

APPENDIX G

QUALITY ASSURANCE PROJECT PLAN

QUALITY ASSURANCE PROJECT PLAN

BARKER CHEMICAL SITE SITE NO. 932119 SOMERSET, NEW YORK

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1.0 Introduction

This Quality Assurance Project Plan (QAPP) was prepared for the Barker Chemical Site in Somerset, New York. This QAPP presents the policies, organization, objectives, functional activities, and specific quality assurance (QA) and quality control (QC) activities that will be implemented during site management. This QAPP is designed to ensure that all technical data generated is accurate and representative.

All QA/QC procedures are to be implemented in accordance with applicable professional technical standards, NYSDEC and the United States Environmental Protection Agency (USEPA) requirements, government regulations and guidelines, and specific project goals and requirements. This QAPP is prepared in accordance with all NYSDEC and USEPA QAPP guidance documents. This QAPP incorporates the following activities:

- Sample Management and Chain of Custody;
- Document Control;
- Laboratory Quality Control; and
- Review of Project Deliverables.

Analytical samples will be collected in the field utilizing standard operating procedures (SOPs; see **Appendix F** – Field Sampling Plan) and sent to a New York State Department of Health (NYSDOH) Environmental Laboratory Accreditation Program (ELAP) certified laboratory for analysis. Field data compilation, tabulation, and analysis will be checked for accuracy. Calculations and other post-field tasks will be reviewed by field personnel and the project manager.

Equipment used to take field measurements will be maintained and calibrated in accordance with manufacturer's guidelines and recommendations. Records of calibration and maintenance will be kept by assigned personnel. Field testing and data acquisition will be performed in standard fashion following strict guidelines.

Document control procedures will be used to coordinate the distribution, coding, storage, retrieval, and review of all data collected during all sampling tasks. These include, but are not limited to, the sampling of surface water and groundwater.

In addition, the laboratory has developed SOPs for individual analytical methods and internal QC procedures. These documents are an important aspect of their QA program and are available for review upon request.

2.0 Data Quality Objectives

2.1 Background

Data quality objectives (DQOs) are qualitative and quantitative statements, which specify the quality of data required to support site management activities at the site. DQOs focus on the identification of the end use of the data to be collected. The project DQOs are achieved by utilizing a definitive data category as outlined in Guidance for the Data Quality Objectives Process, EPA QA/G-4 (September 1994). All sample analyses will provide definitive data, which are generated using rigorous analytical methods such as reference methods approved by the United States Environmental Protection Agency (USEPA).

The purpose of site management at the Barker Chemical Site is to continually monitor remaining contamination in soil and fill at the site by monitoring its impact on groundwater and surface water.

2.2 QA Objectives for Chemical Data Measurement

Sample analytical methodology for the media sampled and data deliverables are required to adhere to the requirements in NYSDEC Analytical Services Protocol. Laboratories are instructed to complete Sample Preparation and Analysis Summary forms and submit with the data packages. The laboratory is instructed that matrix interferences must be fixed to the extent practicable. To achieve the definitive data category described above, the data quality indicators of precision, accuracy, representativeness, comparability, and completeness are measured during analysis.

2.2.1 Precision

Precision examines the distribution of the reported values about their mean. The distribution of reported values refers to how different the individual reported values are from the average reported value. Precision may be affected by the natural variation of the matrix or contamination within that matrix and by errors made in field or laboratory handling procedures. Precision is evaluated using analyses of a laboratory matrix spike/matrix spike

duplicate (for organics) and matrix duplicates (for inorganics), which indicate analytical precision through the reproducibility of the analytical results. Relative Percent Difference (RPD) is used to evaluate precision and it must meet the method requirements.

2.2.2 Accuracy

Accuracy measures the analytical bias in a measurement system. Sources of error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analysis techniques. These data help to assess the potential concentration contribution from various outside sources. The laboratory objective for accuracy is to equal or exceed the accuracy demonstrated for the applied analytical methods on samples of the same matrix. The percent recovery criterion is used to estimate accuracy based on recovery in the matrix spike/matrix spike duplicate and matrix spike blank samples. The spike and spike duplicate, which will give an indication of matrix effects that may be affecting target compounds is also a good gauge of method efficiency.

2.2.3 Representativeness

Representativeness expresses the degree to which the sample data accurately and precisely represent the characteristics of a population of samples, parameter variations at a sampling point, or environmental conditions. Representativeness is a qualitative parameter, which is most concerned with the proper design of the sampling program or sub-sampling of a given sample. Objectives for representativeness are defined for sampling and analysis tasks and are a function of the investigative objectives. The sampling procedures described in the Field Sampling Plan (**Appendix F** of this SMP) have been selected with the goal of obtaining representative samples for the media of concern.

2.2.4 Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. A DQO for this program is to produce data with the greatest possible degree of comparability. This goal is achieved through using standard techniques to collect and analyze representative samples and reporting analytical results in appropriate units. Complete field documentation will support the assessment of comparability. Comparability is limited by the other parameters (e.g., precision, accuracy, representativeness, completeness, comparability), because only when precision and accuracy are known can data sets be compared with confidence. For data sets to be comparable, it is

imperative that contract-required methods and procedures be explicitly followed.

2.2.5 Completeness

Completeness is defined as a measure of the amount of valid data obtainable from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is important that appropriate QA procedures be maintained to verify that valid data are obtained to meet project needs. For the data generated, a goal of 90% is required for completeness (or usability) of the analytical data. If this goal is not met, then project personnel will determine whether the deviations might cause the data to be rejected.

3.0 Project Organization and Responsibility

Sampling will be completed by experienced environmental professionals.

QA contacts include other technical professionals under contract to the NYSDEC or the owner of the property. A NYSDOH ELAP or USEPA CLP certified laboratory will provide analytical services for project work.

If necessary, data validation and QA staff will include data validation chemists, QA auditors, and other technical specialists who remain independent of the laboratory and project management. The staff will independently validate analytical data to assess and summarize their accuracy, precision, and reliability and determine their usability.

4.0 Sampling Procedures

4.1 Sampling Design

Sampling procedures for surface water and groundwater were designed to further monitor remaining contamination at the site. These procedures are described in **Appendix F** – Field Sampling Plan.

Surface water and groundwater samples will be analyzed for:

- Target Compound List (TCL) volatile organic compounds (VOCs) via USEPA Method 8260C or newer.
- TCL semi-volatile organic compounds (SVOCs) via USEPA Method 8270D or newer.

- TCL pesticides via USEPA Method 8081B or newer.
- Target Analyte List (TAL) metals via USEPA Method 6010C or newer.
- Mercury via USEPA Method 7470A or newer.
- Sulfide via USEPA Method 4500D or newer.
- Sulfate via USEPA Method 300 or newer.

Detection and reporting limits for all analytes will be below applicable Standards, Criteria and Guidance values.

The locations of the micro-wells to be sampled are shown on **Figure 4-1** of the SMP, while the surface water sample locations are shown on **Figure 4-2**.

4.2 QC Samples

Various types of field QC samples are used to check the cleanliness and effectiveness of field handling methods. They are analyzed in the laboratory as samples, and their purpose is to assess the sampling and transport procedures as possible sources of sample contamination and document overall sampling and analytical precision. Rigorous documentation of all field QC samples in the site notebook is mandatory.

- **Trip Blanks** are similar to field blanks with the exception that they are not exposed to field conditions. Their analytical results give the overall level of contamination from everything except ambient field conditions. Trip blanks are prepared at the lab prior to the sampling event and shipped with the sample bottles. Trip blanks are prepared by adding organic-free water to a 40-ml volatile organic analysis (VOA) vial. One trip blank will be used with every batch of water samples shipped for volatile organic analysis. Each trip blank will be transported to the sampling location, handled like a sample, and returned to the laboratory for analysis without being opened in the field.
- **Field Equipment/Rinsate Blanks** are blank samples designed to demonstrate that sampling equipment has been properly prepared and cleaned before field use and that cleaning procedures between samples are sufficient to minimize cross-contamination. Rinsate blanks are prepared by passing analyte-free water over sampling equipment and analyzing the samples for all applicable parameters. If a sampling team is familiar with a particular site, its members

may be able to predict which areas or samples are likely to have the highest concentration of contaminants. Unless other constraints apply, these samples should be taken last to avoid excessive contamination of sampling equipment. Rinsate blanks are not required if dedicated sampling equipment is used for sample collection.

- **Field Duplicates** consist of a set of two (2) samples collected independently at a sampling location during a single sampling event. Field duplicates are sent to the laboratory so that they are indistinguishable from other analytical samples and personnel performing the analysis are not able to determine which samples the field duplicates are from. Field duplicates are designed to assess the consistency of the overall sampling and analytical system.
- **Matrix Spike Samples and Spike Duplicate (MS/MSD)** are representative but randomly chosen client samples that are spiked with known concentrations of specific compounds as stipulated by the methodology prior to sample preparation and analysis. The matrix spike (MS) and matrix spike duplicate (MSD) are subjected to the entire analytical procedure to assess both accuracy and precision of the method for the matrix by measuring the percent recovery and relative percent difference of the two spiked samples. The samples are used to assess matrix interference effects on the method, as well as to evaluate instrument performance. MS/MSDs are analyzed at a frequency of one each per 20 samples per matrix.

Field QC samples and the frequency of analysis for this project are summarized in **Table 1** of this QAPP.

4.3 Sample Types and Methods

Sample types and methods are described in **Appendix F** - Field Sampling Plan of this SMP. Please refer to that appendix for details.

4.4 Sample Documentation

Sample documentation is described in **Appendix F** - Field Sampling Plan of this SMP. Please refer to that appendix for details.

4.5 Field Instrumentation

All instruments and equipment used during sampling and analysis will be operated, calibrated, and maintained according to manufacturer's guidelines and recommendations. Operation, calibration, and maintenance will be performed by personnel properly trained in these procedures. Documentation of calibration information will be maintained in the appropriate notebook and will be available upon request.

5.0 Sample Handling and Chain of Custody

This section describes procedures for sample handling and chain of custody to be followed by sampling personnel and the analytical laboratory. The purpose of these procedures is to ensure that the integrity of the samples is maintained during their collection, transportation, storage, and analysis. All chain of custody requirements comply with USEPA sample-handling protocols.

All samples will be obtained, handled, and characterized in accordance with NYSDEC ASP methods. Samples will be relinquished to a NYSDEC ELAP or USEPA CLP certified analytical laboratory. All chain of custody requirements will be strictly adhered to for designated analyses.

Sample identification documents will be carefully prepared so that sample identification and chain of custody can be maintained, and sample disposition controlled. Sample identification documents include field notebooks, sample labels, custody seals, chain of custody records, and laboratory sample log-in and tracking forms.

The primary objective of the chain of custody procedures is to provide an accurate written record that can be used to trace the possession and handling of a sample from the moment of its collection through its analyses. A sample is in custody if it is:

- In someone's physical possession;
- In someone's view;
- Locked up; or
- Kept in a secured area that is restricted to authorized personnel.

5.1 Sample Containers and Preservation

Samples must be collected in laboratory certified analyte-free sample bottles containing preservatives as required. A list of preservatives and holding times for each type of analysis is included in **Table 2** of this QAPP. These bottles will be provided by the analytical laboratory. All containers provided by the laboratory must be pre-cleaned (Level 1), with certificates of analysis available for each bottle type. Certificates of analysis provided by the vendor are kept on file by the laboratory.

All samples will be stored on ice (or blue ice) pending delivery to the laboratory.

Sample preservation will be verified at the lab just prior to extraction, digestion, and/or analysis and the pH will be recorded in the extraction/digestion logbook. The pH may be checked upon arrival, if desired. If the samples are improperly preserved, a QA/QC discrepancy form will be submitted to the lab manager and QA coordinator for appropriate follow-up action (i.e., evaluation of the data during the data validation process and, if necessary, additional instruction of personnel regarding proper procedures).

5.2 Field Custody Procedures

- Laboratory certified analyte-free sample bottles containing preservatives as required must be obtained from the laboratory. All containers will be prepared in a manner consistent with the NYSDEC ASP bottle-washing procedures. Coolers or boxes containing cleaned bottles should be sealed with a custody tape seal during transport to the field or while in storage prior to use.
- All containers will have assigned lot numbers to ensure traceability through the supplier.
- As few persons as possible should handle samples.
- The sample collector is personally responsible for the care and custody of samples collected until the samples are transferred to another person or the laboratory under proper chain of custody procedures.
- The sample collector will record sample data in the field notebook.
- The project manager will determine whether proper custody procedures were followed during the fieldwork and decide if additional samples are required.

5.2.1 Custody Seals

Custody seals are preprinted adhesive-backed seals with security slots designed to break if the seals are disturbed. A custody seal is placed over the cap of individual sample bottles by the sample collector. Sample shipping containers (i.e., coolers) are sealed in as many places as necessary to ensure security. Seals must be signed and dated before use. Strapping tape should be placed around the lid to ensure that seals are not accidentally broken during shipment and in a manner that allows easy removal by laboratory personnel. On receipt at the laboratory, the custodian must check and certify (by completing logbook entries) that seals on the coolers and bottles are intact.

5.2.2 Chain-of-Custody

The chain of custody (COC) must be fully completed by the field technician who has been designated by the project manager as responsible for sample shipment to the appropriate laboratory for analysis. In addition, if samples are known to require rapid turnaround by the laboratory, or if extremely high concentrations are suspected in a sample, the person completing the chain of custody should note these constraints in the “Remarks” section of the form. More detail regarding chain of custody procedures can be found in **Appendix F** - Field Sampling Plan of this SMP.

5.3 Sample Handling, Packaging and Shipping

The transportation and handling of samples must be completed in a manner that not only protects the integrity of the sample but also prevents any detrimental effects due to the possible hazardous nature of the samples. Regulations for packaging, marking, labeling, and shipping hazardous materials are promulgated by the United States Department of Transportation (DOT) in the Code of Federal Regulations, 49 CFR 171 through 177.

5.3.1 Sample Packaging

If samples need to be shipped to the laboratory, they must be packaged carefully to avoid breakage or contamination and must be shipped to the laboratory at proper temperatures. The following sample packaging requirements will be followed:

- Sample bottle lids must never be mixed.
- The sample bottle should never be completely filled except for VOA bottles. At a minimum, a 10% void space should be left in the bottle to allow for

expansion.

- Foam inserts should be used as inert packing material when shipping low hazard water samples via a common carrier to the laboratory.
- Low-hazard environmental samples are to be cooled. “Blue ice” or some other artificializing material, or ice placed in plastic bags, may be used. Ice will not be used as a substitute for packing material.
- A duplicate custody record must be placed in a plastic bag and taped to the inside of the cooler lid. Custody seals are affixed to the sample cooler.
- The cooler will be labeled as containing a hazardous material if it contains medium or high-hazard samples. Labeling requirements differ depending on the type of material being shipped; the majority of water samples may be shipped as a class “9” hazardous material with the proper shipping name “OTHER REGULATED SUBSTANCES (ENVIRONMENTAL SAMPLES).”
- A hazardous material shipping manifest will be completed for each cooler of medium to high-hazard samples and affixed to the lid of the cooler.
- Low-hazard environmental samples do not require a hazardous material shipping manifest. The words “LABORATORY SAMPLES” should be printed on the top of the cooler for low-hazard samples.
- Samples packaged and shipped as limited-quantity radioactive material must comply with NYSDOT and shipper regulations for package contamination limits, surface exposure rate, and air bill completion.

5.3.2 Shipping Containers

Environmental samples will be properly packaged and labeled for transport to the subcontracted laboratory for analysis. A separate chain of custody record must be prepared for each container. The following requirements for marking and labeling of shipping containers will be observed:

- Use abbreviations only where specified.
- The words “This End Up” or “This Side Up” must be clearly printed on the top of the outer package. Upward-pointing arrows should be placed on the sides of the package. The words “Laboratory Samples” should also be printed on the top of the package.

- After a container has been closed, two custody seals are placed on the container: one on the front and one on the back. The seals are protected from accidental damage by placing packaging tape over them.
- Field personnel will make timely arrangements for transportation of samples to the laboratory. When custody is relinquished to a shipper, field personnel will telephone the laboratory custodian to inform him of the expected time of arrival of the sample shipment and to advise him of any time constraints on sample analysis.

5.3.3 Shipping Procedures

- The shipping containers in which the samples are packed must be accompanied by a chain of custody. When transferring samples, the individuals relinquishing and receiving them must sign, date, and note the time on the COC to document sample custody transfer.
- Samples must be transported to the laboratory for analysis with a separate chain- of-custody accompanying each shipment. Shipping containers must be sealed with custody seals. The method of shipment, name of courier, and other pertinent information are entered in the “Remarks” section of the chain of custody record.
- All shipments must be accompanied by the chain of custody record identifying their contents. The original record accompanies the shipment, and the yellow copy is retained by the field person shipping the samples.
- If sent by mail, the package is registered with return receipt requested. If sent by common carrier, a bill of lading is used. Freight bills, Postal Service receipts, and bills of lading are retained as part of the permanent documentation.
- Samples must be shipped to the analytical laboratory within 24 hours after collection.

5.4 Laboratory Custody Procedures

The designated sample custodian at the laboratory will be responsible for maintaining the chain of custody for samples received at the lab. Among other things, the custodian must adhere to the following basic requirements:

- When the samples arrive at the lab, the custodian will complete a Cooler Receipt & Preservation Form for each cooler/shipping container.
- Upon receipt, the coolers are examined for the presence and condition of custody seals, shipping papers, etc. Shipping labels are removed and placed on scrap paper and added to the receiving paperwork. The custodian then completes the chain of custody record by signing and recording the date and time the package is opened.
- Acceptance criteria for cooler temperature is 0-4°C. If a cooler exhibits a temperature outside this range, the anomalies are noted on the Cooler Receipt & Preservation Form.
- The custodian will then unload the samples from the cooler(s)/container(s), assign an identification number to each sample bottle, and affix a barcode label to each sample bottle for logging in and out of the Laboratory Information Management System (LIMS).

Adherence to this procedure will ensure that all samples can be referenced in the computer tracking system. All sample control and chain of custody procedures applicable to the analytical laboratory are presented in laboratory SOPs available for review.

6.0 Calibration Procedures and Frequency

To obtain a high level of precision and accuracy during sample processing procedures, laboratory instruments must be calibrated properly. Several analytical support areas must be considered so the integrity of standards and reagents is upheld prior to instrument calibration. The following sections describe the analytical support areas and laboratory instrument calibration procedures.

6.1 Analytical Support Areas

Prior to generating quality data, several analytical support areas must be considered; these are detailed in the following paragraphs.

6.1.1 *Standard/Reagent Preparation*

Primary reference standards and secondary standard solutions shall be obtained from the National Institute of Standards and Technology (NIST), or other reliable commercial

sources to verify the highest purity possible. The preparation and maintenance of standards and reagents will be accomplished according to the methods referenced. All standards and standard solutions are to be formally documented (i.e., in a logbook) and should identify the supplier, lot number, purity/concentration, receipt/preparation date, preparers name, method of preparation, expiration date, and any other pertinent information. All standard solutions shall be validated prior to use. Care shall be exercised in the proper storage and handling of standard solutions (e.g., separating volatile standards from nonvolatile standards). The laboratory shall continually monitor the quality of the standards and reagents through well documented procedures.

6.1.2 Balances

The analytical balances shall be calibrated and maintained in accordance with manufacturer specifications. Calibration is conducted with two Class AS weights that bracket the expected balance use range. The laboratory shall check the accuracy of the balances daily and they must be properly documented in permanently bound logbooks.

6.1.3 Refrigerators/Freezers

The temperature of the refrigerators and freezers within the laboratory shall be monitored and recorded daily. This will verify that the quality of the standards and reagents is not compromised, and the integrity of the analytical samples is upheld. Appropriate acceptance ranges (2 to 6°C for refrigerators) shall be clearly posted on each unit in service.

6.1.4 Water Supply System

The laboratory must maintain a sufficient water supply for all project needs. The grade of the water must be of the highest quality (analyte-free) to eliminate false- positives from the analytical results. Ultraviolet cartridges or carbon absorption treatments are recommended for organic analyses and ion-exchange treatment is recommended for inorganic tests. Appropriate documentation of the quality of the water supply system(s) will be performed on a regular basis.

6.2 Laboratory Instruments

Calibration of instruments is required to verify that the analytical system is operating properly and at the sensitivity necessary to meet established quantitation limits. Each instrument for organic and inorganic analyses shall be calibrated with standards appropriate

to the type of instrument and linear range established within the analytical method(s). Calibration of laboratory instruments will be performed according to specified methods.

In addition to the requirements stated within the analytical methods, the contract laboratory will be required to analyze an additional low-level standard at or near the detection limits. In general, standards will be used that bracket the expected concentration of the samples. This will require the use of different concentration levels, which are used to demonstrate the instrument's linear range of calibration.

Calibration of an instrument must be performed prior to the analysis of any samples and then at periodic intervals (continuing calibration) during the sample analysis to verify that the instrument is still calibrated. If the contract laboratory cannot meet the method required calibration requirements, corrective action shall be taken. All corrective action procedures taken by the contract laboratory are to be documented, summarized within the case narrative, and submitted with the analytical results.

7.0 Analytical Methods

All laboratory analyses will be performed by an accredited and appropriately (NYSDEC ELAP) certified analytical laboratory. Analyses to be completed by the laboratory for the Barker Chemical Site are listed in **Table 1** of this QAPP.

7.1 Analytical Capabilities

The analytical laboratory is fully equipped for analysis of all types of water, air, and soil samples for chemical contaminants, bacteriological quality, and general characterization. Proven and approved analytical techniques are used, backed up by a rigorous system of QC and QA checks to ensure reliable and defensible data. All laboratory work is performed in accordance with guidelines established by USEPA, the NYSDOH, and the National Institute of Occupational Safety and Health (NIOSH).

Organic analysis is accomplished by gas chromatography (GC), high performance liquid chromatography (HPLC), and or gas chromatography/mass spectrometry (GC/MS). The laboratory uses two types of instruments for analysis of metals in various matrices: Atomic Absorption Spectrometry (AAS) and Inductively Coupled Plasma (ICP). Laboratory

procedures to be utilized for sample preparation and analysis are referenced in the NYSDEC ASP.

7.1.1 Method Detection Limits

Method detection limits are determined according to procedures outlined in 40 CFR Part 136, Appendix B or USEPA CLP. General analytical detection limits are usually determined by the lowest point on the curve. Detection limits are determined at least annually for all appropriate analytical methods and for purposes of site management, the detection and reporting limits for all analytes will be below applicable Standards, Criteria and Guidance values. A listing of the laboratory's method detection limits is available upon request.

7.2 Internal Quality Control Samples

Internal quality control (QC) checks are used to determine if analytical operations at the laboratory are in control, as well as determining the effect sample matrix may have on data being generated. Two types of internal checks are performed and are described as laboratory QC and matrix-specific QC samples. The type and frequency of specific QC samples performed by the contract laboratory will be according to the specified analytical method and project specific requirements. Acceptable criteria and target ranges for these QC samples are presented within the referenced analytical methods.

QC results which vary from acceptable ranges shall result in the implementation of appropriate corrective measures, potential application of qualifiers, and/or an assessment of the impact these corrective measures have on the established data quality objectives.

The matrix-specific QC samples are described in Section 4.2. Laboratory QC samples consist of laboratory blanks, calibration standards, reference standards, surrogate standards, internal standards, laboratory duplicate or matrix spike duplicate, and check standards samples as appropriate to the methodology. These laboratory QC samples are described as follows:

7.2.1 Laboratory Blanks

Three types of laboratory blanks, one or more of which will be utilized depending on the analysis, are described below:

- Method blanks consist of analyte-free water and are subjected to every step of the analytical procedure to determine possible contamination.
- Reagent blanks are similar to method blanks but incorporate only one of the preparation reagents in the analysis. When a method blank indicates significant contamination, one or more reagent blanks are analyzed to determine the source.
- Calibration blanks consist of a pure reagent matrix and are used to zero an instrument's response, thus establishing the baseline.

7.2.2 Calibration Standards

A calibration standard may be prepared in the laboratory by dissolving a known amount of a pure compound in an appropriate matrix. The final concentration calculated from the known quantities is the true value of the standard. The results obtained from these standards are used to generate a standard curve and thereby quantify the compound in the environmental sample. A minimum of three (3) calibration standards will be used to generate a standard curve for all analyses.

7.2.3 Reference Standards

A reference standard is prepared in the same manner as a calibration standard but from a different source. Reference standards may be obtained from the USEPA. The final concentration calculated from the known quantities is the "true" value of the standard. The important difference in a reference standard is that it is not carried through the same process used for the environmental samples but is analyzed without digestion or extraction. A reference standard result is used to validate an existing concentration calibration standard file or calibration curve.

7.2.4 Matrix Spike Sample

A sample spike is prepared by adding to an environmental sample (before extraction or digestion) a known amount of pure compound of the same type that is to be assayed for in the environmental sample. Spikes are added at 1 to 10 times the expected sample concentration or approximately 10 times the method detection limit. These spikes simulate the background and interferences found in the actual samples, and the calculated percent recovery of the spike is taken as a measure of the accuracy of the total analytical method.

7.2.5 *Blank Spike Sample*

A blank spike is the same as a spike sample except the spike is added to analyte-free water. The blank spike is used to determine whether the sample preparation and analysis are under control.

7.2.6 *Surrogate Standards*

A surrogate is prepared by adding a known amount of pure compound to the environmental sample; the compound selected is not one expected to be found in the sample, but is similar in nature to the compound of interest. Surrogate compounds are added to the sample prior to extraction or digestion. Surrogate spike concentrations indicate the percent recovery of the analytes and, therefore, the efficiency of the methodology.

7.2.7 *Internal Standards*

Internal standards are similar to surrogate standards in chemical composition but are used to quantify the concentration of analytes sampled based on the relative response factor. Internal standards are added to the environmental sample just prior to instrumental analysis.

7.2.8 *Laboratory Duplicate or Matrix Spike Duplicate*

Laboratory duplicates are aliquots of the same sample that are split prior to analysis and treated exactly the same throughout the analytical method. Spikes and duplicates for the batch are normally aliquots of the same sample. For organics, spikes are added at approximately 10 times the method detection limit. The relative percent difference (RPD) between the values of the matrix spike and matrix spike duplicate for organics or between the original and the duplicate for inorganics is taken as a measure of the precision of the analytical method. In general, the tolerance limit for RPDs between laboratory duplicates should not exceed 20% for validation in homogeneous samples.

7.2.9 *Check Standards*

Inorganic and organic check standards are prepared with reference standards or are available from the USEPA. They are used as a means of evaluating analytical techniques of the analyst. Check standards or samples are subjected to the entire sample procedure, including extraction, digestion, etc., as appropriate for the analytical method utilized. The check standard or sample can provide information on the accuracy of the analytical method independent of various sample matrices.

7.3 Laboratory Instrumentation

Laboratory capabilities will be demonstrated initially for instrument and reagent/standards performance as well as accuracy and precision of analytical methodology. A discussion of reagent/ standard procedures and brief descriptions of calibration procedures for major instrument types follow.

All standards are obtained directly from USEPA or through a reliable commercial supplier with a proven record for quality standards. All commercially supplied standards will be traceable to USEPA or National Institute of Standards and Technology (NIST) reference standards and appropriate documentation will be obtained from the supplier. In cases where documentation is not available, the laboratory will analyze the standard and compare the results to a known USEPA-supplied or previous NIST-traceable standard.

All sections of the laboratory will have SOPs for standard and reagent procedures to document specific standard receipt, documentation, and preparation activities. In general, the individual SOPs incorporate the following items:

- Documentation and labeling of date received, lot number, date opened, and expiration date;
- Documentation of traceability;
- Preparation, storage, and labeling of stock and working solutions; and
- Establishing and documenting expiration dates and disposal of unusable standards.

Each laboratory instrument will be labeled clearly with a unique identifier that relates to all laboratory calibration documentation. Laboratory SOPs and calibration procedures are detailed in the laboratory's Quality Assurance Manual, available upon request.

8.0 Calculation of Data Quality Indicators

8.1 Precision

Precision is evaluated using analyses of a field duplicate or a laboratory MS/MSD that indicate analytical precision through the reproducibility of the analytical results. RPD is used to evaluate precision by the following formula:

$$RPD = \frac{(X_1 - X_2)}{[(X_1 + X_2)/2]} \times 100\%$$

where:

X₁ = Measured value of sample or matrix spike

X₂ = Measured value of duplicate or matrix spike duplicate

Precision will be determined using MS/MSD (for organics) and matrix duplicates (for inorganics) analyses.

8.2 Accuracy

Accuracy is defined as the degree of difference between the measured or calculated value and the true value. The closer the numerical value of the measurement comes to the true value or actual concentration, the more accurate the measurement is. Analytical accuracy is expressed as the percent recovery of a compound or element that has been added to the environmental sample at known concentrations before analysis. Analytical accuracy may be assessed using known and unknown QC samples and spiked samples. It is presented as percent recovery. Accuracy will be determined from matrix spike, matrix spike duplicate, and matrix spike blank samples, as well as from surrogate compounds added to organic fractions (i.e., volatiles, semi-volatiles, PCB), and is calculated as follows:

$$Accuracy (\%R) = \frac{(X_s - X_u)}{K} \times 100\%$$

where:

X_s = Measured value of the spike sample

X_u = Measured value of the unspiked sample

K = Known amount of spike in the sample

8.3 Completeness

Completeness is calculated on a per matrix basis for the project and is calculated as follows:

$$Completeness (\%C) = \frac{(X_v - X_n)}{N} \times 100\%$$

where:

X_v = Number of valid measurements

X_n = Number of invalid measurements

N = Number of valid measurements expected to be obtained

9.0 Corrective Actions

Laboratory corrective actions shall be implemented to resolve problems and restore proper functioning to the analytical system when errors, deficiencies, or out-of-control situations exist at the laboratory. Full documentation of the corrective action procedure needed to resolve the problem shall be filed in the project records, and the information summarized in the case narrative. A discussion of the corrective actions to be taken is presented in the following sections.

9.1 Incoming Samples

Problems noted during sample receipt shall be documented by the laboratory. The Project Manager shall be contacted immediately for problem resolution. All corrective actions shall be documented thoroughly.

9.2 Sample Holding Times

If any sample extraction or analyses exceed method holding time requirements, the Project Manager shall be notified immediately for problem resolution. All corrective actions shall be documented thoroughly.

9.3 Instrument Calibration

Sample analysis shall not be allowed until all initial calibrations meet the appropriate requirements. All laboratory instrumentation must be calibrated in accordance with method requirements. If any initial/continuing calibration standards exceed method QC limits, recalibration must be performed and, if necessary, reanalysis of all samples affected back to the previous acceptable calibration check.

9.4 Reporting Limits

The laboratory must meet the method required detection limits listed in NYSDEC ASP. If difficulties arise in achieving these limits due to a sample matrix, the laboratory must

notify the Project Manager for problem resolution. To achieve those detection limits, the laboratory must utilize all appropriate cleanup procedures to retain the project required detection limits. When any sample requires a secondary dilution due to high levels of target analytes, the laboratory must document all initial analyses and secondary dilution results. Secondary dilution will be permitted only to bring target analytes within the linear range of calibration. If samples are analyzed at a secondary dilution with no target analytes detected, the Project Manager will be immediately notified so that appropriate corrective actions can be initiated.

9.5 Method QC

All method-specified QC samples shall meet the method requirements referenced in the analytical methods. Failure of method-required QC will result in the review and possible qualification of all affected data. If the laboratory cannot find any errors, the affected sample(s) shall be reanalyzed or re-extracted/redigested, then reanalyzed within method-required holding times to verify the presence or absence of matrix effects. If matrix effect is confirmed, the corresponding data shall be flagged accordingly using the flagging symbols and criteria. If matrix effect is not confirmed, then the entire batch of samples may have to be reanalyzed or re-extracted/redigested, then reanalyzed at no cost. The Project Manager shall be notified as soon as possible to discuss possible corrective actions should unusually difficult sample matrices be encountered.

9.6 Calculation Errors

All analytical results must be reviewed systematically for accuracy prior to submittal. If upon data review, calculation or reporting errors exist, the laboratory will be required to reissue the analytical data report with the corrective actions appropriately documented in the case narrative.

10.0 Data Reduction, Reporting, Validation, and Usability

10.1 Data Reduction

Laboratory analytical data are first generated in raw form at the instrument. These data may be either in a graphic or printed tabular format. Specific data generation procedures and calculations are found in each of the referenced methods. Analytical results must be reported consistently. Identification of all analytes must be accomplished with an authentic

standard of the analyte traceable to NIST or USEPA sources. Individuals experienced with an analysis and knowledgeable of requirements will perform data reduction.

10.2 Deliverables

Deliverable provided by the contract laboratory include the following:

- Category B Data Package; and
- Quality Assurance Reports.

10.2.1 *Category B Data Package*

All analytical data will be reported by the laboratory with NYSDEC ASP Category B deliverables. The Category B data package includes:

1. A detailed summary of the report contents and any quality control outliers or corrective actions taken.
2. Chain of Custody documentation.
3. Sample Information including date collected, date extracted, date analyzed, and analytical methods.
4. Data (including raw data) for:
 - samples
 - laboratory duplicates
 - method blanks
 - spikes and spike duplicates
 - surrogate recoveries
 - internal standard recoveries
 - calibrations
 - any other applicable QC data.
5. Method detection limits and/or instrument detection limits.
6. Run logs, standard preparation logs, and sample preparation logs.
7. Percent solids (where applicable).

10.2.2 *Quality Assurance Reports*

For the laboratory, a general QA report summarizing problems encountered throughout the laboratory effort, including sample custody, analyses, and reporting is generated. This report identifies areas of concern and possible resolutions in an effort to

ensure data quality.

Upon completion of a project sampling effort, analytical and QC data will be included in a comprehensive report that summarizes the work and provides a data evaluation. A discussion of the validity of the results in the context of QA/QC procedures will be made, as well as a summation of all QA/QC activity.

Serious analytical or sampling problems will be reported to the Project Manager. Time and type of corrective action, if needed, will depend on the severity of the problem and relative overall project importance. Corrective actions may include altering procedures in the field, conducting an audit, or modifying laboratory protocol. All corrective actions will be implemented after notification and approval of the Project Manager.

In addition to the laboratory report narrative, QA data validation reports that include any contractual requirements will also be provided to the Project Manager. These QA reports will be submitted with the analytical data, on a monthly basis, or at the conclusion of the project.

10.3 Data Validation and Usability

Prior to the submission of the Periodic Review Report to NYSDEC, all data will be evaluated for precision, accuracy, and completeness.

QA/QC requirements from both methodology and company protocols will be strictly adhered to during sampling and analytical work. All data generated will be reviewed by comparing and interpreting results from instrument responses, retention time, determination of percent recovery of spiked samples or blanks, and reproducibility of duplicate sample results. All calculations and data manipulations are included in the appropriate methodology references.

Control charts and calibration curves will be used to review the data and identify outlying results.

10.3.1 Data Validation

A third-party validator will be responsible for an independent review of all analytical work performed under the NYSDEC ASP or USEPA CLP protocol. The functions will be to

assess and summarize the quality and reliability of the data for the purpose of determining its usability and to document for the historical record any factors affecting data usability, such as discrepancies and poor laboratory practices. The data validator will be responsible for determining completeness and compliance.

Information available to the data validator for the completion of these functions include the NYSDEC ASP Category B data package, information from the sampling team regarding field conditions and field QA samples, chain of custody and shipping forms. The data package is designed to provide all necessary documentation to verify compliance with NYSDEC ASP or USEPA CLP protocol and the accuracy and reliability of the reported results.

In order to effectively review the data package, the data validator will obtain a general overview of each case. This includes the exact number of samples, their assigned numbers, and their matrix. The data validator will deliver the data validation report within 30 days of receipt of the data package.

If a problem arises between the data validator and the laboratory, the data validator must submit written questions to the laboratory. The laboratory will be required to respond in writing within 10 working days to correct any deficiencies. If the data validator does not receive a written response from the laboratory within the specified time period, the data in question shall be considered noncompliant.

Sampling locations will be obtained from the sampling records, such as the chain-of-custody forms. This information is necessary for preparation of the data summary, evaluation of adherence to sample holding times, discussion of matrix problems, and discussion of contaminants detected in the samples.

The following is a brief outline of the data validation process:

- Compilation of all samples with the dates of sampling, laboratory receipt, and analysis;
- Compilation of all QC samples, such as field blanks, field duplicates, MS/MSD samples, laboratory blanks, and laboratory replicates;

- Review of chain of custody documents for completeness and correctness;
- Review of laboratory analytical procedure and instrument performance criteria;
- Qualification of data outside acceptable QC criteria ranges;
- Preparation of a memorandum summarizing any problems encountered and the potential effects on data usability;
- Preparation of a data summary, including validated results, with sample matrix, location, and identification; and
- Tabulation of field duplicates, laboratory replicate, and blank results.

Copies of all laboratory reports, data validation and usability reports, as well as all data summary packages, will be provided to the Project Manager.

10.3.2 Data Usability Summary Reports

Data Usability Summary Reports (DUSRs) will be prepared for all analytical results by an Environmental Scientist having a Bachelor's degree in a relevant natural or physical science or field of engineering and also having experience in environmental sampling, analysis and data review. The DUSRs provide a thorough evaluation of analytical data without the costly and time-consuming process of third-party data validation. The primary objective of the DUSRs is to determine if the analytical data meets the criteria for data quality and use.

Any QC exceedances must be numerically specified in the DUSRs with the corresponding QC summary sheet from the data package attached to the DUSRs. All data that would be rejected by the EPA Region 2 Data Validation Guidelines must also be rejected in the DUSRs.

Once the data packages have been reviewed the DUSRs proceed to describe the samples and the analytical parameters. Data deficiencies, analytical protocol deviations and quality control problems are identified and their effect on the data will be discussed. The DUSRs will also include recommendations on resampling/reanalysis. All data qualifications must be documented following the NYSDEC ASP (2005 revision) guidelines.

Table 1
Proposed Sampling and Analysis Summary

Analyte	Location	Media	Number	QA/QC	Total
TCL VOCs (Method 8260C)	Wells	Groundwater	8	1	9
TCL SVOCs (Method 8270D)	Wells	Groundwater	8	1	9
TCL Pesticides (Method 8081B)	Wells	Groundwater	8	1	9
TAL Metals (Method 6010C)	Wells	Groundwater	8	1	9
Mercury (Method 7470A)	Wells	Groundwater	8	1	9
Sulfide (Method 4500D)	Wells	Groundwater	8	1	9
Sulfate (Method 300)	Wells	Groundwater	8	1	9
TCL VOCs (Method 8260)	Surface Water Bodies	Surface Water	3	1	4
TCL SVOCs (Method 8270D)	Surface Water Bodies	Surface Water	3	1	4
TCL Pesticides (Method 8081B)	Surface Water Bodies	Surface Water	3	1	4
TAL Metals (Method 6010C)	Surface Water Bodies	Surface Water	3	1	4
Mercury (Method 7470A)	Surface Water Bodies	Surface Water	3	1	4
Sulfide (Method 4500D)	Surface Water Bodies	Surface Water	3	1	4
Sulfate (Method 300)	Surface Water Bodies	Surface Water	3	1	4

Table 2
Sample Preservation and Holding Times

Parameter	Method Number	Container Type and Size	Preservation	Holding Time*
Surface Water and Groundwater Samples				
VOCs	8260C	3 x 40-ml. glass VOA vial with Teflon-lined cap	HCL, pH<2; Cool to 4° C	14 days
SVOCs	8270D	2 x 1000 ml amber glass	Cool to 4°C	7 days to extract; analyze 40 days from extraction
Pesticides	8081B	2 x 1000 ml amber glass	Cool to 4°C	7 days to extract
Metals + Mercury	6010C/ 7470A	1 x 500 ml plastic	HNO3, pH<2; Cool to 4° C	180 Days, Hg 28 days
Sulfide	4500D	2 x 250 ml plastic	ZnOAC, NaOH, pH>9; Cool to 4° C	7 Days
Sulfate	300	1 x 250 ml plastic	Cool to 4°C	28 Days

* Holding times are based on verified time of sample receipt (VTSR) at the laboratory