# Quality Assurance Program Plan for the Analysis of Soil-Gas Samples Collected with BEACON's Passive Soil-Gas System

# Prepared by

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Section No. Contents Revision No. 8

Date: May 25, 2010

Page 1 of 2

#### **Table of Contents**

1 0	T . 1 .	
1.0	Introducti	nn
1.0	muoducu	ננטו

- 2.0 Organization and Responsibilities
  - 2.1 Structure
  - 2.2 Roles and Responsibilities
    - 2.2.1 President
    - 2.2.2 Laboratory Director
    - 2.2.3 Analysts
    - 2.2.4 Operations Manager
- 3.0 Quality Assurance Objectives for Measurement Data
  - 3.1 Precision
  - 3.2 Accuracy
  - 3.3 Representativeness
  - 3.4 Completeness
  - 3.5 Comparability
  - 3.6 Project QA Objectives
- 4.0 Sample Custody Procedures
  - 4.1 Chain-Of-Custody Form
  - 4.2 Sample Receipt
  - 4.3 Sample Security and Accessibility
  - 4.4 Sample Retention and Disposal
  - 4.5 Document Control
    - 4.5.1 Record Keeping
    - 4.5.2 Laboratory Records
- 5.0 Calibration Procedures and Frequency
  - 5.1 Calibration Program
  - 5.2 Chemical Calibration
    - 5.2.1 Initial Calibration
    - 5.2.2 Continuing Calibration
    - 5.2.3 GC/MS Tuning and Mass Calibration
    - 5.2.4 Analytical Standards

Section No. Contents Revision No. 8

Date: May 25, 2010

Page 2 of 2

# **Table of Contents**

6.0	Analytical Procedures			
	6.1	Analytical Method		
	6.2	Instrumentation		
	6.3	Detection Limits		
7.0	Data Reduction, Validation, and Reporting			
	7.1	Data Collection		
	7.2	Data Reduction		
	7.3	Data Validation		
	7.4	Reporting		
	7.5	Data Storage		
8.0	Internal Quality Control Checks and Frequency			
	8.1	Calibration		
	8.2	Blanks		
	8.3	Surrogates		
	8.4	Duplicates		
	8.5	General Laboratory Controls		
9.0	Performance and System Audits			
10.0	Preventative Maintenance Procedures			
11.0 Statistical Assessment of Data Quality		tical Assessment of Data Quality		
	11.1	Calculation of Precision		
	11.2	Calculation of Accuracy		
12.0	Nonconformance and Corrective Action Procedures			
	12.1	Analytical Systems		
	12.2	Samples, Sample Receipt, and Chain-of-Custody Documents		
	12.3	General Laboratory Equipment		
	12.4	Laboratory Reports and Documentation		

Performance Sample Evaluations

Quality Assurance Reports to Management

12.5

13.0

Section No. 1.0 Revision No. 8 Date: May 25, 2010

Page 1 of 1

# 1.0 INTRODUCTION

The objective of Beacon Environmental Services, Inc. (BEACON) is to provide analytical data that are valid and defensible and which are provided in a timely manner for purposes of screening a site for volatile and semivolatile organic compounds (VOCs and SVOCs). This Quality Assurance Program Plan (QAPP) has been prepared in direct response to these goals. This plan describes the quality assurance program to be implemented and the quality control procedures to be followed for the analysis of passive soil-gas samples by gas chromatography/mass spectrometry (EPA Methods 8260C, 8270, TO-17 and TO-1). EPA Methods 8260C and 8270 are modified for the injection of the sample by thermal desorption.

Section No. 2.0 Revision No. 8 Date: May 25, 2010

Page 1 of 2

#### 2.0 ORGANIZATION AND RESPONSIBILITIES

#### 2.1 Structure

Beacon Environmental Services, Inc., (BEACON) is a privately owned company that provides services for the collection and analysis of passive soil gas samples collected in accordance with BEACON's Quality Assurance Program Plan for Passive Soil-Gas Sampling. BEACON's office is located at 323 Williams Street, Bel Air, MD 21014, which is approximately seven miles north of I-95 between Baltimore, MD and Philadelphia, PA.

## 2.2 Roles and Responsibilities

All laboratory personnel are involved with the Quality Assurance (QA) Program. The extent of their involvement depends on their assignment in the laboratory; however, all laboratory personnel are trained for their role in supporting the analyses of samples.

#### 2.2.1 President

The President of BEACON has overall responsibility for the operation of the laboratory. The specific responsibilities include:

- providing support and resources for the QA Program
- maintaining laboratory staffing
- coordinating training of personnel in all aspects of the laboratory
- approving equipment acquisition
- developing the laboratory budget
- maintaining and implementing the marketing program
- implementing the operational aspects of the QA Program
- ensuring the laboratory data quality as the Quality Assurance Officer
- reviewing data requirements for each project with Laboratory Director
- ensuring corrective actions specified by the Laboratory Director are implemented

## 2.2.2 Laboratory Director

The Laboratory Director reports to the President of BEACON. The Laboratory Director is primarily responsible for on-schedule completion of assigned laboratory work and for supervising all laboratory activities, including implementation of the Quality Assurance/Quality Control (QA/QC) program. The Laboratory Director enlists and encourages the cooperation of all the staff in the program. Specific responsibilities of the Laboratory Director include:

- ensuring that all analyses are performed according to the methods and protocols specified by the client
- reviewing all analytical data by (i) checking documentation for completeness and proper sample identification, (ii) checking raw data for calculation, interpretation, or clerical errors, (iii) assuring that produced quality control data are acceptable
- ensuring laboratory data quality

Section No. 2.0 Revision No. 8 Date: May 25, 2010

Page 2 of 2

- coordinating analytical work to ensure that all tasks are completed within established time frames
- overseeing preventative maintenance activities
- establishing analytical priorities and reviewing data requirements for each project
- reviewing initiated corrective actions and recommending additional measures, if necessary
- ensuring corrective actions are implemented
- reviewing quality control data to determine if test data are acceptable
- supervising the updating of accuracy, precision, and method detection limits
- performing periodical system audits to assure compliance with all quality assurance requirements
- evaluating and implementing changes in methodology and quality control measures
- identifying quality control problems and taking measures to correct or eliminate the problem source
- validating all data and assuring that data sets are accurate before reporting

# 2.2.3 Analysts

Analysts report to the Laboratory Director. They are responsible for on-schedule performance and documentation of all analyses assigned. Moreover, their responsibilities include:

- performing required analyses according to test methods specified
- assuring that all analytical equipment has been properly calibrated before beginning the analyses
- assuring that all identifying information (including sample control numbers, project numbers, and client information) have been accurately transcribed into records or computer data bases
- assuring that all calculations are correct
- assuring that appropriate confirmatory tests or procedures have been completed
- identifying, documenting, and beginning corrective actions on any quality control problem that relates to the analytical method
- maintaining equipment in working conditions and documenting all preventive maintenance and repairs

## 2.2.4 Operations Manager

The Operations Manager reports to the President of BEACON and is responsible for the administrative aspects of the laboratory. Specific responsibilities include:

- coordinating the preparation of passive soil-gas samplers and the shipment of kits
- receiving samples as primary sample custodian
- initiating paperwork for sample analyses on appropriate laboratory documents (including establishing project files and sample receipt records) as required for analysis
- obtaining, filing, and distributing pertinent project information to laboratory staff
- reporting laboratory results to the client
- understanding and following aspects of QA program related to job function
- managing BEACON central file system, which includes project statements of work or proposals, quality assurance plans, chain-of-custody records, and final data reports
- initiating and tracking archives for all laboratory documents

Section No. 3.0 Revision No. 8 Date: May 25, 2010

Page 1 of 2

# 3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

This section presents the QA objectives for the chemical data in terms of precision, accuracy, completeness, representativeness, and comparability.

#### 3.1 Precision

Precision is the mutual agreement among individual measurements of the same property and is a measure of the random error component of the data collection process. The overall precision of the data is the sum of that resulting from the sampling and analysis. The sampling precision is assessed by collecting field sample duplicates, when appropriate, and accompanying every sample batch with a trip blank. The analytical precision is determined by preparing and analyzing spiked replicate samples. Precision can be expressed in several different ways, each of which has its uses; for multiple measurements these include the standard deviation, the relative standard deviation, and the range.

## 3.2 Accuracy

Accuracy is the degree of agreement of a measured value with the true or expected value of the measured quantity. It is a measure of the bias or systematic error of the entire data collection process. Sources of these errors include the sampling process, field and laboratory contamination, sample handling, sample matrix, sample preparation methods, and calibration and analysis procedures. Sampling accuracy is assessed by evaluating the results of sample preparation blanks, ambient-air control samples, field sample location duplicates, and trip blanks. Analytical accuracy is assessed through the use of calibration verification samples, method blanks, laboratory control samples, and sample preparation blanks.

#### 3.3 Representativeness

Data representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representation is a quantitative parameter that is most concerned with the proper design of the sampling program. The sampling program has been designed so that the samples collected are as representative as possible of the medium being sampled and that a sufficient number of samples will be collected. Representativeness is addressed by the description of the sampling techniques and the rationale used to select the sampling locations.

# 3.4 Completeness

Completeness is defined as the percentage of measurements made that are judged to be valid data. To achieve this objective, every effort is made to avoid sample loss through accidents or inadvertence. Accidents during sample transport or lab activities that cause the loss of the original sample will result in irreparable loss of data. The assignment of a set of laboratory numbers to a batch of samples that have undergone chain-of-custody inspection makes it more difficult for the analyst to overlook samples when setting up a batch of samples for analysis. The laboratory numbers also make it easy during the data compilation stage to pick out the samples which have not been analyzed and to order their analysis before the data are reported and before holding times have been exceeded. The completeness of each batch of

Section No. 3.0 Revision No. 8 Date: May 25, 2010

Page 2 of 2

samples can be calculated by dividing the total number of analyses completed by the number that should have been performed on that batch times 100.

## 3.5 Comparability

Data comparability is a measure of the confidence with which one data set can be compared to another. It cannot be described in quantitative terms, but must be considered in designing the sampling plans, analytical methodology, quality control, and data reporting. The use of standard EPA-verified sampling techniques and validated, EPA-approved analytical methods assures that the parameters being measured are comparable with data generated from other sources with comparable equipment. Reporting of data in units used by other organizations also assures comparability.

# 3.6 Project QA Objectives

Unless otherwise specified, the accuracy objective is 70-130%, for analytical precision  $\pm 25\%$ , and completeness 99%. The accuracy and precision are based on the analysis of the standard spiking of a blank adsorbent sampler. The accuracy is the percent recovery of the target analytes, and the precision is the standard deviation of successive percent recoveries. The results of samples for which the recovery of the standards does not fall within these limits will be qualified as being outside the control limits. Beacon voluntarily participates in performance testing using blind samples to evaluate our method.

Section No. 4.0 Revision No. 8 Date: May 25, 2010

Page 1 of 3

#### 4.0 SAMPLE CUSTODY PROCEDURES

The laboratory chain-of-custody procedures will document sample possession from the time of shipment to the time of receipt and final analysis, in accordance with BEACON procedures and federal guidelines. The National Enforcement Investigations Center (NEIC) of U. S. EPA defines <u>custody of evidence</u> in the following ways (i) it is in your actual possession, (ii) it is in your view, after being in your physical possession, (iii) it was in your possession and then you locked or sealed it up to prevent tampering, and (iv) it is in a secure area.

# 4.1 Chain-Of-Custody Form

A chain-of-custody form serves as permanent documentation of sample validity in which is recorded all pertinent aspects of sample collection and handling prior to delivery to the laboratory. A chain-of-custody form is generated when project adsorbent samplers are shipped to the client. A completed chain-of-custody form contains the following information: (i) client name and project site, (ii) sample identification name/number, (iii) attendant chain-of-custody names, signatures, dates, and times (if applicable), including name of courier, (iv) analyses requested, (v) date and time of receipt, and (vi) special instructions. The laboratory has the final responsibility to ensure that all necessary documentation is properly recorded. Deviations from established protocols are recorded on the chain-of-custody form. The laboratory will make note of samples collected for analysis without a correctly prepared and relinquished chain-of-custody form and contact the client prior to initiating analysis.

# 4.2 Sample Receipt

The Operations Manager and those designated are authorized sample custodians. Each authorized employee shall be prepared to testify in a court of law as to the nature and extent of access to, or possession of, any sample in the custody of BEACON. The sample custodian performs the following tasks after receiving samples: (i) inspects sample shipping containers for presence/absence and condition of custody seals, samples, and field kit equipment, (ii) records condition of both shipping containers and sample containers on the chain-of-custody form, (iii) verifies and records agreement or non-agreement of information on sample documents on the chain-of-custody form, (iv) verifies the number of sample containers received is equal to the number of samples listed on the chain-of-custody form, (v) logs all samples into sample receipt logbook that records sample identifications, requested analytical method, project identification number, any discrepancies in packaging or labeling, date, and name of sample custodian (vi) signs chain-of-custody shipped with samples, (vii) communicates any problems or discrepancies to the Laboratory Director, (viii) places samples in a project specified bag and places the bag in the sample refrigerator, and (ix) places chain-of-custody and field deployment forms in client file.

# 4.3 Sample Security and Accessibility

The laboratory is maintained in a safe and secure manner at all times. The facility is locked when not occupied and is monitored for fire and unauthorized access. BEACON personnel escort all visitors at all times while inside the facility.

Section No. 4.0 Revision No. 8 Date: May 25, 2010

Page 2 of 3

When not actually employed in analysis, the samples are stored in a refrigerator. In the event of unsupervised intrusion, such as by police, firefighting personnel, or by burglary, such incident will be documented on the chain-of-custody form. Clients will be notified by phone and in writing.

# 4.4 Sample Retention and Disposal

There are no established holding times for soil-gas samples collected on adsorbent cartridges; however, because the medium is an adsorbent, it can be held for more than 28 days without any demonstrated loss, as verified through holding times studies. As standard practice to meet client turn-around time requirements, samples are analyzed typically within five (5) days of sample receipt. BEACON's sampler design allows for a secondary adsorbent cartridge to be included in each sampler. That secondary cartridge may be held for confirmatory or duplicate analysis according to contractual obligations. Following analysis of the passive soil-gas samples, adsorbent cartridges are reconditioned for use in a future project or disposed of if they no longer meet quality control standards.

## 4.5 Document Control

BEACON's goal of the document control program is to assure that all documents for the project are accounted for when the project is completed. Accountable documents include chain-of-custody records, sample receipt logbooks, instrument logbooks, field notes, and other documents that may relate to the collection of samples and sample analyses.

# 4.5.1 Recordkeeping

All data entries are made in indelible, water-resistant ink. The date of the entry and the observer is clear on each entry. The observer uses his or her full name or initials. All information is recorded in a notebook or on other records at the time the observations are made. Recording information on loose pieces of paper is not allowed. When a mistake is made, the wrong entry is crossed out with a single line, initialed and dated by the person making the entry, and the correct information is recorded. Obliterating or writing over an incorrect entry is not allowed, nor is the use of correction tape or fluid on any laboratory records. Each page in logbooks is sequentially numbered and shows the laboratory name.

# 4.5.2 Laboratory Records

Following are some of the records that are used to document activities in the laboratory. These are in addition to the documents discussed elsewhere is this OA manual.

## **Project File**

The project file is a folder that is established when the project is initiated. The project file is labeled with the project number, project name and location, and client name. The chain-of-custody forms, field notes, correspondence, instrument printouts, and a copy of the final report are placed in the project file.

Section No. 4.0 Revision No. 8 Date: May 25, 2010

Page 3 of 3

# **Instrument Run Log**

An instrument run log that records daily observations and operations is maintained for each major analytical instrument. The instrument log contains the following: (i) identification of all analyses made in sequence (including those analyses or runs that are not acceptable or project related), (ii) the autosampler sequence, if applicable, (iii) date, (iv) the project number, (v) analyst, (vi) maintenance notes, and (vii) a description of any corrective action taken.

# Refrigerator/Freezer Logs

The maintenance and documentation of the operating temperatures of refrigerators and freezers are recorded in logs for each piece of equipment. Proper storage of samples and standards in refrigerators and freezers is critical to maintenance of their integrity. Each page of the logs is numbered sequentially and provides descriptive information on the piece of equipment. The following information is recorded on a daily basis: (i) date, (ii) analyst, (iii) temperature, and (iv) any corrective action required.

# **Conditioning Oven Log**

The maintenance and documentation of the operating temperature and performance of the conditioning oven are recorded in the conditioning oven log. The temperature, gas flow, and gas leak checks are recorded in the log when adsorbent samplers are conditioned to assure that the adsorbent samplers are properly conditioned. Each page of the conditioning oven log is numbered sequentially and provides descriptive information on the oven. The following information is recorded in the log when adsorbent samplers are conditioned: (i) date and time when conditioning starts and ends, (ii) the project number, (iii) adsorbent material, (iv) oven set temperature, (v) gas flow rate, (vi) temperature check, (vii) gas leak and flow check, (viii) analyst, and (ix) any corrective or maintenance action required.

Section No. 5.0 Revision No. 8 Date: May 25, 2010

Page 1 of 2

## 5.0 CALIBRATION PROCEDURES AND FREQUENCY

# **5.1** Calibration Program

A formal calibration program controls instruments and equipment used by BEACON. The program verifies that equipment is of the proper type, range, accuracy, and precision to provide data compatible with specified requirements. All instruments and equipment that measure a quantity, or whose performance is expected at a stated level, are subject to calibration. Chemical calibration or standardization, which refers to those operations in which instrument response is related to analyte mass, is discussed in this section.

## 5.2 Chemical Calibration

Chemical calibrations consist of initial and continuing calibrations, which are discussed in subsequent sections. The calibration criteria are based on those given in EPA Method 8260C and as described in the Solid Waste Manual (SW-846), as well as U. S. EPA Method TO-1 and TO-17 as described in the Compendium for Methods for the Determination of Toxic Organic Compounds in Ambient Air (EPA/600/4/89/017). Chemical calibrations are documented and stored in computer files as well as on hard-copy printouts.

## 5.2.1 Initial Calibration

The initial multi-point calibration consists of the establishment of a calibration or standard curve, which associates instrument response and analyte mass or concentration. The curve is constructed by measuring the responses of a series of spiked adsorbent cartridges. Calibration factors (CFs) are calculated using the internal calibration technique. The percent relative standard deviation of the CFs must be below 30% for the curve to be assumed linear and valid. The percent relative standard deviation (RSD) is calculated by dividing the standard deviation for the CF by the mean of the CFs and multiplying by 100. The low-point calibration mass is below the reporting limit to ensure accuracy and eliminate reporting false negatives.

#### 5.2.2 Continuing Calibration

The initial calibration curve and tuning criteria is verified at the beginning of each analytical sequence for all GC/MS methods. An analytical sequence, including the initial calibration, continuing calibration, method blank, and sample analyses, will not exceed 24 hours. The criterion for the acceptance of the continuing calibration is based on the percent difference between the calculated mass injected and the value obtained by the initial calibration comparison. For GC/MS analysis, certain System Performance Check Compound (SPCC) and Calibration Check Compound (CCC) criteria must be met prior to sample analysis. The target compound value obtained during the continuing calibration must be within  $\pm 30\%$  of the initial calibration for the calibration to be verified valid. If the continuing calibration varies more than  $\pm 30\%$  then corrective action must be taken to restore the system and a new calibration verification run or a new calibration curve must be prepared before any more samples are run.

Section No. 5.0 Revision No. 8 Date: May 25, 2010

Page 2 of 2

### 5.2.3 GC/MS Tuning and Mass Calibration

When analyzing samples with GC/MS instrumentation, it is necessary to demonstrate that the GC/MS meets the standard mass spectral abundance criteria prior to data collection. The method specific criteria must be demonstrated prior to any standards, blanks, or samples being analyzed and for each 24-hour analytical sequence.

# 5.2.4 Analytical Standards

Analytical standards are purchased from ISO 9001 registered, and NIST-NVLAP accredited suppliers. All identifying paperwork accompanying standards purchased are kept in a standards log that include (i) name of standard, (ii) supplier of standard, (iii) date received, (iv) date of expiration, and (v) concentration of standard. All standards are stored at or below 4° Celsius in standard vial sealers. Information is listed on each vial identifying the standard, the concentration of the standard, and the expiration date. All dilutions of standards performed at BEACON to prepare specific concentrations are documented in the Standard Preparation Log, which records: (i) date of preparation, (ii) analyst, (iii) lot number, concentration, and supplier, (iv) volume and type of solvent, (v) concentration of final standard, and (vi) any necessary comments. The prepared standards are labeled with the date and concentration and stored at or below 4° Celsius in sealed amber glass vials.

Section No. 6.0 Revision No. 8 Date: May 25, 2010

Page 1 of 2

#### 6.0 ANALYTICAL PROCEDURES

# 6.1 Analytical Method

The analytical methods followed are based on U. S. EPA Methods 8260C, 8270 as described in the Solid Waste Manual (SW-846), as well as U. S. EPA Method TO-17, and TO-1 as described in the Compendium for Methods for the Determination of Toxic Organic Compounds in Ambient Air (EPA/625/R-96/010b). Methods 8260C and 8270 have been modified for thermal desorption of adsorbent cartridges to screen sites for the targeted compounds. A summary of each of the analytical methods with quality assurance procedures and acceptance criteria specific to each method is provided under separate cover.

Sample matrices are adsorbed vapors. All analytical methods used by BEACON have the same procedure for sample introduction, which is thermal desorption. The thermal desorber heats sample cartridges in a helium atmosphere to temperatures ranging from 200° to 350° C, the actual temperature dependent on the adsorbent used. To check for contamination in the system, a system or method blank is run at the beginning of an analytical batch, as well as after any high-level detection that results in potential carry over. If necessary, blanks are run until the system is clean and, if needed, the system is conditioned until the baseline of the respective detector is stable. A blank is run following the conditioning to check for cleanliness of the system. A blank is run after the daily continuing calibration.

If the sample response of any target compound falls outside the calibration range, the concentration of the compound will be estimated and reported as such. However, the design of the passive soil-gas sampler with two adsorbent cartridges enables the analyst to perform a second analysis from each location where the analytical acceptance criteria does not initially pass.

## 6.2 Instrumentation

The analytical methods are performed with gas chromatographs connected to mass spectrometers (GC/MS). All GC/MS analytical systems are equipped with thermal desorption sample introductory systems for analysis. The analytical instruments employed for each method are described in the method specific procedures, which are provided under separate cover.

#### **6.3** Detection Limits

Method detection limits (MDL) are determined using the U.S. EPA procedure published in 40 CFR Part 136, Appendix B. The MDL is defined as "the minimum concentration of a substance 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." This procedure requires that "all sample processing steps of the analytical method be included in the determination of the method detection limit." MDLs therefore are influenced by the sample matrix and sample preparation process as well as the analytical instrumentation.

Section No. 6.0 Revision No. 8 Date: May 25, 2010

Page 2 of 2

A minimum of seven replicates spiked at one to five times the expected MDL are analyzed. The MDL is calculated by multiplying the standard deviation for n replicates by the one-tailed Student's t-value for n-1 degrees of freedom and a 99% confidence level. On average the MDL for target individual analytes is less than five nanograms per adsorbent sampler. Because the definition of an MDL is concerned only with detection of an analyte and not its accurate quantitation, MDLs are not used in reporting data. Instead, data are reported using reporting limits, which are levels above that which reliable quantitative results can be obtained. The reporting limit used for the method analytes is 25 nanograms per adsorbent sampler. MDL studies are performed periodically to ensure that the values are at least five times less than the reporting limits.

When the measured mass of an analyte is below its reporting limit but the instruments response meets the identification criteria for the method, the measured mass is reported with a "J" (estimated) flag. If the analyte is not detected or the identification criteria are not met, the reporting limit is shown (e.g., <25).

Section No. 7.0 Revision No. 8 Date: May 25, 2010

Page 1 of 2

## 7.0 DATA REDUCTION, VALIDATION, AND REPORTING

The procedures for data handling followed in the laboratory are an important part of the laboratory quality assurance program. BEACON treats all records and project data as client confidential. Client information will not be shown to anyone outside BEACON without the client's approval.

## 7.1 Data Collection

For all analyses, the raw data are collected by the associated computer system and software and sorted in data files. The raw data are handled such that manual transcriptions are avoided to as great an extent as possible. The standards chosen for the methods contain the analytes to be targeted on the adsorbent cartridges. After the analyst has fully quantified the data, it is electronically transcribed to a PC-based database for further processing.

## 7.2 Data Reduction

Data reduction includes all processes that change either the reporting of values or numbers of data items. The data reduction processes used in the laboratory include establishment of calibration curves, calculation of sample concentrations from instrument responses, and computation of quality control parameters. The masses of analytes present on the adsorbent cartridges are determined, using the calibration function (CF) from the continuing calibration verification (Section 5.2.2), with the analytical system specific software program.

#### 7.3 Data Validation

Data validation is a systematic process of reviewing data against a set of criteria to identify outliers or errors and to delete suspect values or to flag them for the user. Laboratory data review starts with the analyst and the laboratory quality control procedures discussed in Section 9.0. The analyst reviews the quality control data as the data are generated against the method specific criteria and takes the specified corrective action when the data are out of control. After analyses and data workup are complete, the analyst checks the data for errors in transcriptions and calculations.

The Laboratory Director is responsible for final validation of the data. Validation starts with verifying that the required quality control procedures were effectively in place and followed by the analyst, and that the generated documentation shows that samples, data, and analytical results were acquired and processed in a controlled and traceable manner.

The following aspects affecting data quality are checked in manual and computerized fashion before data entry and issuance of the final report: (i) the initial and continuing calibration verifications must meet method specific criteria and (ii) the method blanks must be devoid of all targeted compounds at quantities greater than the reporting limits. Any sample sets or data that are judged unacceptable are identified and the appropriate corrective action measures initiated. Data qualifiers are assigned to all applicable data.

Section No. 7.0 Revision No. 8 Date: May 25, 2010

Page 2 of 2

Data validation also encompasses a review of (i) proper chain of custody sample login and sample handling procedures, (ii) holding times, (iii) efficiency of the preparation of adsorbent samplers prior to shipment (evaluated by analysis of a preparation blank), (iv) the method used to analyze samples, (v) internal standard areas and surrogate standard recoveries, (vi) blank system checks, (vii) calculations to verify and ensure computations performed correctly, (viii) transcription of raw and final data, and (ix) detection and reporting limits.

# 7.4 Reporting

Tabular laboratory report forms are generated from the data stored in the computer database and are placed in the project file. All sample identifications, calculations, and final report contents shall be manually reviewed prior to issuance of the final report. Project specific quality control objectives determine the reporting format, which can include the following: (i) narrative, (ii) tabular results of sample analysis, (iii) tabular results of method blank analysis, (iv) tabular results of continuing calibration verifications, (v) chromatograms of samples, method blanks, and calibration verifications, and (vi) raw quantitation data. CLP-equivalent data packages are available, upon request, including Forms 1, 2, 4, 5, 6, 7, and 8.

# 7.5 Data Storage

The project files containing all client data are placed in the central files numerically according to the project number. All project files are maintained securely within BEACON's corporate office, which has a monitored alarm system and is locked at all times when BEACON personnel are not present. Unless superseded by program, project, or client specific requirements, the disposal date of the archived files is five years from the archive date. Unless otherwise specified by the client, electronic data are maintained by BEACON for a period of two years. This guidance is consistent with the Good Laboratory Practices in 21 CFR Part 58 and Good Manufacturing Practices in 21 CFR Part 820. The Laboratory Director is responsible for ensuring that all electronic data are stored to prevent deterioration and that records are maintained identifying the storage drive, archive date, and discard date.

Section No. 8.0 Revision No. 8 Date: May 25, 2010

Page 1 of 2

# 8.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

This section describes the quality control procedures that will be followed during sample analysis, including analysis of quality control samples. The quality control requirements with frequencies and acceptance criteria are as follows: (i) a multi-point calibration is performed initially, and as required, and is accepted if the percent relative standard deviation (RSD) for the CFs over that range is less than 30%, (ii) calibration verification is performed according to method specific criteria (which are provided under separate cover), and (iii) method blank checks are performed at the beginning of sample batch analysis, plus after analysis of a high level sample, and are repeated until the system is confirmed clean. The results of an analysis that fails a QC criterion will be reported and flagged to indicate the problem.

#### 8.1 Calibration

The criteria and frequencies for initial and continuing calibrations, as well as system tunes, are discussed in Section 5.0.

#### 8.2 Blanks

A method blank is performed by analyzing a conditioned adsorbent cartridge by thermal desorption. This checks for system cleanliness. One or more trip blanks, which are cartridges prepared, transported, and analyzed with field samples but intentionally not exposed, are also analyzed with each project.

# 8.3 Internal Standards (IS)

Internal standards are organic compounds that are similar to the analytes of interest in chemical composition, extraction, and chromatography, but are not normally found in environmental samples and are used for internal calibration. For analyses following EPA Method 8260C, quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve. Internal standards are spiked onto all standards, blanks, and samples. If retention time of an IS changes by more than 30 seconds or the response of an IS changes by a factor of two (-50% to +100%) from that in the daily continuing calibration check, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When used as a diagnostic tool to monitor retention times and responses (area counts) in all samples, spikes, blanks, and standards, internal standards effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance. Sample results are flagged when the IS retention time and/or response do not fall within the method specific control limits. When IS changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration, method variables includings flow and temperatures, and system cleanliness are checked and retuning and recalibration and detector cleaning are performed if needed. For analyses following TO-17, the IS must fall within ±40% to pass acceptance criteria.

# 8.4 Surrogates

Surrogates are organic compounds that are similar to the analytes of interest in chemical composition, extraction, and chromatography, but are not normally found in environmental samples. For analysis of samples by GC/MS methods, surrogate compounds are spiked onto all standards, blanks, and samples in

Section No. 8.0 Revision No. 8 Date: May 25, 2010

Page 2 of 2

order to monitor the analysis of the samples. The mass of each surrogate is quantitatively determined in each analytical run and percent recoveries are calculated. Surrogate recoveries are monitored for analysis performance. For example, in the case of high petroleum contamination on samples the bromofluorobenzene recoveries normally exceed recoveries requiring that sample to be closely analyzed for false negatives and false positives as well as ion masking.

## 8.4 Duplicates

Duplicates are a pair of subsamples of a field sample that are taken through the entire preparation and analysis process to estimate the precision of the method. Because only one analysis of each adsorbent cartridge is possible, sample duplicates cannot be analyzed. However, two adsorbent cartridges are contained within each sampler; therefore, two samples can be analyzed from each location as a sample location duplicate.

# **8.5** General Laboratory Controls

In addition to instrument calibration and the analysis of quality control samples, the following controls will be implemented: (i) reagents and solvents will be certified (ii) reagent storage environment and duration will meet EPA guidelines, (iii) regular laboratory screening will be performed to assure a clean air environment, (iv) volumetric measurements will be made with certified glassware and recommended syringes, (v) data reduction computations will be independently checked, and (vi) only fully trained personnel will perform laboratory analyses.

Section No. 9.0 Revision No. 8 Date: May 25, 2010

Page 1 of 1

# 9.0 PERFORMANCE AND SYSTEM AUDITS

The Laboratory Director maintains and summarizes the performance of the systems at BEACON. Continual calibration and MDL studies are ongoing. Each laboratory procedure affecting data quality and validity will be reviewed periodically to assure performance. Performance evaluation (PE) samples are not available for the analysis of soil-gas samples passively collected on an adsorbent media.

Section No. 10.0 Revision No. 8 Date: May 25, 2010

Page 1 of 1

# 10.0 PREVENTATIVE MAINTENANCE PROCEDURES

Periodic preventative maintenance is required for all sensitive equipment. Instrument manuals will be kept on file for reference if equipment needs repair. The troubleshooting section of factory manuals may be used in assisting personnel in performing maintenance tasks. The individual responsible for the instrument documents maintenance in the laboratory analytical log.

Replacement parts are kept on hand to minimize down time. Table 10.1 lists specific maintenance procedures followed to ensure the consistent performance of the instruments and equipment.

**Table 10.1 Preventive Maintenance Procedures** 

Equipment	Action	Frequency
Gas Chromatograph	Flows and pressures checked	Prior to and during analysis
	Unions checked for leaks	Prior to and during analysis
	Gas line filters replaced	Color indicating
	Detectors cleaned	As needed
	Relays replaced	As needed
Markes UltrA-Unity Thermal	Software checks for leaks	Prior to each analysis
Desorber	Replace worn O-ring	After leaks are determined by software
	Set the trap flow	As the analytical method requires
	Check for trap contamination	Each analysis
	Replace trap	When needed
	Replace purge gas	As needed
Gas Chromatograph/Mass	Tune MSD	As needed
Spectrometer	Check the calibration vial	Every six months
	Check the foreline pump oil	Weekly or when discolored
	Replace the foreline pump oil	Every six months or as needed
	Clean the ion source	When performance deteriorates
	Check for leaks	Prior to analysis
Conditioning Oven	Flows and pressures checked	Prior to and during conditioning
-	Unions checked for leaks	Prior to and during conditioning
Refrigerators	Temperatures checked and logged	Each work day
	Defrosted and cleaned	Quarterly
Analytical Balances	Balanced checked	Prior to use
•	Zero checked	Prior to use
	Deflection checked	Prior to use
	100 mg calibration mass checked	Prior to use
	If any checks fail, balance	
	maintenance performed	

Page 1 of 3

# 11.0 STATISTICAL ASSESSMENT OF DATA QUALITY

The statistical tests necessary to verify proper analytical function are performed as soon as practical after the measurements on which they are based are available. The results of the tests are compared with the control limits to determine if the data can be used. If the limits are exceeded, the Laboratory Director is notified and a decision is made concerning the appropriate action to be taken.

#### 11.1 Calculation of Precision

Precision is the mutual agreement among individual measurements of the same property, usually under similar conditions. Precision can be expressed in several different ways, such as standard deviation, relative standard deviation, relative percent difference, and range.

#### **Standard Deviation**

The standard deviation measures the dispersion of replicate values about their mean.

$$s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \overline{x})^2}{n-1}}$$

where:

s = standard deviation

 $x_i$  = replicate values

 $\overline{x}$  = mean of replicate values n = number of replicate runs

## **Relative Standard Deviation**

$$RSD = \frac{s}{\overline{x}}$$

The relative standard deviation (RSD) is used for replicate measurements and is the ratio of the standard deviation of the measurements to their mean.

where:

s = standard deviation of replicate run values

 $\overline{x}$  = mean of the replicate run values

Page 2 of 3

#### **Relative Percent Difference**

The relative percent difference (RPD) is used for duplicate measurements and is calculated by dividing the difference between the values by their mean and multiplying by 100.

$$RPD = \frac{|x_1 - x_2|}{(x_1 + x_2)} \times 100$$

where  $x_1$  and  $x_2$  are the duplicate/replicate values

#### **Percent Difference**

The percent difference (%D) is a measure of the difference between a reference value and a measured one.

$$\%D = \frac{|r - x|}{r} \times 100$$

where:

r = reference valuex = measured value

## 11.2 Calculation Of Accuracy

Accuracy is the degree of agreement of a measured value with the true or expected value or the measured quantity. The accuracy of control sample measurements is generally expressed as a percent recovery. For samples without a background level of the analyte, such as reference materials, laboratory control samples, and performance evaluation samples, the percent recovery (%R) is calculated from:

$$\%R = \frac{X}{T} \times 100$$

where:

X =the found concentration

T = the true or assumed concentration.

Section No. 11.0 Revision No. 8 Date: May 25, 2010

Page 3 of 3

The percent recovery for measurements in which a known amount of analyte (a spike) is added to an environmental sample (matrix spike) is calculated from:

$$\%R = \frac{X - B}{T} \times 100$$

where:

X = the found concentration

B = the background concentration T = the true or assumed concentration.

Section No. 12.0 Revision No. 8 Date: May 25, 2010

Page 1 of 1

#### 12.0 NONCOMFORMANCE AND CORRECTIVE ACTION PROCEDURES

Action taken to improve improper performance of instruments and analytical systems must be scrupulously documented. In addition, care must be taken that appropriate personnel are alerted to conditions that may affect data quality.

## 12.1 Analytical Systems

Any corrective action taken to adjust or maintain the instrument will be recorded in the laboratory analytical log as appropriate. The following actions require performing an initial calibration: (i) detector cleaning or replacement, (ii) chromatographic column replacement, (iii) changing any pressure regulator or temperature regulator setting, and (iv) failure of continuing calibration verifications to meet criteria. The Laboratory Director and any other analyst will be informed when initial calibration is required. Any unusual difficulty encountered in calibration will be brought to the attention of each analyst, the Laboratory Director, and the President.

# 12.2 Samples, Sample Receipt, and Chain-of-Custody Documents

Any discrepancies noted in sample container labeling or sample chain-of-custody documents are reported to the client immediately, and noted on both the chain-of-custody document and sample receipt log.

# 12.3 General Laboratory Equipment

The monitoring of individual laboratory units and the recording of observations in the log reserved for the individual unit will often indicate actual or impending malfunction. Observations of possible malfunction will be immediately reported to the Laboratory Director or his designated substitute. Equipment found to be nonfunctional shall be conspicuously labeled as such. Repair and/or replacement action taken will be documented in the log/file designated for that unit.

## 12.4 Laboratory Reports and Documentation

In the event that an error or errors are found in previously transcribed data, the Laboratory Director will be notified immediately. In the case of previously transmitted reports, any errors found will be immediately communicated to the client. All corrected reports will be conspicuously labeled as "AMENDED" and include the signature of the authorizing party and date.

## 12.5 Performance Sample Evaluations

The results of any performance sample evaluations are made immediately available to each participating analyst and supervisor. Any serious deviation from expected or true values constitutes cause for immediate corrective action.

Section No. 13.0 Revision No. 8 Date: May 25, 2010

Page 1 of 1

# 13.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Fundamental to the success of this QAPP is the active participation of management in the project. Because of the small size of BEACON, the management is constantly aware of project activities and actively participates in development, review, and operation of the project. No formal reports are anticipated.