate the TE-5170V VFC TSP High Volume Air Sampler. It simulates change in the resistance by merely rotating the knob on the top of the calibrator. The infinite resolution lets the technician select the desired flow resistance. The TE-5028 calibration kit includes: carrying case, 30" slack tube water manometer, adapter plate, 3' piece of tubing, and TE-5028A orifice with flow calibration certificate. Optional electronic manometer is available.



Each TE-5028A is individually calibrated on a primary standard positive displacement device (Rootsmeter) which is directly traceable to NIST.

**** It is recommended by the EPA that each calibrator should be re-calibrated annually. (1998 Code of Federal Regulations Parts 50 to 51, Appendix B to Part 50, Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere, 9.2.17 page 30.)**

Parts

1. Shelter Box - 48" x 20" x 20" 72 lbs

TSP VFC Sampler
TE-5170V 110volt, 60hz
TE-5170VX 220volt, 50hz
TE-5170VXZ 220volt, 60hz



30" Water Manometer
TE-5030



8" X 10" VFC TSP
Stainless Steel Filter Holder
TE-5003V



TSP Volumetric Flow Controller
TSP
TE-10557TSP



24 Hour Chart Recorder
TE-5009 110volts, 60hz
TE-5009X 220volts, 50hz
TE-5009XZ 220volts, 60hz



7 Day Mechanical Timer
TE-5007 110volt, 60hz
TE-5007X 220volt, 50hz
TE-5007XZ 220volt, 60hz



VFC Blower Motor Assembly
TE-5070 110volt
TE-5070X 220volts



Elapsed Time Indicator
TE-5012 110volt, 60hz
TE-5012X 220volt, 50hz
TE-5012XZ 220volt, 60hz



Male Stagnation Fitting with
tubing
TE-10618



Bulk Head Fitting(attached to
shelter)
TE-10617



Box of Charts
TE-106



2. Lid Box - 19" x 14" x 14" 9 lbs
Gabled Roof
TE-5001-10



*** Save the shipping containers and packing material for future use.

Assembly

1. Open shelter box and remove Anodized Aluminum Shelter.
2. Taped to the bottom is the TE-5003V TSP VFC filter holder with TE-5005-9 gasket, TE-5030 30" water manometer and male tube fitting.
3. Strapped to the leg is the TE-10557TSP with look up table and the TE-5070 VFC Blower Motor Assembly.
4. Screw TE-5003V Filter Holder onto TE-10557TSP and TE-5070 Blower Motor Assembly (tubing, power cord, and pressure tap on side of filter holder to the right) make sure TE-5005-9 gasket is in place.
5. Lower Filter Holder, VFC, and Blower Motor down through top support pan on shelter.
6. Connect clear tubing from bulkhead fitting to pressure tap on side of filter holder.
7. Connect black tubing from the TE-5070 blower motor to the pressure tap located underneath the TE-5009 recorder
8. Open lid box

Gabled Roof Assembly

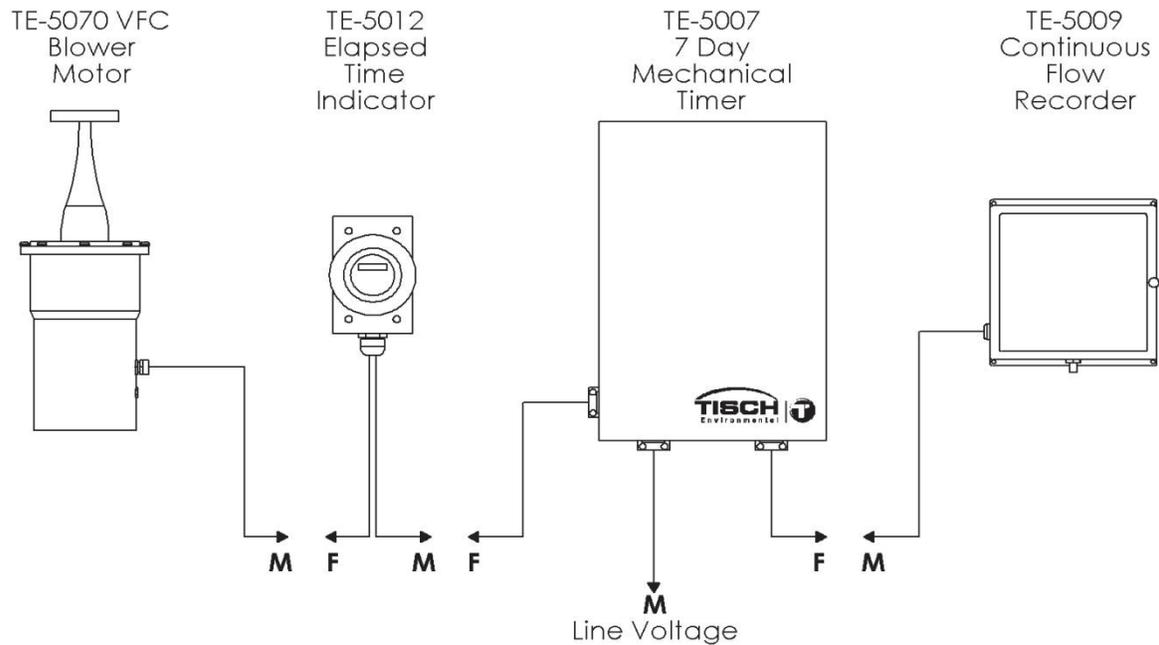
The following steps are accompanied by pictures to aid your understanding of gabled roof assembly. **Please be aware that the pictures are standardized and may not match the equipment that you are using.** The gabled roof is used on both TSP and PUF models, and the assembly procedure is the same for both products.

<p><u>Lid Hardware</u> 5 pcs 10-24 x 1/2 pan head screws 5 pcs 10-24 stop nuts 1 pc 6-32 x 3/8 pan head screw 1 pc 6-32 hex nut 1 pc 20" chain with "S" hook 1 pc TE-5001-10-9 roof back catch 1 pc TE-5001-10-10 front catch 1 pc TE-5001-10-11 rear lid hasp</p>	
<p><u>Step 1</u> Secure TE-5001-10-10 front catch to the shelter using 2 10-24 pan head screws with stop nuts. <i>*Do not tighten completely, this may need to be adjusted after final assembly*</i></p>	
<p><u>Step 2</u> Secure TE-5001-10-9 roof back catch to the back of shelter using #6-32 pan head screw with stop nut.</p>	
<p><u>Step 3</u> Secure TE-5001-10-11 rear lid hasp inside the lid with the slot angled up using (2) #10-24 pan head screws with stop nuts. <i>*Do not tighten completely, this may need to be adjusted after final assembly*</i></p>	

<p><u>Step 4</u> Remove (4) #10-24 x ½” pan head screws from the rear of the shelter, attach the lid to the shelter by placing the lid hinge plates on the “OUTSIDE” of the shelter, line the hinges up with the (4) threaded holes in the back of the shelter. Use the (4) #10-24X ½” pan head screws that were removed preciously to attach the lid hinges to the shelter. <i>*Tighten completely*</i></p>	
<p><u>Step 5</u> Adjust the front and rears catch to be sure that the lid slots lowers over it when closing. Tighten the roof back hasp and front catch completely.</p>	 
<p><u>Step 6</u> Attach the chain and “S” hook assembly to the side of the shelter with a #6-32 x 3/8” pan head screw.</p>	
<p><u>Step 7</u> The Lid can now be secured in an open or closed position with the “S” hook.</p>	

Electrical Set-Up

TE-5170-V Electrical Set-Up



M = 3-Prong Plug (Male)
 F = 3-Prong Socket (Female)

Note: Standard 3-prong plug may require adapter for use in global installations. Please consult your local electrical standards.

1. The TE-5070 VFC Blower Motor plug set connects into the TE-5012 Elapsed Time Indicator socket.
2. The plug of the ETI cord set plugs into the TE-5007 7-Day Mechanical Timer timed socket on the left side of timer.
3. The other socket on timer (on the right) is hot all the time and normally gets plugged into TE-5009 recorder.
4. The 3 prong plug of timer connects into the line voltage.

Operations

Calibration Procedure

The following is a step by step process of the calibration of a TE-5170V Volumetric Flow Controlled TSP Particulate Sampling System. Following these steps are example calculations determining the calibration flow rates for the sampler. The air flow through these types of sampling systems is controlled by a Volumetric Flow Controller (VFC) or dimensional venturi device.

This calibration differs from that of a mass flow controlled TSP sampler in that a slope and intercept does not have to be calculated to determine air flows. Also, the calibrator orifice Q_{actual} slope and intercept from the orifice certification worksheet can be used here, unlike a mass flow controlled TSP where $Q_{standard}$ slope and intercept are used. The flows are converted from actual to standard conditions when the particulate concentrations are calculated. With a Volumetric Flow Controlled (VFC) sampler, the calibration flow rates are provided in a Flow Look Up Table that accompanies each sampler.

The attached example calibration worksheet uses a TE-5028A Variable Orifice Calibrator which uses an adjustable or variable orifice, which we recommend when calibrating a VFC.

Proceed with the following steps to begin the calibration:

Step 1: Mount the calibrator orifice and top loading adapter plate to the sampler. A sampling filter is generally not used during this procedure. Tighten the top loading adapter hold down nuts securely for this procedure to assure that no air leaks are present.

Step 2: Turn on the sampler and allow it to warm up to its normal operating temperature.

Step 3: Conduct a leak test by covering the holes on top of the orifice and pressure tap on the orifice with your hands. Listen for a high-pitched

squealing sound made by escaping air. If this sound is heard, a leak is present and the top loading adapter hold-down nuts need to be re-tightened.

Avoid running the sampler for longer than 30 seconds at a time with the orifice blocked. This will reduce the chance of the motor overheating. Also, never try this leak test procedure with a manometer connected to the pressure tap on the calibration orifice or the pressure tap on the side of the sampler. Liquid from either manometer could be drawn into the system and cause motor damage.

Step 4: Connect one side of a water manometer or other type of flow measurement device to the pressure tap on the side of the orifice with a rubber vacuum tube. Leave the opposite side of the manometer open to the atmosphere.

Step 5: Connect a water manometer to the quick disconnect located on the side of the aluminum outdoor shelter (this quick disconnect is connected to the pressure tap on the side of the filter holder).

Step 6: Make sure the TE-5028A orifice is all the way open (turn the black knob counter clock-wise). Record both manometer readings, the one from the orifice and the other from the side of the sampler. To read a manometer one side goes up and the other side goes down, you add both sides, this is your inches of water. Repeat this process for the other four points by adjusting the knob on the variable orifice (just a slight turn) to four different positions and taking four different readings. You should have five sets of numbers, ten numbers in all.

Step 7: Remove the variable orifice and the top loading adapter and install a clean filter. Set your timer.

Step 8: Record the ambient air temperature, the ambient barometric pressure, the sampler serial number, the orifice serial number, the orifice Qactual slope and intercept with date last certified, todays date, site location and the operators initials.

One example calibration sheet and one blank calibration sheet are attached to this manual. To download the electronic spreadsheet, please visit www.tisch-env.com (download the TE-5170V High Vol TSP VFC with G-

Factor excel spreadsheet). It is highly recommended to download the electronic excel spreadsheet and use spreadsheet features to complete calculations. (If you do not have a G Factor then go to “TE-5170V High Vol. TSP” excel spreadsheet on web site and then go to page 20)

G-Factor Excel Spreadsheet Instructions

The TE-5170V calibration worksheet can be found on our website at www.tisch-env.com. If you have the G Factor that accompanies each VFC, go to “TE-5170V High Vol. TSP VFC with G-Factor”, if you do not have a G Factor then go to “TE-5170V High Vol. TSP” excel spreadsheet.

Note: Calibration orifices should be sent back to Tisch Environmental for calibration on an annual basis per US EPA Compendium Method IO-2.1 Part 7.3.2 *Sampling of Ambient Air For Total Suspended Particulate Matter (SPM) and PM₁₀ Using High Volume (HV) Sampler*

1. Enter the following information in the corresponding cells in the worksheet:

Site Information

Location	The location of the instrument
Date	Current Date
Tech	Technician performing the calibration
Serial #	Serial number of the instrument, Pxxxx
VFC G Factor	The g-factor of the VFC you are calibrating. This can be found on the lookup table documentation (first page of this doc) or the sticker located on the VFC.

Calibration Orifice Information

Make	The make of the orifice, typically Tisch Environmental
Model	The model number of the orifice, typically TE-5028A

Serial #	The Serial number of the calibration orifice you are using
Qa Slope (m)	The Qa slope of the calibration orifice you are using. This is found on the calibration documentation provided with the calibration orifice
Qa Int (b)	The Qa intercept of the calibration orifice you are using. This is found on the calibration documentation provided with the calibration orifice
Calibration Due Date	The date that the calibration of the orifice is due. Orifice should be calibrated on an annual basis. Call Tisch Environmental at 1-TSP-AND-PM10 to schedule a recalibration.

Ambient Conditions

Temp (Deg F)	Enter the current ambient temperature at calibration, Ta in Degrees K and Ta in degrees C will be calculated by the spreadsheet
Barometric Pressure	Enter the ambient barometric pressure (Pa) inches of Hg at calibration, the Pa in mmHg will be calculated by the spreadsheet

2. Enter the calibration information by performing each calibration point and entering the following information into each corresponding row for each point:

Calibration Information

Orifice “H ₂ O	The pressure measured at the orifice port using a manometer. The first point should be performed with the orifice knob turned counter-clockwise or wide open, then four consecutive points turning the orifice knob clockwise (not closed) Good idea to take a few extra points here.
Sampler “H ₂ O	The pressure measured at the sampler side port using a manometer (clear tubing that is connect to bulk head fitting that is connected to side of filter holder)

The calibrator flow is calculated (Qa) using the formula:

$$Qa = \frac{1}{Slope} \times \sqrt{"H2Ox \left(\frac{Ta}{Pa}\right) - Intercept}$$

The calculated flow in m³/min will be calculated using the g-factor formula, this flow will correspond to the flow found in the lookup table supplied with the VFC.

The percent difference will be calculated using the formula:

$$\% \text{ Difference} = \frac{\text{Calculated Flow} - (Qa) \text{ Calibrator Flow}}{(Qa) \text{ Calibrator Flow}} \times 100$$

As per stated in the method IO-2.1, % Difference calculations should be less than +-4%

3. To calculate the total air volume during the sample enter the following information:

Calculate Total Air Volume Using G-Factor

Average Temperature	Enter the average temperature of the sample throughout the sample period in Deg F. The temperature will then be calculated in Deg K
Barometric Pressure	Enter the average barometric pressure of the sample throughout the sample period in Inches of Hg. The barometric pressure in mmHg will then be calculated
Clean Filter "H ₂ O	Enter the clean filter pressure in inches of water prior to sampling
Dirty Filter "H ₂ O	Enter the dirty filter pressure in inches of water after sampling. The average sample pressure will then be calculated in mmHg
Runtime	Enter the total runtime in hours (xx.xx) of the sample

Using the g-factor formula, Po/Pa will be calculated and an average flow rate of the sample will be calculated in m³/min. Using this information the total sample volume will be calculated.

If you do not have a G Factor, go to TE-5170V High Vol. TSP on web site

An example of a Volumetric Flow Controlled Sampler Calibration Data Sheet has been attached with data filled in from a typical calibration. This includes the transfer standard orifice calibration relationship which was taken from the Orifice Calibration Worksheet that accompanies the calibrator orifice.

The slope and intercept are taken from the Qactual section of the Orifice Calibration Worksheet.

The first step is to convert the orifice readings to the amount of actual air flow they represent using the following equation:

$$Q_a = 1/m[\text{Sqrt}((H_2O)(T_a/P_a)) - b]$$

where:

Q_a = actual flow rate as indicated by the calibrator orifice, m³/min

H₂O = orifice manometer reading during calibration, in. H₂O

T_a = ambient temperature during calibration, K (K = 273 + °C)

P_a = ambient barometric pressure during calibration, mm Hg

m = slope of Q actual orifice calibration relationship

b = intercept of Q actual orifice calibration relationship.

Once these actual flow rates have been determined for each of the five run points, they are recorded in the column titled Q_a, and are represented in cubic meters per minute. EPA guidelines state that at least three of these calibrator flow rates should be between 1.1 to 1.7 m³/min (39 to 60 CFM). This is the acceptable operating flow rate range of the sampler. If this condition is not met, the sampler should be recalibrated. An air leak in the calibration system may be the source of this problem. In some cases, a filter may have to be in place during the calibration to meet this condition.

The sampler H₂O readings need to be converted to mm Hg and recorded in the column titled P_f. This is done using the following equation:

$$P_f = 25.4 (\text{in. H}_2\text{O}/13.6)$$

where: P_f is recorded in mm Hg

in. H₂O = sampler side pressure reading during calibration.

P_o/P_a is calculated next. This is used to locate the sampler calibration air flows found in the Look Up Table. This is done using the following equation:

$$P_o/P_a = 1 - P_f/P_a$$

where: P_a = ambient barometric pressure during calibration, mm Hg.

Using P_o/P_a and the ambient temperature during the calibration, consult the Look Up Table to find the actual flow rate. Record these flows in the column titled Look Up.

Calculate the percent difference between the calibrator flow rates and the sampler flow rates using the following equation:

$$\% \text{ Diff.} = (\text{Look Up Flow} - Q_a) / Q_a * 100$$

where: Look Up Flow = Flow found in Look Up Table, m³/min
 Q_a = orifice flow during calibration, m³/min.

The EPA guidelines state that the percent difference should be within + or - 3 or 4%. If they are greater than this a leak may have been present during calibration and the sampler should be recalibrated.

Example Calculations

The following example problems use data from the attached VFC sampler calibration worksheet.

After all the sampling site information, calibrator information, and meteorological information have been recorded on the worksheet, actual air flows need to be determined from the orifice manometer readings taken during the calibration using the following equation:

$$1. Q_a = 1/m[\text{Sqrt}((H_2O)(T_a/P_a)) - b]$$

where:

2. Q_a = actual flow rate as indicated by the calibrator orifice, m³/min
3. H_2O = orifice manometer reading during calibration, in. H_2O
4. T_a = ambient temperature during calibration, K ($K = 273 + \text{ }^\circ\text{C}$)
5. P_a = ambient barometric pressure during calibration, mm Hg
6. m = slope of Q actual orifice calibration relationship
7. b = intercept of Q actual orifice calibration relationship.

Note that the ambient temperature is needed in degrees Kelvin to satisfy the Q_a equation. Also, the barometric pressure needs to be reported in millimeters of mercury (if sea level barometric pressure is used it must be corrected to the site elevation). In our case the two following conversions may be needed:

$$8. \text{ degrees Kelvin} = [5/9 (\text{degrees Fahrenheit} - 32)] + 273$$

$$9. \text{ millimeters of mercury} = 25.4(\text{inches of H}_2\text{O}/13.6)$$

Inserting the numbers from the calibration worksheet test number one we get:

$$10. \quad Q_a = 1/.92408[\text{Sqrt}((3.8)(293/749.3)) - (-0.00383)]$$

$$11. \quad Q_a = 1.0821573[\text{Sqrt}((3.8)(.3910316)) + 0.00383]$$

$$12. \quad Q_a = 1.0821573[\text{Sqrt}(1.48592) + 0.00383]$$

$$13. \quad Q_a = 1.0821573[1.2189831 + 0.00383]$$

$$14. \quad Q_a = 1.0821573[1.2228131]$$

$$15. \quad Q_a = 1.323$$

It is possible that your answers to the above calculations may vary. This is most likely due to different calculators carrying numbers to different decimal points. This should not be an area of concern as generally these variations are slight.

With Q_a determined, the sampler H_2O reading needs to be converted to mm Hg using the following equation:

$$16. \text{ Pf} = 25.4 (\text{in. H}_2\text{O}/13.6)$$

where:

17. Pf is recorded in mm Hg

18. in. H_2O = sampler side pressure reading during calibration

Inserting the numbers from the worksheet:

$$19. \quad \text{Pf} = 25.4(6.4/13.6)$$

$$20. \quad \text{Pf} = 25.4(.4705882)$$

$$21. \quad \text{Pf} = 11.95294 \text{ mm Hg}$$

Po/Pa is calculated next. This is done using the following equation:

22. $P_o/P_a = 1 - P_f/P_a$
23. where: Pa = ambient barometric pressure during calibration, mm Hg.
Inserting the numbers from the worksheet:
24. $P_o/P_a = 1 - 11.95294/749.3$
25. $P_o/P_a = 1 - .0159521$
26. $P_o/P_a = .984$

Use Po/Pa and the ambient temperature during the calibration (Ta) to locate the flow for the calibration point in the Look Up table. Record this in the column titled Look Up. Calculate the percent difference using the following equation:

27. $\% \text{ Difference} = (\text{Look Up flow} - Q_a)/Q_a * 100$
Inserting the numbers from the worksheet:
28. $\% \text{ Difference} = (1.287 - 1.323)/1.323 * 100$
29. $\% \text{ Difference} = (-0.036)/1.323 * 100$
30. $\% \text{ Difference} = (-0.0272108) * 100$
31. $\% \text{ Difference} = -2.72$

It is possible that your answers to the above calculations may vary. This is most likely due to different calculators carrying numbers to different decimal points. This should not be an area of concern as generally these variations are slight.

The above calculations have to be performed for all five calibration points. Once this is done, the calibration is complete.

Use of Look-Up-Table to Determine Flow Rate

(NOTE: Individual Look-Up Tables will vary.)

1. Suppose the ambient conditions are:

Temperature: $T_a = 20^\circ\text{C}$

Barometric Pressure: $P_a = 749.3$ mm Hg (this must be station pressure which is not corrected to sea level)

2. Assume system is allowed to warm up for stable operation.

3. Measure filter pressure differential, P_f . This reading is the set-up reading plus pick-up reading divided by 2 for an average reading. This is taken with a differential manometer with one side of the manometer connected to the stagnation tap on the filter holder (or the Bulkhead Fitting) and the other side open to the atmosphere. Filter must be in place during this measurement.

Assume that:

Set-up Reading (clean filter): $P_f = 12.6$ in H₂O

Pick-up Reading (dirty filter): $P_f = 16.0$ in H₂O

$$P_f = (12.6 + 16.0)/2 = 14.3 \text{ in H}_2\text{O}.$$

4. Convert $P_f =$ to same units as barometric pressure.

$$P_f = 14.3 \text{ in H}_2\text{O} / 13.61 \times 25.4 = 26.687729 \text{ mm Hg}$$

$$P_f = 26.69 \text{ mm Hg}$$

5. Calculate pressure ratio.

$$P_o/P_a = 1 - (P_f/P_a)$$

NOTE: P_f and P_a MUST HAVE CONSISTENT UNITS

$$P_o/P_a = 1 - (26.69 / 749.3)$$

$$P_o/P_a = .964$$

6. Look up Flow Rate from table.

Table 1 is set up with temperature in °C and the Flow Rate is read in units of m³/min (actual, ACMM). In table 2 the temperature is in °F and Flow Rate is read in ft³/min (actual, ACFM).

a) For the example we will use Table 1.

Locate the temperature and pressure ratio entries nearest the conditions of:

$$T_a = 20^\circ\text{C}$$

$$P_o/P_a = .964$$

Example: Look-Up Table for Actual Flow Rate in Units of m³/min

	Temperature °C				
Po/Pa	20	22	24	26	28
0.961	1.250	1.254	1.258	1.261	1.265
0.962	1.251	1.255	1.259	1.263	1.266
0.963	1.253	1.256	1.260	1.264	1.268
0.964	1.254	1.258	1.262	1.265	1.269
0.965	1.255	1.259	1.263	1.267	1.271
0.966	1.257	1.261	1.264	1.268	1.272

b) The reading of flow rate is:

$$Q_a = 1.254 \text{ m}^3/\text{min (actual)}$$

If your Po/Pa number is not in look up table ie; >.979 then interpolate.

7. Determine flow rate in terms of standard air.

$$Q_{\text{std}} = 1.254 \text{ m}^3/\text{min} \left(\frac{749.3 \text{ mm Hg}}{760 \text{ mm Hg}} \right) \left(\frac{298\text{K}}{(273 + 20) \text{K}} \right)$$

$$Q_{\text{std}} = 1.257 \text{ std m}^3/\text{min}$$

Sampler Operation

1. After performing calibration procedure, remove filter holder frame by loosening the four wing nuts allowing the brass bolts and washers to swing down out of the way. Shift frame to one side and remove.
2. Carefully center a new filter, rougher side up, on the supporting screen. Properly align the filter on the screen so that when the frame is in position the gasket will form an airtight seal on the outer edges of the filter.
3. Secure the filter with the frame, brass bolts, and washers with sufficient pressure to avoid air leakage at the edges (make sure that the plastic washers are on top of the frame).
4. Wipe any dirt accumulation from around the filter holder with a clean cloth.
5. Close shelter lid carefully and secure with the "S" hook.
6. Make sure all cords are plugged into their appropriate receptacles and the rubber tubing between the blower motor pressure tap and the TE-5009 continuous flow recorder is connected (be careful not to pinch tubing when closing door).
7. Prepare TE-5009 continuous flow recorder as follows:
 - a. Clean any excess ink and moisture on the inside of recorder by wiping with a clean cloth.
 - b. Depress pen arm lifter to raise pen point and carefully insert a fresh chart.
 - c. Carefully align the tab of the chart to the drive hub of the recorder and press gently with thumb to lower chart center onto hub. Make sure chart is placed under the chart guide clip and the time index clip so it will rotate freely without binding. Set time by rotating the drive hub clock-wise until the correct time on chart is aligned with time index pointer.

- d. Make sure the TE-160 pen point rests on the chart with sufficient pressure to make a visible trace.
8. Prepare the 7-Day Timer as instructed below.
9. At the end of the sampling period, remove the frame to expose the filter. Carefully remove the exposed filter from the supporting screen by holding it gently at the ends (not at the corners). Fold the filter lengthwise so that sample touches sample.
10. It is always a good idea to contact the lab you are dealing with to see how they may suggest you collect the filter and any other information that they may need.

Timer Preparation

TE-5007 7-Day Mechanical Timer

1. To set the "START" time, attach a (bright) "ON" tripper to the dial face on the desired "START" time. Tighten tripper screw securely.
2. To set the "STOP" time, attach a (dark) "OFF" tripper to the dial face on the desired "STOP" time. Tighten tripper screw securely.
3. To set current time and day, grasp dial and rotate **clockwise only** until correct time and day appear at time pointer.

Troubleshooting

note: this is a general troubleshooting guide, not all problem may apply to every sampler

<u>Problem</u>	<u>Solution</u>
Brush Motor Won't Turn On	<ul style="list-style-type: none"> -Check Motor brushes(Change every 300-400 hours) -Check Motor(Should be replaced after 2 brush changes about 1200 hours) -Check power supply -Ensure that all electrical connections are secure -Make sure timer is on -Make sure flow controller(if applicable) is adjusted properly -Check for loose or damaged wires
Brushless Motor Won't Turn On	<ul style="list-style-type: none"> -Ensure that all electrical connections are secure -Check power supply -Make sure timer is on -Check for loose or damaged wires
Mechanical timer not working	<ul style="list-style-type: none"> -Make sure trippers are set properly -Make sure that trippers are not pressed against switch at start up, the timer need to rotate a few degrees before the trippers hit the switch -Check for loose or damages wires -Check power supply -Check electrical hook up diagram to ensure correct installation -Check Motor
Digital timer not working	<ul style="list-style-type: none"> -Check timer settings -Make sure current date and time are correct -Make sure power cords are properly connected -Check fuse on main PC board (F3) -Check Power Supply -Check Motor
Elapsed Time Indicator not working	<ul style="list-style-type: none"> -Check Power Supply -Check electrical connections

Flow Rate Too Low	<ul style="list-style-type: none"> -Check for leaks -Check filter media placement -Ensure only one piece of filter paper is installed -Check Flow Controller -Check flow valve(TE-1000PUF samplers only) -Ensure proper voltage is being supplied -Check calibration
Chart Recorder not working	<ul style="list-style-type: none"> -Replace pen point -Make sure pen point is touching chart -Make sure pen point is on "0" -Make sure tubing from motor is in place -Check Power Supply -Check motor
Air Leaks	<ul style="list-style-type: none"> -Make sure all gaskets are in place -Make sure all connections are secure -Makes sure connections are not over tightened -Check for damaged components: Filter holder screen, gaskets, motor flanges

Maintenance and Care

A regular maintenance schedule will allow a monitoring network to operate for longer periods of time without system failure. Our customers may find that the adjustments in routine maintenance frequencies are necessary due to the operational demands on their sampler(s). We recommend that the following cleaning and maintenance activities be observed until a stable operating history of the sampler has been established.

1. Make sure all gaskets (including motor cushion) are in good shape and that they seal properly.
2. The power cords should be checked for good connections and for cracks (replace if necessary).

CAUTION: DO NOT allow power cord or outlets to be immersed in water!

3. Inspect the filter screen and remove any foreign deposits.
4. Inspect the filter holder frame gasket each sample period and make sure of an airtight seal.
5. Check or replace motor brushes every 300 to 400 running hours.
6. Make sure elapsed time indicator is working properly.

Motor Brush Replacement

(110 volt Brush part #TE-33392)

(220 volt Brush part #TE-33378)

The following steps are accompanied by pictures to aid your understanding of motor brush replacement procedures. **Please be aware that the pictures are standardized and may not exactly match the equipment that you are using.** Motor brush removal and replacement does not change based on motor or brush type, so do not be confused if your equipment differs from what is pictured.

CAUTION: Unplug the unit from any line voltage sources before any servicing of blower motor assembly.

1. Remove the VFC device by removing the eight bolts. This will expose the gasket and the TE-115923 motor.
2. Turn assembly on side, loosen the cord retainer and then push cord into housing and at the same time let motor slide out exposing the brushes.
3. Looking down at motor, there are 2 brushes, one on each side. Carefully pry the brass quick disconnect tabs (the tabs are pushed into end of brush) away from the expended brushes and toward the armature. Try to pry the tabs as far as you can without damaging the armature.
4. With a screwdriver loosen and remove brush holder clamps and release brushes. Carefully, pull quick disconnect tabs from expended brushes.



5. Carefully slide quick disconnect tabs into tab slot of new brush.



6. Push brush carbon against armature until brush housing falls into brush slot on motor.



7. Put brush holder clamps back onto brushes.
8. Make sure quick disconnect tabs are firmly seated into tab slot. Check field wires for good connections.



9. Assemble motor after brush replacement by replacing the motor cushion and metal motor ring(s), placing housing over and down on the motor (at same time pull power cord out of housing), being careful not to pinch any motor wires beneath the motor spacer ring.

10. Secure power cord with the cord retainer cap.

11. Replace VFC device on top of motor making sure to center gasket.

****IMPORTANT**** To enhance motor life:

1. Change brushes before brush shunt touches armature.
2. Seat new brushes by applying 50% voltage for 10 to 15 minutes, the TE-5075 brush break in device allows for the 50% voltage.



110v VFC Motor



110v VFC Motor Brush (orange)



220v VFC Motor



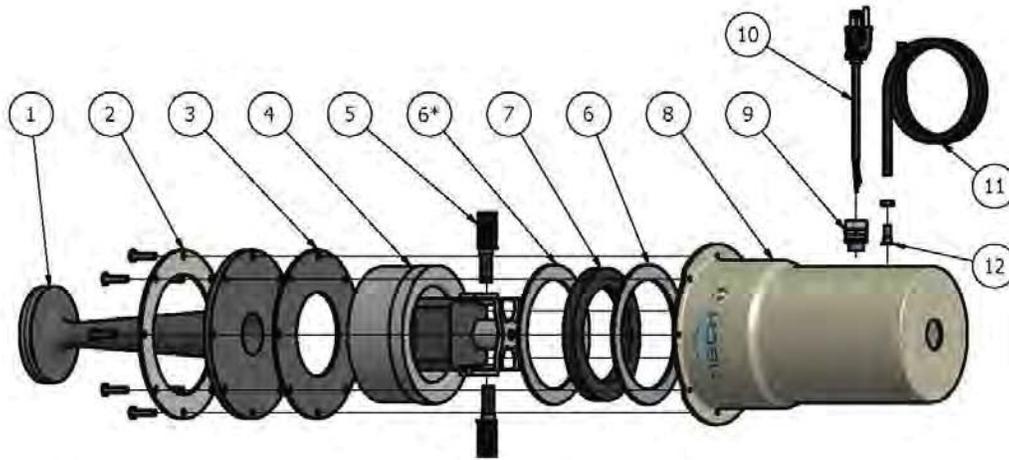
220v VFC Motor Brush (brown)

Warranty

Instruments manufactured by Tisch Environmental, Inc. are guaranteed by warranty to be free of defects in materials and workmanship for one year after shipment from Tisch Environmental factories. The liability of Tisch Environmental, Inc. is limited to servicing or replacing any defective part of any instrument returned to the factory by the original purchaser. All service traceable to defects in original material or workmanship is considered warranty service and is performed free of charge. The expense of warranty shipping charges to and from our factory will be borne by Tisch Environmental. Service performed to rectify an instrument malfunction caused by abuse, acts of god or neglect, and service performed after the one-year warranty period will be charged to the customer at the current prices for labor, parts, and transportation. Brush-type and brushless motors will carry a warranty as far as the original manufacture will pass through its warranty to Tisch Environmental, Inc. The right is reserved to make changes in construction, design specifications, and prices without prior notice.

Assembly Drawings

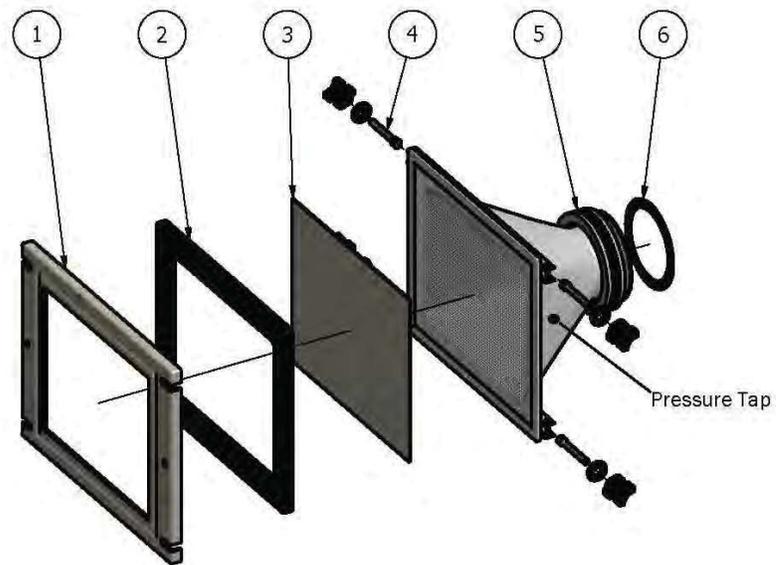
TE-5070 Blower Motor Assembly with VFC Device



TE-5070 w/ TE-10557 Assembly			
ITEM	QTY	PART NUMBER	DESCRIPTION
1	1	TE-10557	Volumetric Flow Controller
2	1	TE-5070-3	Clamp Ring w/ 8 Hole Pattern for VFC Motor Housing
3	1	TE-5070-1	Gasket w/ 8 Holes VFC Flange Gasket
4	1	TE-115923	Motor for 110V VFC Blower
		TE-116111	Motor for 220V VFC Blower
5	2	TE-33392	Motor Brushes for 110V Motor VFC
		TE-33378	Motor Brushes for 220V Motor VFC
7	1	TE-5005-4	Motor Cushion
6	2	TE-5005-5	Motor Spacer Ring
8	1	TE-5070-2	VFC Aluminum Blower Motor Housing
9	1	TE-5005-7	Cord Retainer w/ Nut
10	1	TE-5010-4	Power Cord
11	1	TE-5005-6	Tubing 3 ft. Piece
12	1	TE-5005-8	Pressure Tap

*ONLY FOR 110V MOTOR TE-115923

TE-5003V Filter Holder Assembly



TE-5003V Filter Holder Assembly			
ITEM	QTY	PART NUMBER	DESCRIPTION
1	1	TE-3000-2	Hold Down Frame
2	1	TE-5018	8" x 10" Gasket
3	1	N/A	Filter Paper
4	4	TE-5003-9	Plastic Thumb Nut, Brass Bolt, Washer, and Rivet
5	1	TE-5028-9	Aluminum Threaded Ring
6	1	TE-5005-9	Filter Holder Gasket (Between Filter Holder and Blower Motor)

Calibration Worksheets



TE-5170V Calibration Worksheet

Site Information

Location: Cleves Ohio	Site ID: 145	Date: 31-Oct-14
Sampler: TE-5170V	Serial No: P8644 TSP	Tech: Jim Tisch

Site Conditions

Temp (deg F): 68.0	Barometric Press (in Hg): 29.50
Ta (deg K): 293	Pa (mm Hg): 749
Ta (deg C): 20	

Calibration Orifice

Make: Tisch	Qa Slope: 0.92408
Model: TE-5028A	Qa Intercept: -0.00383
Serial#: 2978.00	Calibration Due Date: 24-Oct-15

Calibration Data

Run Number	Orifice "H2O	Qa (m3/min)	Sampler "H2O	Pf (mm Hg)	Po/Pa	Look Up (m3/min)	% Diff
1	3.80	1.323	6.40	11.944	0.984	1.287	-2.72
2	3.80	1.323	6.80	12.691	0.983	1.286	-2.80
3	3.80	1.323	7.20	13.437	0.982	1.284	-2.95
4	3.75	1.315	9.25	17.263	0.977	1.278	-2.81
5	3.75	1.315	10.20	19.036	0.975	1.275	-3.04

Calculations

Calibrator Flow (Qa) = 1/Slope*(SQRT(H2O*(Ta/Pa))-Intercept)

Pressure Ratio (Po/Pa) = 1-Pf/Pa

% Difference = (Look Up Flow-Calibrator Flow)/Calibrator Flow* 100

NOTE: Ensure calibration orifice has been certified within 12 months of use

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TE-5170V Sampler Calibration Worksheet (Using G-Factor)

Site and Calibration Information

<u>Site</u>		<u>Calibration Orifice</u>	
Location:	Cleves, OH	Make:	Tisch Environmental
Date:	Oct 31, 2014	Model:	TE-5028A
Tech.:	Jim Tisch	Serial:	1179
Sampler:	TE-5170V	Qa Slope (m):	0.92408
Serial #:	28644 TSP	Qa Int (b):	-0.00383
VFC G-Factor:	0.0974264900	Calibration due date:	10/24/15

Ambient Conditions

Temp (deg F):	68.0	Barometric Press (in Hg):	29.50
Ta (deg K):	293	Pa (mm Hg):	749.3
Ta (deg C):	20.0		

Calibration Information

Run Number	Orifice "H2O	Qa m3/min	Sampler "H2O	Pf mm Hg	Po/Pa	Calculated m3/min	% of Diff
1	3.80	1.323	6.40	11.944	0.984	1.287	-2.72
2	3.80	1.323	6.80	12.691	0.983	1.286	-2.80
3	3.80	1.323	7.20	13.437	0.982	1.284	-2.95
4	3.75	1.315	9.25	17.263	0.977	1.277	-2.81
5	3.75	1.315	10.20	19.036	0.975	1.274	-3.04

Calculate Total Air Volume Using G-Factor

Enter Average Temperature During Sampling Duration (Deg F)	62.00
Average Temperature During Sampling Duration (Deg K)	289.67
Enter Average Barometric Pressure During Sampling Duration (In Hg)	29.40
Average Barometric Pressure During Sampling (mm Hg)	746.76
Enter Clean Filter Sampler Inches of Water	12.60
Enter Dirty Filter Sampler Inches of Water	16.00
Average Filter Sampler (mm Hg)	26.69
Enter Total Runtime in Hours (xx.xx)	23.90
	Po/Pa : 0.964
	Calculated Flow Rate (m3/min): 1.254
	Total Flow (m3): 1797.57

Calculations

$$\text{Calibrator Flow (Qa)} = 1/\text{Slope} * (\text{SQRT}(\text{H2O} * (\text{Ta}/\text{Pa})) - \text{Intercept})$$

$$\text{Pressure Ratio (Po/Pa)} = 1 - \text{Pf}/\text{Pa}$$

$$\% \text{ Difference} = (\text{Look Up Flow} - \text{Calibrator Flow}) / \text{Calibrator Flow} * 100$$

NOTE: Ensure calibration orifice has been certified within 12 months of use

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Calibration Certificate



TISCH ENVIRONMENTAL, INC.
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 877.263.7610 TOLL FREE
 513.467.9009 FAX

ORIFICE TRANSFER STANDARD CERTIFICATION WORKSHEET TE-5028A

Date - Oct 24, 2014 Roots-meter S/N 9833620 Ta (K) - 296
 Operator Tisch Orifice I.D. - 2978 Pa (mm) - 755.65

PLATE OR VDC #	VOLUME START (m3)	VOLUME STOP (m3)	DIFF VOLUME (m3)	DIFF TIME (min)	METER	ORIFICE
					DIFF Hg (mm)	DIFF H2O (in.)
1	NA	NA	1.00	1.1880	4.5	1.50
2	NA	NA	1.00	0.9230	7.5	2.50
3	NA	NA	1.00	0.8380	9.0	3.00
4	NA	NA	1.00	0.7790	10.5	3.50
5	NA	NA	1.00	0.5860	18.0	6.00

DATA TABULATION

Vstd	(x axis) Qstd	(y axis)	Va	(x axis) Qa	(y axis)
0.9950	0.8375	1.2254	0.9940	0.8367	0.7665
0.9910	1.0737	1.5819	0.9901	1.0727	0.9896
0.9891	1.1803	1.7329	0.9881	1.1791	1.0840
0.9871	1.2671	1.8718	0.9861	1.2659	1.1709
0.9771	1.6674	2.4507	0.9761	1.6657	1.5331
Qstd slope (m) =		1.47574	Qa slope (m) =		0.92408
intercept (b) =		-0.00613	intercept (b) =		-0.00383
coefficient (r) =		0.99985	coefficient (r) =		0.99985
y axis = SQRT[H2O(Pa/760) (298/Ta)]			y axis = SQRT[H2O(Ta/Pa)]		

CALCULATIONS

$$Vstd = \text{Diff. Vol} [(Pa - \text{Diff. Hg}) / 760] (298 / Ta)$$

$$Qstd = Vstd / \text{Time}$$

$$Va = \text{Diff Vol} [(Pa - \text{Diff Hg}) / Pa]$$

$$Qa = Va / \text{Time}$$

For subsequent flow rate calculations:

$$Qstd = 1/m \{ [\text{SQRT} (H2O (Pa/760) (298/Ta))] - b \}$$

$$Qa = 1/m \{ [\text{SQRT} H2O (Ta/Pa)] - b \}$$

APPENDIX

