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1. Introduction

This Quality Control Document (QCD) has been developed by O'Brien & Gere on behalf of Fall Creek Redevelopment, LLC for the Focused Site Investigation (FSI) at the Site in Ithaca, New York. This document is provided as part of the FSI Work Plan (O'Brien & Gere 2008), which was generated to assess the potential for environmental impact under the basement slabs of the existing and former Ithaca Gun buildings located at 121-125 Lake Street in Ithaca, New York.

The quality control (QC) information and assumptions about laboratory analyses presented in this QCD will be utilized during the data validation that will be performed on Site environmental samples.

2. Project Summary

During the FSI at the Ithaca Gun Site, soil and ground water samples will be collected to assess the concentration of target constituents from the Site. Samples will be submitted to Life Science Laboratories, Inc. (LSL) of Syracuse, New York for analysis.

The analyses to be performed are presented in Table 1 and the number of samples to be collected during the FSI is presented in Table 2.

During the investigation, sample analysis will include the following parameters, which are addressed in this QCD:

- Total volatile organic compounds (VOCs)
- Toxicity characteristic leachate procedure (TCLP) VOCs
- Total semivolatile organic compounds (SVOCs)
- Total metals and mercury
- TCLP metals and mercury
- Total cyanide.

Data packages will be provided to O'Brien & Gere and will include comprehensive documentation associated with the sample preparation and analysis process.

In the event that the data usability completeness objective of 95% is not achieved, samples may be recollected at the discretion of the Project Manager

The field tasks, sample locations and rationale for this investigation are summarized in the Work Plan.

Analyses will meet the requirements listed in the analytical methods listed in Table 2 and the quality control requirements and corrective actions listed in Tables 4-A, 4-B, and 4-C. The most recent laboratory control limits will be used to evaluate the sample data.

The laboratory will report non-detected sample results to the quantitation limits (QLs). The QL represents the minimum concentration that can be identified and quantified above the method detection limit (MDL). The MDL represents the minimum concentration that can be reported with 99 percent confidence that the analyte concentration is greater than zero. Results that are less than the QLs but greater than the MDLs will be reported by the laboratory using the "J" flag. The laboratory-generated MDLs, which are applicable at the time of analysis, will be provided by the laboratory along with the sample results. The QLs listed in Tables 3-1A, 3-1B, 3-2A, 3-2B, 3-3A and 3-3B, or the most recent detection limits, will be reported by the laboratory. The action limits listed in Tables 3-1A, 3-1B, 3-2A, 3-2B, 3-3A and 3-3B, or the most recent standards, will be used to evaluate the analytical data. Laboratories periodically update the MDL and QL values as part of internal laboratory policy.

In the case of matrix interference, the laboratory will perform sample cleanup as provided by the methods. Interferences will be identified and documented. Samples may be diluted only if analytes of concern generate responses in excess of the linear range of the instrument. When matrix interferences

are present, samples will be cleaned up during the preparation process using appropriate methods. The clean-up, extraction and sample preparation methods will be listed in the data package case narrative. If the laboratory has taken appropriate actions and matrix interferences prevent the laboratory from achieving the specified QLs / MDLs, the Project Manager will be contacted as soon as the situation is identified. The Laboratory Project Manager will document in the data package case narrative how the laboratory demonstrated good analytical practices in order to attempt to achieve the specified analytical limits.

The analytical data will be reported to the contractor in New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) Category B deliverable format, including the forms described in the NYSDEC guidance, in both hardcopy and electronic data format.

As part of the NYSDEC Data Usability Summary Report (DUSR) process (NYSDEC, 2002), full data validation will be performed on 100% of the data for each analysis type as described in Section 8 of this document.

The laboratory will provide complete data packages to O'Brien & Gere within 4 weeks of receipt of the last sample in a sampling event.

Data will be managed in a relational database management system (DBMS). Laboratory analytical data will be provided in electronic disk deliverable (EDD) format for direct upload into the DBMS. Associated validation qualifiers will be manually entered into the DBMS.

Records will be incorporated into the final project files for the samples. The project files will be archived by the Engineer for a period of 10 years.

The project schedule will be identified at a later date.

3. Project Organization and Responsibilities

3.1. Project Participants

While each person involved in the FSI and generation of data is implicitly a part of the QA program for the project, certain individuals have specific, designated responsibilities. Within O'Brien & Gere, these are the Project Officer, Project Manager, Quality Assurance (QA) Officer, Field Leader, Data Management Personnel, and Sampling Personnel.

Life Science Laboratories, Inc. (Life Science Laboratories) of Syracuse, New York will provide analytical services for the FSI. Laboratory personnel with QA/QC responsibilities include the Laboratory Project Manager and Laboratory Sample Custodian. Samples will not be sent to another laboratory without the permission of the O'Brien & Gere Project Manager. Samples will not be subcontracted to a laboratory for sample analysis through another laboratory.

The following sections describe the relationship among the project participants.

3.2. NYSDEC Project Manager

The NYSDEC has assigned Mr. Gary Holmes as the Project Manager for the Site. As such, he will be responsible for reviewing submissions and overseeing project activities on behalf of NYSDEC.

3.3. Fall Creek Redevelopment, LLC Project Manager

Mr. Wallace Diehl is the Fall Creek Redevelopment, LLC Project Manager for the Site. As such, he will be responsible for reviewing submissions and overseeing project activities.

3.4. O'Brien & Gere Personnel

3.4.1. Project Officer

Mr. Peter Grevelding will serve as the Project Officer. As such, he will be responsible for the overall corporate management of the FSI and for the completion of tasks specified in the QCD. It will be his responsibility to provide for the allocation of staff and other resources required to complete the project within the specified schedule and budget.

3.4.2. Project Manager

Ms. Debra Wright will serve as the Project Manager and client contact. As such, she will have responsibility for the implementation and completion of each of the tasks identified in the QCD. She will manage the day-to-day project operations and administrative aspects of the project and will function as the client and regulatory contact for the project. In addition, she will have responsibility for coordinating the technical aspects, strategy, and oversight of the FSI and field sampling activities.

3.4.3. QA Officer

Ms. Karen Storne will serve as the QA Officer. As such, she will be responsible for overall project QA. She will review project plans and revisions to such plans to maintain proper QA throughout the FSI. In addition, she or her designee, will be responsible for data quality review, corrective actions, and coordinating QA/QC efforts between O'Brien & Gere and the laboratory.

3.4.4. Field Leader

Ms. Debra Wright will also serve as the Field Leader. As such, he will oversee field and related activities as described in the QCD. The sampling personnel will report to the Field Leader who will be responsible for leading, coordinating, and supervising the day-to-day field activities. The Field Leader's responsibilities include:

- Communicate and coordinate with laboratory prior to sample collection and during shipment of sample coolers to the laboratory
- Develop and implement field-related sampling plans and schedule
- Coordinate and manage field staff
- Supervise or act as the field sample custodian
- Implement QC for technical data, including field measurements
- Adhere to work schedules
- Coordinate and oversee technical efforts of subcontractors assisting the field team
- Identify problems at the field team level and resolve difficulties
- Implement and document corrective action procedures

3.4.5. Data Management Personnel

Data management staff from O'Brien & Gere will provide data management services.

3.4.6. Sampling Personnel

Experienced chemists, engineers, geologists, hydrogeologists, and/or environmental technicians will conduct sampling tasks required by the FSI. Their responsibilities will include the documentation of proper sample collection protocols, sample collection, equipment decontamination, and chain-of-custody documentation. The sampling personnel will report to the Field Leader.

3.5. Laboratory Personnel

3.5.1. Project Manager

Mr. Anthony Crescenzi of Life Science Laboratories will serve as the Laboratory Project Manager. As such, he will be responsible for the laboratory's QA/QC activities associated with the project. The specific duties of the Laboratory Project Manager include determining whether analyses are conducted within the method requirements and that laboratory custody procedures are followed. Moreover, the Laboratory Project Manager monitors daily precision and accuracy records, maintains detailed copies of all procedures, reschedules analyses based on unacceptable data accuracy or precision, and identifies and implements corrective actions necessary to maintain QA standards.

The Laboratory Project Manager or his designee will conduct initial data assessments of analytical data results, based on the requirements of the QCD, and report the findings in the data packages. Major QA/QC issues will be reported to the O'Brien & Gere QA Officer.

3.5.2. Laboratory Sample Custodian

Adam Schotz of Life Science Laboratories will serve as the Laboratory Sample Custodian. As such, his responsibilities will include verifying proper sample entry and sample handling procedures by laboratory personnel. The Laboratory Sample Custodian will report to the Laboratory Project Manager.

4. Field Sampling QA/QC Procedures

4.1. Sample Custody

The analytical laboratory will supply appropriate sample containers in coolers, as well as preservatives (as appropriate). QA measures for this project will begin with the sample containers – pre-cleaned containers will be purchased from a USEPA-certified manufacturer (I-Chem 200 or equivalent).

Immediately after collection, samples will be transferred to properly labeled sample containers, and properly preserved. Table 2 lists the proper sample container, sample volumes, and preservation. Samples requiring refrigeration for preservation will be promptly transferred to coolers packed with wet ice and/or ice packs. If field storage is required, the samples will be stored in a secured storage facility and an approximate cooler temperature of 4 °C will be maintained. Samples will be shipped or transported within 24 hours of being collected and will arrive at the laboratory no later than 48 hours after sample collection.

4.2. Field Custody Procedures

The field sampler is personally responsible for the care and custody of the sample until transferred.

The field logbook will be used to note information regarding collection of samples and any notable observations. Entries will be signed and dated. Field logbooks will be waterproof and bound. The logbook will be dedicated to the project and pages will not be removed. Corrections will be made by drawing a single line through the incorrect data and initialing and dating the correction that was made to the side of the error. An initialed diagonal line will be used to indicate the end of an entry or the end of the day's activities.

The following information will be recorded in the field logbook by the field sampling team:

- Name and title of author, date, and time of Site entry, and physical/environmental conditions during the field activity
- Meteorological data
- Project number, client name, and Site name
- Name and title of field crew members
- Sample media
- Sample collection method, including equipment utilized
- Number and volume of samples collected
- Description of sample locations
- Date and time of sample collection
- Sample and QA/QC identification numbers
- Field observations
- Field measurements made and equipment used
- Calculations, results, and calibration data for field sampling and measurements
- References for maps and photographs of the sample location

- Location of samples in custody if not relinquished to laboratory at end of day
- Dates and method of sample shipments.

A completed sample identification label or tag will be attached to each investigative or QC sample and the sample placed in a shipping container. The identification on the label/tag must be sufficient to enable cross-reference with the logbook. The sample label/tag will be recorded using waterproof, non-erasable ink and will be attached to the sample container using adhesive.

The sample labels/tags will contain the following information:

- Sample number identification
- Project number
- Date and time of sample collection
- Designation of the sample as a grab or composite
- Type of sample matrix
- Sample location
- Sampler initials
- Whether the sample is preserved or unpreserved
- Space for laboratory sample number (only on the sample tag)
- General types of analysis to be performed.

Chain-of-custody records will be kept starting at the time that sample containers are placed in the coolers for transportation to the laboratory. One completed chain-of-custody record must be kept with each sample cooler at all times.

When transferring the possession of samples, individuals relinquishing and receiving will sign, date, and note the time on the chain-of-custody. Custody of samples must be continuous between parties and time gaps must not be present. Each shipment of samples to the laboratory must have its own chain-of-custody record with the contents of the shipment, method of shipment, name of courier, and other pertinent information written on the record. The original record accompanies the shipment and the copies are kept with the field logbook and distributed to the O'Brien & Gere Project Manager. Freight bills, postal service receipts, and bills of lading will be retained as permanent documentation.

If the samples are shipped, the courier's air bill will be attached to the chain-of-custody and the air bill number will be written on the chain-of-custody form.

The chain-of-custody documentation will be recorded using waterproof, non-erasable ink. One sample will be entered on each line of the chain-of-custody record and not be split among multiple lines.

The chain-of-custody form will contain the following information:

- Project identification and number
- Sample description/location
- Required analysis
- Date and time of sample collection
- Type and matrix of sample

- Number of sample containers
- Analysis requested/comments
- Sampler signature/date/time
- Date and signature of the field representative
- Date and signature of the laboratory representative
- Carrier used to ship coolers
- Air bill number (if shipped by a commercial carrier).

In the case that high concentrations are suspected to be present in the samples, a note to that effect will be included on the chain-of-custody form.

Environmental samples will be packed by the field representative prior to shipment using the following procedures:

- Select a sturdy cooler in good repair and clean. Secure and tape the drain plug with fiber or duct tape.
- Be sure the lids on all bottles are tight (will not leak) and placed in to tightly sealed plastic bags.
- Put ice that has been placed in properly sealed heavy-duty polyethylene bags on top of, and/or between the samples. Pack samples securely to eliminate breakage during shipment with ice packs to maintain the inside temperature at approximately 4°C.
- Place chain-of-custody record into a Ziploc plastic bag, tape the bag to the inner side of the cooler lid, and close the cooler and securely tape (preferably with fiber tape) the top of the cooler shut. The field sampler will initial and date the seal. The seals must be broken to open the cooler and will indicate tampering if the seal is broken before receipt at the laboratory. Two custody seals will be affixed to the latch and lid of the cooler. The custody seals will consist of adhesive-backed tape that easily rips if it is disturbed.
- A label containing the name and address of the shipper will be placed on the outside of the cooler.

The field sampling team will transport or ship the cooler via an overnight delivery service or hand deliver to the laboratory. Prior to shipment of sample coolers, the field sampling team will contact the laboratory to notify the laboratory of the shipment.

Samples will remain in the custody of the sampler until transfer of custody is completed. Transfer consists of:

- Delivery of samples to the Laboratory Sample Custodian, and/or
- Signature of the Laboratory Sample Custodian on the chain-of-custody form as receiving the samples and signature of sampler as relinquishing the samples.

4.4. Laboratory Custody Procedures

When the samples arrive at the laboratory, the Laboratory Sample Custodian will sign the courier's air bill or bill of lading (unless hand-delivered) and will note the cooler temperature on the chain-of-custody form. If the cooler temperature is greater than 6 °C, the O'Brien & Gere Project Manager will

be notified. If the cooler arrives at the laboratory after hours, an external chain-of-custody will be properly filled out and will accompany the cooler until the laboratory receives the cooler.

The Laboratory Sample Custodian's duties and responsibilities upon sample receipt will be to:

- Document receipt of samples by signing the record with the date and time of sample receipt.
- Note the cooler temperature on the chain-of-custody form.
- Inspect sample shipping containers for the presence or absence of custody seals (only if shipped via overnight courier) and for container integrity.
- Sign the appropriate forms or documents, verify, and record the agreement or disagreement of information on sample documents and, if there are discrepancies, record the problem and notify the O'Brien & Gere Project Manager.
- Assign a laboratory number for each sample upon receipt. That sample number will be placed on the sample label which will remain attached to the sample container.
- Log sample information into the laboratory sample tracking system.
- Label sample with a unique, sequential laboratory sample number.
- Place samples in the walk-in cooler or sample storage area that is a secure, limited-access storage.

The laboratory will immediately contact the O'Brien & Gere Project Manager if issues pertaining to sample condition or documentation are detected (broken security seal; broken, open, or otherwise compromised sample bottles; chain-of-custody information in disagreement with sample labels, etc.).

At the laboratory, the analysts will be required to log samples and extracts in and out of storage as the analysis proceeds. Samples and extracts will be returned to secure storage at the close of business. Written records will be kept of each time the sample or extract changes hands. Care must be exercised to properly complete, date, and sign items needed to generate data.

Procedures to be followed by the laboratory include:

- Samples will be handled by the minimum number of people possible.
- The laboratory will set aside a secured sample storage area consisting of a clean, dry, refrigerated, isolated room.
- A specific person will be designated sample custodian. Incoming samples will be received by the custodian who will indicate receipt by signing the chain-of-custody form.
- The custodian will ensure that samples which are heat-sensitive, light-sensitive, radioactive, or which require special handling in other ways, are properly stored and maintained prior to analysis.
- The analytical area will be restricted to authorized personnel only.
- After sample analyses are complete, the analytical data will be kept secured and released to authorized personnel only.

If QC samples have not been properly identified during sample collection, the Laboratory Project Manager will contact the O'Brien & Gere Project Manager to assign QC samples prior to the start of sample analysis.

4.5. Final Record File Chain-of-Custody Procedures

The final record file will be the central repository for documents that constitute evidence relevant to sampling and analysis activities as described in this QCD. O'Brien & Gere is the custodian of the record file and maintains the contents of evidence files for the Site including relevant records, reports, logs, field notebooks, pictures, subcontractor reports, and data reviews.

The final file will be stored at O'Brien & Gere and will consist of the following:

- Laboratory data packages including summary and raw data from the analysis of environmental and QC samples, chromatograms, mass spectra, calibration data, work sheets, and sample preparation log
- Chain-of-custody records
- Field logbooks and data
- Pictures and drawings
- Correspondence.

The record file will be maintained in a secured, limited access area until submittals for the project have been reviewed and approved, and for a minimum of 10 years past the submittal date of the final report.

5. Laboratory QA/QC procedures

A brief description of laboratory quality assurance/ quality control (QA/QC) analyses is presented in the following sections.

5.1. GC/MS tuning

Tuning and performance criteria are established to verify mass resolution, identification, and to some degree, instrument sensitivity. These criteria are not sample specific; conformance is determined using standard materials. Therefore, these criteria should be met in all circumstances.

5.2. Calibration

Compliance requirements for satisfactory instrument calibration are established to verify that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of analysis, and continuing calibration and performance checks document satisfactory maintenance and adjustment of the instrument on a day-to-day basis.

5.3. Blanks

Corrective action procedures are implemented for blank analyses if target compounds are detected at concentrations greater than the requirements presented in corrective action Tables 4-A, 4-B and 4-C. The criteria for evaluation of blanks apply to any blank associated with a group of samples. If problems with a blank exist, data associated with the project must be carefully evaluated to determine whether or not there is an inherent variability in the data for the project, or if the problem is an isolated occurrence not affecting other data.

5.4. Internal standards performance

Internal standards, which are compounds not found in environmental samples, will be spiked into samples, blanks, method spikes and method spike duplicates (MS/MSDs), and laboratory control samples (LCSs) at the time of sample preparation. Internal standards will meet the criteria specified in the corrective action tables.

5.5. Surrogate evaluation

Accuracy and matrix biases for individual samples are monitored for organic analyses using surrogate additions. Surrogates are compounds similar in nature to the target analytes; the surrogates are spiked into environmental samples, blanks, and quality control samples prior to sample preparation for organic analyses. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of

the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results is frequently subjective.

5.6. Laboratory control samples

Laboratory control samples (LCSs) are standard solutions that consist of known concentrations of the complete list of target analytes spiked into laboratory analyte-free matrix. They are prepared or purchased from a certified manufacturer from a source independent from the calibration standards to provide an independent verification of the calibration procedure. These QC samples are then prepared and analyzed following the same procedures employed for environmental sample analysis to assess method accuracy independently of sample matrix effects. The laboratory prepares and analyzes a LCS with each group of twenty samples of similar matrix that are extracted, digested, or analyzed at the same time. Percentage recoveries are evaluated to assess the efficiency of the preparation and analysis method independent of environmental sample matrix effects.

5.7. MS/MSD and laboratory duplicate samples

MS/MSD and laboratory duplicate analyses are performed on environmental samples at a frequency of one per every twenty samples of similar matrix. MS/MSD samples are spiked at the laboratory with the complete list of target analytes. MS/MSD and laboratory duplicate data are generated to evaluate precision and accuracy of the analytical method with respect to sample matrices.

5.8. Analyte identification and quantitation

The objective of the qualitative criteria is to minimize the number of erroneous identifications of compounds. An erroneous identification can either be a false positive (reporting a compound present when it is not) or a false negative (not reporting a compound that is present). The identification criteria can be applied much more easily in detecting false positives than false negatives. Negatives, or non-detected compounds, on the other hand represent an absence of data and are, therefore, much more difficult to assess. The objective for quantitative requirements is to maximize the accuracy of data and sensitivity of the instrument. Unless sample screening indicates the presence of high concentration target analytes, samples are analyzed undiluted to maximize sensitivity. Samples are reanalyzed at the appropriate dilution when concentrations exceed the linear calibration range to maximize accuracy.

In the case of matrix interference, the laboratory performs sample cleanup as provided by the methods. Interferences are identified and documented. Samples are diluted only if analytes of concern generate responses in excess of the linear range of the instrument. When matrix interferences are present, samples are cleaned up during the extraction processes using appropriate methods. The clean-up, extraction and sample preparation methods are listed in the data package case narrative.

5.9. Corrective action

Generally, the following corrective actions are taken by the laboratory. When calibration, instrument performance, and blank criteria are not met, the cause of the problem is located and corrected. The analytical system is then recalibrated. Sample analysis does not begin until calibration, instrument performance, and blank criteria are met. When matrix spike, reference standard, or duplicate analyses

are out of control, the analyses of these samples are investigated. Depending on the results of the overall QC program for the sample set, the data may be accepted, accepted with qualification, or determined to be unusable.

5.10. Preventive maintenance

Preventative maintenance procedures are carried out on laboratory equipment in accordance with the laboratory procedures. Maintenance activities involving are recorded in laboratory documents.

6. Field QA/QC procedures

A brief description of field QA/QC samples is presented in the following sections.

6.1. Field Duplicate Samples

Collection of field duplicate samples provides for the evaluation of the laboratory's precision performance by comparing analytical results of two samples from the same location. They are also collected to evaluate field sample collection precision procedures. Samples are collected from one location and sent to the laboratory blind (with two different sample identifications). Duplicates of aqueous samples are obtained by alternately filling samples containers from the same sampling device for each parameter. Duplicates of aqueous samples submitted for VOC analysis from monitoring wells are filled from the same bailer full of water whenever possible and are the first set of containers filled. Duplicates of solid samples submitted for VOC analysis are obtained from discrete locations without mixing. Duplicates for the remaining analyses require homogenization by filling a decontaminated stainless steel tray or bowl with the sample and mixing it with a decontaminated stainless steel instrument. The mixed sample is divided in half and scooped alternatively from each half to fill the sample container. One field duplicate sample will be collected for every 20 environmental samples (minimum frequency of 5%) or one per matrix for less than 20 samples. If less than 20 samples are collected, one field duplicate sample will be collected.

6.2. MS/MSD and Duplicate Samples

MS/MSD samples are duplicate samples that have spiking solutions added at the laboratory during sample preparation. MS/MSD samples are considered identical to the original sample. The percent recovery of the spiked amount indicates the accuracy of the extraction as well as interferences caused by the matrix. Relative percent differences (RPD) between spike sample recoveries will indicate the precision of the data. Duplicates of aqueous samples are obtained by alternately filling samples containers from the same sampling device for each parameter. One MS/MSD sample set will be collected for every 20 environmental samples submitted to the laboratory (minimum frequency of 5%) or one MS/MSD for less than 20 samples.

For inorganic analyses, duplicate analyses will be performed on environmental samples at a frequency of one per sample matrix and every 20 samples of similar matrix. Duplicate samples will be prepared and analyzed within the same batch as the environmental samples. Duplicate data are generated to determine precision of the analytical method with respect to sample matrices.

6.3. Field Blanks

Field blanks will consist of samples of analyte-free water that are passed through and/or over decontaminated sampling equipment. One field blank will be collected per set of sampling equipment per sampling event. Field blanks will not be required if dedicated sampling equipment is utilized. The field blank samples will be subject to the same analyses as the environmental samples. One field blank will be collected per 10 samples or once per day, whichever is more conservative.

6.4. Trip Blanks

Trip blanks will be prepared as the other preservation containers and will contain analyte-free solvent. The trip blank will undergo shipment from the sampling site to the laboratory in coolers with the environmental samples to be analyzed for VOCs. Trip blanks will be analyzed for VOCs to determine if contamination has taken place during sample handling and/or shipment. Trip blanks will be utilized for samples at a frequency of one each per shipment per cooler sent to the laboratory for VOCs.

6.5. Temperature Blanks

Temperature blanks will consist of vials of water that have undergone shipment from the sampling site to the laboratory in coolers with the environmental samples to be analyzed for the sampling program. The temperature of these blanks will be measured at the laboratory upon receipt of the sample cooler to verify compliance with the cooler temperature requirement.

7. Data Validation and Usability

7.1. Scope of Validation

Data validation will be performed on the data collected for the FSI utilizing the NYSDEC DUSR guidance (NYSDEC 2002). O'Brien & Gere data validators will provide data validation services.

Upon request by the data validator, the laboratory will provide additional or supplemental information within three working days of the request.

7.2. Validation Procedures

Data Validation is a process of determining the suitability of a measurement system for providing useful analytical data. Data validation is essentially a three-step process in which the analytical data's quality assurance/quality control information is first compared to a series of QA/QC criteria. Based on the results of this comparison, the analytical data are then assigned qualifiers, which provide an indication of the data's usability. Finally, an overall evaluation of the data's usability is performed.

Full validation will be performed for the samples collected for each type of analysis for the FSI. Full data validation will consist of a review of data summary forms and supportive raw analytical data that are provided in the data packages.

Evaluation of laboratory data will be performed utilizing the QA/QC criteria established in this QA/QC Plan, as listed in Tables 4-A, 4-B, and 4-C, the analytical methods, and laboratory established control limits.

In accordance with the DUSR process, the following questions will be answered during the validation:

1. Is the data package complete as defined under the project requirements for the NYSDEC ASP Category B?
2. Have the holding times been met?
3. Do all the QC data: blanks, instrument tunings, calibration standards, calibration verifications, surrogate recoveries, spike recoveries, duplicate analyses, laboratory controls and sample data fall within the protocol required limits and specifications?
4. Have the data been generated using established and project-specific protocols?
5. Does an evaluation of the raw data confirm the results provided in the data summary sheets and quality control verification forms?
6. Have the correct data qualifiers been applied?

Data affected by excursions from the previously described QA/QC criteria will be qualified using the following USEPA Region II data validation guidance documents or the most current documents and professional judgment:

- United States Environmental Protection Agency (USEPA). 2006a. *USEPA Region II Evaluation of Metals Data for the CLP Program, SOP HW-2* Revision 13. New York, NY.
- United States Environmental Protection Agency (USEPA). 2006b. *USEPA Region II Validating Semivolatile Organic Compounds by SW-846 Method 8270, SOP HW-22* Revision 3. New York, NY.
- United States Environmental Protection Agency (USEPA). 2006c. *USEPA Region II Validating Volatile Organic Compounds by SW-846 Method 8260B, SOP HW-24* Revision 2. New York, NY.

Since selected USEPA Region II validation guidelines apply to data generated using contract laboratory program (CLP) methods, the application of these validation guidelines will be modified to reflect the QA/QC criteria established in this QCD and the analytical methods, since non-CLP methods will be used in the analysis of samples collected for this project.

Data validators will be responsible for reviewing the QC parameters as listed below. Data validators will recalculate approximately ten percent of the laboratory sample calculations using raw data when verifying sample results for full validation. In addition, data validators will review approximately ten percent of the raw data to verify that compound identification was performed correctly and transcription errors are not present for full validation.

Data quality will be evaluated using current laboratory control limits as provided in the data packages. Sample data will be qualified based on excursions from control limits. Data validators will check corrective action reports and results of reanalysis if available. Corrective actions implemented by the laboratory will be referenced in the data validation report.

Data will be qualified using the following validation approach:

- If percent recoveries are less than laboratory control limits but greater than ten percent (greater than thirty percent for aqueous metals and inorganic parameters), non-detected and detected results are qualified as approximate (UJ, J) to indicate minor excursions.
- If percent recoveries are greater than laboratory control limits, detected results are qualified as approximate (J) to indicate minor excursions.
- If percent recoveries are less than ten percent (less than thirty percent for aqueous metals and inorganic parameters), detected results are qualified as approximate (J) and non-detected results are qualified as rejected (R) to indicate major excursions.
- If relative percent differences (RPDs) for matrix spikes (MSs) and matrix spike duplicates (MSDs) are outside of laboratory control limits, detected results are qualified as approximate (J).
- If RPDs for field duplicates are outside of validation criteria, detected and non-detected results are qualified as approximate (UJ, J).
- For USEPA Method 8260B, volatile organic compound (VOC) target analytes are evaluated using the criteria of 15 percent relative standard deviation (%RSD) or correlation coefficient criteria of 0.990 for initial calibration curves. Calibration verifications are evaluated using a criterion of 20 percent difference (%D) for calibration check compounds and a criterion of 50 %D

for the remaining target analytes. Initial calibrations and calibration verifications are also evaluated using the criterion of a response factor (RF) value of greater than or equal to 0.010 for ketones and alcohols and a value of 0.05 for the remaining target analytes.

- For USEPA Method 8270C, semivolatile organic compound (SVOC) target analytes are evaluated using the criteria of 15 percent %RSD or correlation coefficient criteria of 0.990 for initial calibration curves. Calibration verifications are evaluated using a criterion of 20 %D for calibration check compounds and a criterion of 50 %D for the remaining target analytes. Initial calibrations and calibration verifications are also evaluated using the criterion of a RF value of greater than or equal to 0.05.
- For USEPA Methods 8260B and 8270C, sample result internal standard areas are evaluated using control limits of 50 percent to 200 percent recovery of the areas in the associated calibration verifications.
- The following actions are taken for blank evaluation:
 1. If methylene chloride, acetone or 2-butanone is detected in the sample at a concentration that is less than ten times the concentration in the associated blank, the sample result is identified as non-detected and qualified as “U”.
 2. If other target analytes are detected in the sample at a concentration that is less than five times the concentration detected in the associated blank, the sample result is identified as non-detected and qualified as “U”.
 3. For blank impacted sample concentrations that are less than the QL, the QL is reported and the “U” qualifier is added.
 4. For blank impacted sample concentrations that are greater than the QL, the “U” qualifier is added to the existing sample concentration.
 5. The highest concentrations of the target analytes are used to evaluate the associated samples.
- Qualification of organic data for MS/MSD analyses excursions will be performed only when both MS and MSD percent recoveries are outside of laboratory control limits.
- Organic data will be rejected in the case that both MS/MSD recoveries are less than ten percent.
- Qualification of data will not be performed if MS/MSD or surrogate recoveries are outside of laboratory control limits due to sample dilution.
- In the case that excursions were detected in more than one quality control sample of the same matrix within one sample delivery group, samples will be batched according to collection date and qualified accordingly.
- For organic analyses, qualification of data associated with MS/MSD or field duplicate excursions will be limited to the un-spiked sample or the field duplicate pair, respectively.
- Field duplicate data will be evaluated against relative RPD criteria of less than 100 percent for solid samples and 50 percent for aqueous samples when results are greater than five times the reporting limit. When sample results for field duplicate pairs are less than five times the reporting limit, the data will be evaluated using control limits of plus or minus two times the reporting limit.
- Inorganic laboratory duplicate data will be evaluated against laboratory control limits established for RPD criteria when results are greater than five times the reporting limit. When sample results

for laboratory duplicate pairs are less than five times the reporting limit, the data will be evaluated using control limits of plus or minus two times the reporting limit.

- Serial dilution results will be evaluated by the laboratory for data with initial sample concentrations that are greater than 50 times the IDL. Qualifiers will be applied to data that exceeds the ten percent difference.
- Results for samples submitted for organic analyses impacted by cooler temperatures of greater than 10°C, will be qualified as approximate. Inorganic results will not be qualified for elevated cooler temperatures.
- Results for samples submitted for organic and inorganic analyses that are impacted by percent solids of 50 percent or less, will be qualified as approximate.

In accordance with the USEPA guidance, and utilizing professional judgment, the following qualifiers will be used in the data validation:

"R" Indicates that the reporting limit or sample result is determined to be unusable due to a major deficiency in the data generation process. The data should not be used for any qualitative or quantitative purposes.

"U" Indicates that the analyte was analyzed for, but a concentration was not detected. The sample quantitation limit is presented. This qualifier is also used in the validation process to signify that the detection limit of an analyte was raised due to blank contamination.

"J" Indicates that the concentration should be considered approximate. This qualifier is used when the data validation process identifies a deficiency in the data generation process. This qualifier is also applied by the laboratory for organic analyses when the analyte concentration was greater than the MDL but less than the QL. In the latter case, the identification of the analyte is not in question but the quantitation of the analyte concentration may be uncertain.

"UJ" Indicates that the analyte was analyzed for, but a concentration was not detected. The sample quantitation limit is presented, and should be considered approximate. This qualifier is used when the data validation process identifies a deficiency in the data generation process.

"JN" Indicates that there is presumptive evidence that the analyte is present, but it has not been confirmed due to column confirmation excursions.

The following guidelines will be used regarding the assignment of qualifiers and the evaluation of data:

- The data quality evaluation results in only one type of qualifier ("U", "J", "UJ," or "R") for each analyte; in a case when several qualifiers are applicable to the same analyte, the cumulative effect of the various QA/QC excursions is employed in assigning the final data qualifiers. For example, if a sample result is affected by low surrogate recoveries for which the "UJ" qualifier is applied, but low MS/MSD recoveries result in the rejection of the sample result (application of the "R" qualifier), the final data qualifier is the "R" qualifier.

The following parameters will be included in the review for organic and inorganic analyses for full validation (where applicable):

Analyses for VOCs and SVOCs (where applicable):

1. Chain-of-custody
2. Sample collection and sample preservation
3. Holding times
4. GC/MS tuning criteria
5. Initial calibration and calibration verification
6. Blank analysis
7. Surrogate recovery
8. Matrix spike/matrix spike duplicate (MS/MSD) analysis
9. Field duplicate analysis
10. LCS analysis
11. Internal standards performance
12. Target analyte identification, quantitation, and reported detection limits
13. Documentation completeness
14. QCD compliance

Analysis for metals and cyanide (where applicable):

1. Chain-of-custody issues
2. Sample collection and sample preservation
3. Holding times
4. Calibration analysis
5. Contract required detection limit (CRDL) analysis
6. Blank analysis
7. ICP interference check sample analysis
8. MS/duplicate analysis
9. Field duplicate analysis
10. LCS analysis
11. ICP serial dilution analysis
12. Analyte quantitation, and reported detection limits
13. Documentation completeness
14. QCD compliance

Tentatively identified compounds (TICs) for organic analyses will not be evaluated as part of the validation process.

7.3. Data Usability Evaluation

Based on the QA/QC information review and the qualifiers assigned to the analytical data, an overall evaluation of the data's usability will be performed. Data usability is defined as the percentage of data that remains unqualified or is qualified as approximate or non-detected due to blank contamination, divided by the data reported by the laboratory times 100. The percentage usability excludes the data qualified as rejected due to major QA/QC excursions. The non-usable data are defined as the percentage of the data qualified as rejected divided by the data reported by the laboratory times 100. The data usability will be provided for each the complete data set for this project.

The data usability evaluation considers the data parameters of precision, sensitivity, accuracy, representativeness, comparability, and completeness, which are described as follows:

- Precision is evaluated through the review of field duplicate samples, laboratory duplicates, and MS/MSD samples.
- Sensitivity is evaluated through the review of QLs.
- Accuracy is evaluated through the review of MS/MSD samples, internal standards, surrogate recoveries, LCS recoveries, calibration, instruction performance check, ICP interference check analysis, and ICP serial dilutions.
- Representativeness is evaluated through the review of holding times, sample preservation and preparation, blank analysis and target compound identification and quantification.
- Comparability is evaluated through the review of the analytical methods and reporting procedures for consistency.
- Completeness is defined as the overall percentage of sample results that are determined to be usable.

7.4. Data Usability Summary Report

The DUSR will contain separate QA sections in which data quality information collected during the investigation is summarized. The DUSR will include the following:

- Guidelines used to evaluate the data.
- Data qualifiers applied to sample results.
- Summary of samples collected and analyses performed.
- Narrative that identifies major and minor analysis excursions detected for each parameter evaluated for each analysis.
- Additional issues and information that may be beneficial to the data user are discussed.
- Data summary forms.
- Data usability.

The DUSR will be prepared under the direction of the O'Brien & Gere QA Officer.

References

American Water Works Association (AWWA), American Public Health Association (APHA) and Water Environment Federation (WEF). 1992. *Standard Methods for the Examination of Water and Wastewater*, 18th Edition. Washington, D.C.

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United States Environmental Protection Agency (USEPA). 2004. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update IIIB*. Washington D.C.

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