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Center for Environmental Sciences and Engineering Laboratory Quality Assurance Plan

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Quality Assurance Plan

for

Center for Environmental Sciences and Engineering
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1.1 QUALITY ASSURANCE POLICY STATEMENT

ANALYTICAL PERFORMANCE. For over thirteen years the Center for Environmental Sciences and Engineering (CESE) has been providing research grade analytical chemistry services to our clients in a timeframe that meets their needs. As part of our mission, CESE recognizes the integral part that Quality Assurance plays in providing superior data to our clients. As a result of this, CESE has developed a Quality Assurance Plan to provide the framework by which the laboratories operate.

This Quality Assurance Plan summarizes the policies and operational procedures associated with CESE in Storrs, Connecticut**.** CESE is dedicated to providing high quality data that meet the diverse needs of our clients. The Center for Environmental Sciences and Engineering believes that accurate and precise data depend upon an effective quality system. The goals of this Quality Assurance Plan (QAP) are to detail the underlying quality control/ quality assurance principles and to formalize the structure of the CESE quality assurance program.

Quality Assurance (QA) is an integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the customer.

Quality Control (QC) is the overall system of technical activities that measures the attributes and performance of a process, item or service against defined standards to verify that they meet the stated requirements established by the chosen analytical method or by the directives of the customer.

Specific protocols for sample handling and storage, chain-of-custody, and laboratory analyses, data reduction, corrective action, and reporting are described herein. All policies and procedures have been structured in accordance with applicable EPA, BQ-9000, and Connecticut Department of Public Health requirements, regulations, guidance, and technical standards. This manual has been prepared in accordance with the guidance documents listed in Section 13 of this report. Further details on these policies and procedures are contained in standard operating procedures (SOPs) and related documents.

The Center for Environmental Sciences and Engineering performs physical and chemical analyses for organic and inorganic compounds and other parameters in ambient air, atmospheric deposition, biological tissue, surface water (saline and fresh), ground water, sediment, soil, hazardous waste, and biodiesel/ biodiesel blends. CESE's goal is to produce data that are scientifically valid, defensible, and of known and documented quality in accordance with standards developed by State and Federal regulations or requirements.

CESE has developed a proactive program for prevention and detection of improper or unethical actions. Components of this program include: external proficiency testing (single blind); electronic data audits and post-analysis data review by the appropriate laboratory managers and Laboratory Director.

1.2 PROFICIENCY TEST PROGRAM

The Center for Environmental Sciences and Engineering participates in several proficiency test (PT) programs from the State of Connecticut Department of Public Health and other NIST -approved PT providers for the analytes established by EPA for non potable water and solid/ hazardous waste. The specific analytes and matrices analyzed are based on the current scope of the laboratory services.

CESE annually participates in several laboratory intercomparison exercises encompassing a variety of sample matrices and are not considered substitutes for in-house quality control. Traditionally CESE participates in Chesapeake Biological Laboratory (CBL), National Institute of Standards and Technology (NIST), National Oceanic and Atmospheric Administration (NOAA), and National Research Council – Canada intercomparison programs. These include analyses for nutrients in seawater and inorganics in marine mammal tissue.

The biodiesel testing laboratory participates in the American Society for Testing and Materials (ASTM) biodiesel laboratory crosscheck program for B100.

1.3 REVIEW OF REQUESTS FOR THE ACCEPTANCE OF NEW WORK

The CESE Director and the Laboratory Director are responsible for deciding whether or not to accept new work. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. This evaluation includes a review of facilities and instrumentation, staffing, and any special QC or reporting requirements to ensure that analyses can be performed to meet the goals of the client. Upon accepting the project, the Laboratory Director meets with laboratory managers and support personnel and details all of the project requirements and the projected time frame for completion of the analyses. These procedures are documented in SOP# 01-003-06

All analytical measurements are made using published reference methods or methods developed in-house by CESE.

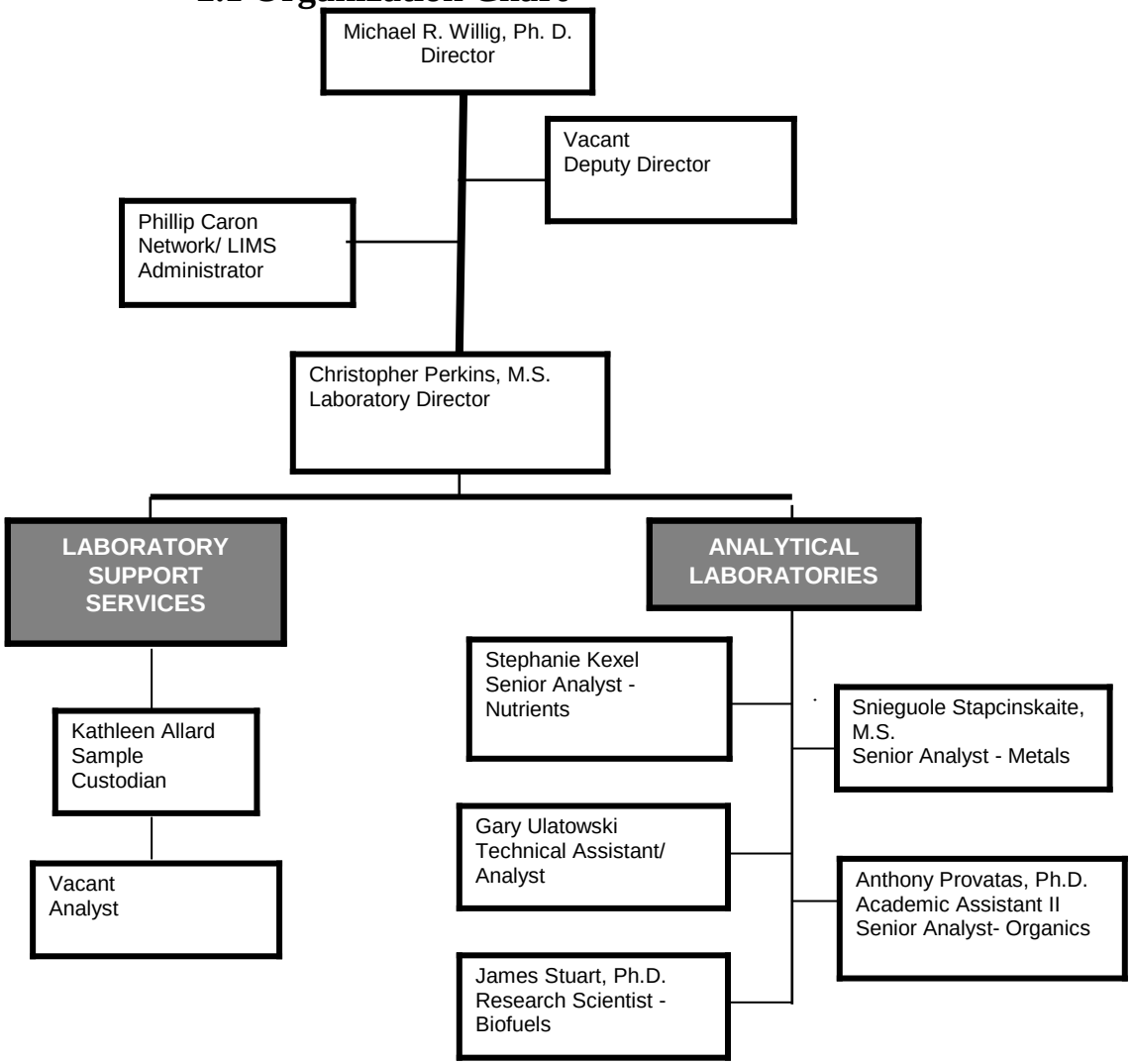
1.4 REFERENCES

Where applicable, CESE shall institute the newest version of the quality standards and analytical methods and shall be instituted within 90 days of the latest effective date. This QAP references these policies and procedures:

- USEPA Quality manual for Environmental Programs, CIO 2105-P-01-0, Effective Date 5/5/00.
- National Biodiesel Accreditation Commission, BQ-9000, Effective Date 3/29/09 (http://www.bq-9000.org/documents/) .

2.0 ORGANIZATION AND RESPONSIBILITIES

2.1 Organization Chart



2.2 Laboratory Director

The Laboratory Director reports to the Center Director. The Laboratory Director has the ultimate responsibility for the quality of the data generated in the analytical laboratories, to include laboratory data, reports, and safety. The Director serves as the focal point for QA/QC and is responsible for the oversight and/or review of quality control data. The officer is responsible for auditing the implementation of the Quality System.

The Laboratory Director is responsible for:

- Ensuring that all technical laboratory staff have demonstrated proficiency in the activities for which they are responsible;
- Ensuring that the training of its personnel is kept up-to-date, including QA/QC training for Lab Analysts;
- Documenting all analytical and operational activities;
- Supervising all personnel;
- Ensuring that all sample acceptance criteria are verified and that samples are logged into the sample tracking system and properly labeled and stored;
- Documenting the quality of all data reported by the laboratory;
- Ensuring that the laboratory has the appropriate resources and facilities to perform requested work;
- Developing a proactive program for prevention and detection of improper, unethical or illegal actions;
- Conducting internal audits on the entire technical operation annually and chair the quality management review meetings, where applicable;
- Responding to external auditor comments and implementing corrective actions, as required;
- Auditing data packages for QC compliance;
- Scheduling internal PE and other QA/QC samples (MDL, Precision-Accuracy, etc.);
- Managing and maintaining all QA/QC documentation including Lab Certifications, SOPs, QAPs, MDL studies, Precision-Accuracy studies, and reports of PE samples and intercomparisons.

2.3 Technical Staff

Technical staff are responsible for sample analysis and identification of corrective actions. The staff report directly to the Laboratory Director. All personnel are responsible for complying with all quality assurance/ quality control (QA/ QC) requirements that pertain to their organizational/technical function. As documented in the employee records, each technical staff member has the experience and education to adequately demonstrate knowledge of their particular function and a general knowledge of laboratory operations, analytical test methods, quality assurance/quality control procedures and records management.

2.3.1 Senior Analyst/ Project Scientist

The Senior Analyst/ Project Scientist is responsible for:

- Scheduling all analytical work performed in the laboratory;
- Assigning work on a daily basis to Analysts and Laboratory Assistants;
- Overseeing employee training on sample preparation techniques and analyses, ensures, that training protocols are up-to-date, and review's the work of analytical laboratory staff members during training periods;
- Ensuring and documents initial and on-going Analyst and Laboratory Assistant proficiency;
- Acting as a lead, answering procedural or analytical questions, and providing corrective action recommendations;
- Ensuring that SOPs are being followed, or for updating SOPs to reflect the current methodologies;
- Ensuring that instrument maintenance is being performed, and coordinates troubleshooting;
- Participating in the development and improvement of methods to improve efficiency;
- Providing good scientific insight regarding analytical issues ;
- Conducting employee performance reviews;
- Ensuring that an adequate supply of all laboratory materials is always on hand;
- Maintaining and promoting a high level of safety and cleanliness in the laboratories, in cooperation with the Health & Safety Officer;
- Reporting health and safety issues/ problems to the Health and Safety Officer;
- Performing all Analyst duties;
- Following procedures described in the QAP and all applicable SOPs;
- Performing any other reasonable tasks assigned by the Laboratory Manager.

2.3.2 Sample Custodian

The Sample Custodian reports to the Laboratory Director and is responsible for:

- Acting as a client lead, coordinating sample login, answering protocol questions, and providing corrective action recommendations;
- Ensuring that sample documentation, receipt, and storage SOPs are being followed, or updating SOPs to reflect the current methodologies;
- Reviewing all Chain of Custodies (COCs) for accuracy and coordinates with clients to resolve discrepancies;
- Participating in the development and improvement of methods to promote efficiency;
- Reporting health and safety issues/ problems to the Health & Safety Officer;
- Following procedures described in the Laboratory QAP and all applicable QAPPs and SOPs.

2.3.3 Analyst/ Assistant Research Scientist

The Analyst/ Assistant Research Scientist is responsible for:

- Preparing and analyzing samples, calculating and evaluating all data, entering data into the reports, and submitting raw data (fully calculated) for review;
- Performing any necessary corrections to datasets in a timely manner;
- Reviewing all COC forms and project sheets to be sure the samples are being analyzed correctly and the proper QC is being performed;
- Working to ensure the generation of high quality data ;

- Performing instrument maintenance;
- Keeping the lab clean, always cleaning an area of the lab after using it;
- Following procedures described in the QAP and all applicable SOPs.

2.3.4 NETWORK/ LIMS Administrator

The LIMS Administrator reports to the Center Director and is responsible for:

- Managing the day-to-day operations of the LIMS;
- Training and supervising students and technical staff working with the LIMS;
- Maintaining a current inventory of hardware, software, documentation, supplies, and other equipment needed for the operation of the LIMS;
- Checking LIMS hardware and software for damages/ defects, troubleshooting problems and arranging for repair or replacement;
- Performing minor maintenance and repair to the network infrastructure;
- Providing for orientation of all users to certify in LIMS procedures;
- Serving as primary technical resource for LIMS users;
- Developing, implementing, and maintaining policies, procedures, and SOPs for use of LIMS;
- Customizing the LIMS tests, interface, and reporting as directed.

2.4 TRAINING

Staff members are trained in new methods utilizing a mentoring process. New staff members (or staff members learning new methodologies) are assigned to their immediate supervisor or an experienced staff member. Each employee has read, understood, and is using the latest version of the laboratory's relevant SOPs, which relates to the new methodology as well as the associated reference material. The development and training associated with new analytical methods is discussed in section 7.3.

The mentoring process occurs as follows:

- The staff member is familiarized with the method by observation of an experienced staff member;
- Supervised practice of the method, with review by trainer and supervisor;
- Unsupervised practice of the method, with review, this may include a blind spike sample or other blind unknown;
- Unsupervised performance of the method.

Completion of these steps is documented on a training checklist signed by the supervisor of the laboratory. For newly trained employees, work is reviewed daily by supervisor until the supervisor is satisfied the employee is competent in the procedure.

Each employee demonstrates continued proficiency by acceptable performance on Laboratory Control Sample (LCS), blind samples and inter-laboratory intercomparison exercises. Training records (e.g., continuing education, participation in technical conferences, internal training activities) are kept in the Administrative Office.

2.5 LABORATORY CAPABILITIES

CESE's state-of-the-art laboratories provide a full range of analytical and engineering development and testing services to support faculty research and to address the research needs of government and industry. The labs, which include sections for organics, trace metal, low level mercury, Biofuel, and nutrient analysis, occupy more than 9,300 square feet of space and are equipped with advanced instrumentation and computers. Samples and associated data are tracked and stored in the CESE LIMS. A complete list of instrumentation can be found in Appendix B.

Inorganic Analysis

The inorganic laboratory at CESE can be broken down into two main divisions. The first of these is the trace metals laboratory, which is divided into instrumentation, trace level mercury and preparation laboratories. The second inorganic division is the nutrients laboratory, which is also divided into instrumentation and wet chemistry laboratories.

Trace Metals

The trace metals division is equipped with an Inductively Coupled Plasma/Mass Spectrometer (ICP/ MS), an ICP/ Optical Emission Spectrometer (ICP/ OES), a Cold-Vapor Atomic Absorption Spectrophotometer (CVAAS), and a combustion AA (DMA-80). Additionally, the trace metals laboratory is equipped with a cold vapor and GC atomic fluorescence systems for ultra-trace mercury analysis, which are housed in a clean room. The preparation laboratory is equipped a microwave digestion apparatus, and two four-foot hoods to meet the needs of the most rigorous and varied inorganic testing requirements. The laboratory also has the capacity to perform EPA-required batch extraction procedures using a high capacity 16-place rotary extractor.

Nutrients

The nutrients division is equipped with one continuous flow auto analyzer for determination of the silica, nitrogen and phosphorus series as well as other ion analyses. The laboratory also includes a Total Organic Carbon (TOC) analyzers and a Carbon, Hydrogen, and Nitrogen (CHN) analyzer to complete the carbon and nitrogen series. An ion chromatograph is used to determine anions in water. Also available is an Ultra-Violet/Visible (UV/ VIS) spectrometer and fluorometer to complete additional tests requested. The UV/ VIS spectrometer serves as a backup instrument in case of failure the auto analyzer. The laboratory is equipped with a walk-in refrigeration unit and one walk-in incubator for determining BOD. The laboratory also includes an eight-foot fume hood and bench space to perform all operations necessary for the testing of nutrients and for the performance of wet chemistries. The wet chemistry laboratory houses the fluorometer, ovens and filtration apparatuses for solids analysis.

Organic Analysis

The organics division is equipped with a state-of-the-art Waters ACQUITY ultra performance liquid chromatograph/ tandem mass spectrometer (UPLC/MS/MS) to support the rigorous analytical needs of cutting-edge environmental research. The instrumentation also includes a Waters Quattro ultra-micro gas chromatograph/ tandem mass spectrometer (GC/MS/MS) and 2 GC/ mass selective detectors (MSD) in standard configurations and with a purge and trap sample introduction module for the analysis of volatile and semi volatile compounds in a variety of matrices. Additionally, CESE has several GCs with electron capture, flame ionization, and nitrogen/phosphorus detectors. The preparation lab includes automated extraction and clean-up instrumentation, including a Genevac

automated concentrator, an automated solvent extractor (ASE), 2 TurboVap concentrators, and a nitrogen blow-down unit (N-VAP).

Biofuel Analysis

The biodiesel laboratory is equipped with one energy dispersive x-ray fluorescence (ED-XRF) spectrometer for the analysis of sulfur and other trace elements, as well as an inductively coupled plasma – optical emission spectrometer (ICP-OES) for calcium, magnesium, phosphorus, potassium, and sodium analysis. The laboratory also includes one gas chromatograph – flame ionization detector (GC-FID) for the analysis of free and total glycerin and a static head-space GC-mass spectral detector (MSD) for methanol analysis. Additional equipment include a Metrohm Rancimat, for oxidation stability, a total acid number titrator, a Phase Technology's cloud point instrument, a Pensky-Martens closed cup flash point tester, and a Kohler distillation apparatus. The laboratory also has a centrifuge for water and sediment analysis, a water bath for copper strip corrosion, and cold soak filtration apparatus. To analyze for biodiesel content, CESE has an A2 Technologies PAL Fourier transform infared spectrometer (FT-IR).

3.0 QUALITY ASSURANCE OBJECTIVES

3.1 DATA QUALITY OBJECTIVES

The overall QA objective for the Center for Environmental Sciences and Engineering Laboratories is to develop and implement procedures for laboratory analysis, chain-of-custody, and reporting that will provide results that are of known and documented quality. Data Quality Objectives (DQOs) are used as qualitative and quantitative descriptors in interpreting the degree of acceptability or utility of data. The principal DQOs are precision, accuracy (bias), representativeness, comparability, completeness and detection limits. DQOs are used as quantitative goals for the quality of data generated in the analytical measurement process. This section summarizes how specific QA objectives are achieved. The specific application of these various activities are contained in the applicable method SOPs.

Precision
Precision is a measure of the degree to which two or more measurements are in agreement.

Precision is assessed through the calculation of relative percent difference (RPD) and relative standard deviation (RSD) for replicate samples. For inorganic and organic analyses, laboratory precision is assessed through the analysis of a sample/ sample duplicate pair and field duplicate pairs.

Accuracy
Accuracy is the degree of agreement between an observed value of a target analyte and an accepted reference or true value.

Laboratory accuracy is determined through the analysis of MS/ MSD, quality control check samples, and laboratory control samples (LCS). Accuracy is further assessed by the analysis of blanks and through the adherence to all sample handling, preservation and holding times.

Representativeness
Representativeness expresses the degree to which data accurately and precisely represent a condition of an entire sample population or an environmental condition within a defined spatial and/ or temporal boundary.

Representativeness is ensured by using the proper analytical procedures, appropriate methods, meeting sample holding times and analyzing field duplicate samples. It is also ensured by the use of appropriate sample homogenization procedures by the laboratory and “clean” sampling techniques by the client.

Completeness
Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The acceptable level for laboratory completeness is 95% usable data.

Comparability
Comparability is an expression of the confidence with which one data set can be compared to another. It examines how variable one data set is to another for ongoing projects.

Comparability is achieved by the use of routine analytical methods, achieving holding times, reporting results in common units, use of consistent detection levels, and consistent rules for reporting data.

Detection Limits

CESE utilizes four types of detection limits: Method detection limit (MDL); Practical quantitation limit (PQL); Limit of detection (LOD); and Contract required quantitation limit (CRQL).

Method Detection Limits (MDLs) in the nutrients, organics, and biofuel divisions are determined for all analytes and methods, where appropriate. The MDL is defined as the minimum concentration of an analyte that can be measured by the method with 99% confidence of its presence in the sample matrix. The MDL has been determined for each test and matrix according to USEPA requirements as part of an initial demonstration of capability. MDLs are determined on an annual basis for each method and sample matrix.

The Practical Quantitation Limit (PQL) is defined in the laboratories as the lowest standard of the initial calibration curve, and is usually between 3 to 10 times the MDL. It is confirmed by running a laboratory control sample spiked at this level with an expected recovery that is dependent upon the analytical method.

Limit of Detection (LOD) is defined in the laboratories as an estimate of the minimum amount of a substance that an analytical process can reliably detect – it can be detected with 99% confidence or produce a signal at least 3x instrument noise level. LOD are similar to the MDL in the nutrients laboratory – estimated as 1/5 to 1/3 of the PQL.

The contract required quantitation limit (CRQL) is defined by the client.

CESE will use the PQL or the LOD as the default reporting limit for all clients who do not have specific reporting limit requirements. CESE will use a contractually generated quantitation limit for clients with specific requirements.

Reported data are flagged (i.e. italicized) when they fall between the MDL/ LOD and the PQL. At no time will data be reported below the MDL/ LOD.

The MDL, LOD and PQL, LOQ study data are compiled by the Laboratory Director and are available from CESE upon request.

4.0 SAMPLE HANDLING

4.1 SAMPLE TRACKING

CESE utilizes several layers of documentation to track samples all the way from sample receipt through analysis. Information regarding every sample received by CESE is entered on to a chain of custody (COC). This information is then entered into CESE's Laboratory Information Management System (LIMS). CESE utilizes the Samplemaster® LIMS, which uniquely identifies each sample to be tested, to ensure that there can be no confusion regarding identity. The sample identification system includes identification for all samples. A unique identification (ID) code is placed on each sample container and the COC. Other forms of documentation used by CESE to track samples include: project information sheets, laboratory bench sheets and notebooks, raw instrument data and final analytical reports.

4.2 SAMPLE ACCEPTANCE POLICY

CESE has a written sample acceptance policy that outlines the circumstances under which samples will be accepted. Samples that do not meet the policy are noted in the analytical data report defining the nature and substance of the variation. If no COC is included with the samples, the Laboratory Director will be notified and the Sample Control Officer will attempt to contact the client. If the client did not generate a COC, the Sample Control Officer will generate an internal COC, with the approval of the client. If the enclosed COC contains errors and the client is unable to be reached, the Laboratory Director will decide on the process by which sample receipt will proceed. Refer to SOP# 01-003-06 for more detailed procedures.

It is the responsibility of the client to provide a complete and accurate COC with each batch of samples. The policy requires or establishes:

- Proper, full, and complete documentation, including the sample identification, the location, date and time of collection, collector's name, preservation type, required turn around time, sample type, analytical tests, and any special remarks concerning the sample;
- Unique identification of samples using durable labels completed in indelible ink;
- Use of appropriate sample containers;
- Receipt within holding times;
- Adequate sample volume;
- Special instructions for unused sample disposal, if applicable.

If, upon receipt of the package, the contents create an unacceptable safety hazard, the package will be resealed, placed in a secure storage area and the client will be notified.

4.3 SAMPLE RECEIPT PROTOCOLS

Upon delivery to the sample receiving area, the condition of the sample, including any abnormalities or departures from standard condition is recorded. The Sample Custodian breaks the custody seal (if applicable) and opens the shipping container. All samples that require thermal preservation have temperature recorded on the sample receipt checklist and are considered acceptable if the arrival temperature is within +/- 2 °C of the required temperature. For samples with a specified temperature of 4 °C, samples with a temperature ranging from just above the freezing temperature of water to 6 °C are considered acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet this criterion. In this case, the samples will be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice. Where applicable, CESE verifies chemical preservation by monitoring pH upon receipt. The general condition of the samples is noted and the samples are compared to the COC for discrepancies. Any discrepancies are noted on the COC and sample receipt checklist (CESE Form 04-001). If the Sample Control Officer is unable to reach the client, the Laboratory Director will decide whether to assume responsibility for the shipment. If the COC contains numerous discrepancies, does not list requested analysis, or is unclear, and the client cannot be reached, it will be the decision of the Laboratory Director as how best to proceed. Once the Sample Control Officer verifies sample integrity, the COC is signed, and sample information is entered into the Laboratory Information Management System (LIMS). If no COC is provided, the Laboratory Director is notified, and the Sample Custodian will attempt to notify the client. If the client is reached but unable to fax a copy of the COC, or if no COC has been generated, an internal COC will be generated by the sample custodian under the direction of the Laboratory Director and or the Laboratory Director with the client's approval. Refer to SOP# 04-001-05 for more detailed procedures.

4.4 SAMPLE STORAGE CONDITIONS

CESE has developed procedures to ensure the probability that any analytes that are originally present in the sample will not be degraded or amplified in concentration. This is also to ensure that the sample storage environment will not affect the sample through the addition of any contaminants not originally present.

Samples which require thermal preservation are stored under refrigeration with a specified storage temperature of 4 °C (temperatures above the freezing point of water to 6 °C is considered acceptable). Tissue samples will be preserved by freezing to -20 °C or less. Each sample container should be tightly sealed and affixed with a unique identification number. All employees have access to CESE's sample storage area.

Temperature is monitored and recorded regularly at each sample storage location, refrigerator or freezer, using a NIST traceable thermometer. Refer to SOP# 04-002-05 for more detailed procedures.

4.5 CHAIN OF CUSTODY (COC)

Clients may specially request an internal tracking COC form. Chain of custody records are used to establish an intact, continuous record of the physical possession, storage and disposal of collected samples, aliquots, extracts, and digestates. The COC records account for all time periods associated with the samples. The COC records identify all individuals who physically handled individual samples, up to sample receipt. The COC forms remain with the samples during transport or shipment. If shipping containers and/ or individual sample containers are submitted with sample custody seals, and any seals are not intact, the lab shall note this on the chain of custody.

Samples are stored in the central walk-in cooler, unless specialized requirements are required which is documented in the LIMS during the sample login process. Access to all samples and sub-samples is controlled during non-working hours. The laboratory area is maintained securely and is restricted to authorized personnel only. Samples remain in the designated area until client acceptance of the final report, or in a timeframe agreed upon by the client and the Laboratory Director.

It is not the standard practice at CESE to generate internal tracking COC except at client request.

4.6 SAMPLE DISPOSAL

Sample disposal is necessary to ensure adequate laboratory sample storage space. All samples are held 30 days after client acceptance of the final report. Clients may request a longer sample retention time, at the discretion of the Laboratory Director.

All samples, digestate, leachates and extracts or other sample preparation products are disposed of according to the Chemical Hygiene Plan of the University of Connecticut (http://www.ehs.uconn.edu/Chemical/chemplan.php), which is in accordance with Federal and State laws and regulations. All hazardous waste is appropriately disposed through the University hazardous waste disposal facility.

CESE reserves the right to refuse the acceptance of samples that may create an unacceptable safety hazard. If this is the case, the package will be resealed, placed in a secure storage area, and the client will be notified.

5.0 CALIBRATION PROCEDURES AND FREQUENCY

5.1 TRACEABILITY OF CALIBRATION

All calibrations and working standards are documented in laboratory logbooks and traceable to certified standards or manufacturer lot number. Certificates attesting to the concentrations are stored in the appropriate laboratory manager's office.

Analytical support equipment includes: balances, refrigerators, freezers, incubators, water baths, temperature measuring devices and volumetric dispensing devices. All such support equipment is:

- Maintained in proper working order. The records of all activities including service calls are kept.
- Calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration are within the specifications required of the application for which is equipment is used or the equipment is removed from service until repaired.

Prior to use, balances, ovens, refrigerators, freezers, incubators and water baths are checked daily with NIST traceable references (where possible) in the expected use range. The acceptability for use or continued use is according to the needs of the analysis or application for which the equipment is being used. Mechanical volumetric dispensing devices (except Class A glassware) are checked for accuracy annually.

Reference standards of measurement (such as Class S or equivalent weights or traceable thermometers) are used for calibration only. Reference standards can be subjected to in-service checks between calibrations and verifications.

Each calibration is dated and labeled with or traceable to the method, instrument, analysis date, and each analyte name, concentration and response (or response factor). Sufficient information is recorded to permit reconstruction of the calibration. Acceptance criteria for calibrations comply with method requirements or are established and documented.

5.2 INSTRUMENT CALIBRATION

Calibration procedures for a specific laboratory instrument will consist of an initial calibration or initial calibration verification when an initial instrument calibration is not performed on the day of analysis. All standards are traceable to certified standards or manufacturer lot number. The SOP for each analysis performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria and the conditions that will require recalibration. In all cases, the initial calibration is verified using an independently prepared calibration verification solution. CESE maintains logbooks which contain the following information: instrument identification, date of calibration, analyst, calibration solutions run and the samples associated with these calibrations.

All results are calculated based on the response curve from the initial calibration and are bracketed by calibration standards or reported as having a lower confidence level.

If the initial calibration fails, the analysis procedure is stopped and evaluated. For example, a second standard may be analyzed and evaluated or a new initial calibration curve may be established and verified. In all cases, the initial calibration must be acceptable before analyzing any samples. If this

does not correct the out of calibration specification, the instrument is tagged as “Out of Calibration”: until placed back in effective service.

6.0 TEST METHODS AND STANDARD OPERATING PROCEDURES

6.1 STANDARD OPERATING PROCEDURES

CESE maintains Standard Operating Procedures (SOPs) that accurately reflect all laboratory activities. These SOPs provide detailed information to personnel on the performance of their work. Copies of all SOPs are accessible to all personnel. Each SOP indicates the effective date and the revision number. In addition to the standard suite of analyses performed, CESE is extremely proficient in method development and can design analytical methodologies that can meet quality control, matrix, and analyte specific requirements. SOP status and revisions are tracked in the SOP Index (Form 02-003-001). Note that the master SOP is controlled by the Laboratory Director and housed on the CESE network (in Laboratory Administration Folder) and all printed copies are considered uncontrolled. All updates will be placed on the CESE network, as above.

SOPs are used to ensure consistency and to save time and effort. Any deviation from an established procedure during an analysis is documented. All SOPs are reviewed on a yearly basis under the direction of the Laboratory Director. In addition, CESE periodically reviews and updates analytical SOPs to incorporate advances in instrumentation/ technology and analytical chemistry research. The SOPs are developed according to the following documents.

- Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality Related Documents, EPA QA/G-6, November 1995;
- Good Laboratory Practices Standards, 40 CFR Part 160 (FIFRA); and
- Specification and Guidelines for Quality System for Environmental Data Collection and Environmental Technology Program, ANSI/ASQC E4-1994, January 1995

7.0 INTERNAL QUALITY CONTROL CHECKS

7.1 LABORATORY QUALITY CONTROL SAMPLES

CESE uses quality control samples to determine the validity of analytical data generated in the laboratories. Quality control samples may include but are not limited to method blanks, initial and continuing calibration verification standards, laboratory control samples, matrix spike and matrix spike duplicates and laboratory duplicates. Quality control samples are treated in the same manner as externally generated field samples and are analyzed at a frequency described in the QAP, the individual SOP's, quality assurance project plan or in the contract with the client. The data acquired from QC procedures are used to estimate the quality of analytical data, to determine the need for corrective action in response to identified deficiencies, and to interpret results after corrective action procedures are implemented. Each method SOP includes a QC section that addresses the minimum QC requirements for the procedure. The internal QC checks may differ slightly for each individual procedure, but in general are described and detailed below. If the quality control sample results fall within the acceptance criteria detailed in the SOP, QAP, QAPP or as prescribed by client contract, then the analytical data are considered valid or acceptable. The project manager performs a scientific review of the data for final validation. Unless specified otherwise by the client, the acceptance criteria for QC sample data are specified in each analytical SOP.

Non-compliant results on field quality control samples do not necessarily reflect an analytical problem and may lead to corrective action, if warranted. Unless CESE staff collect samples, the Center will not be responsible for poor results on field duplicates, field blanks, field spikes, etc. if laboratory QC results are acceptable. Additionally, field quality control data that is non-compliant are not flagged or interpreted by CESE unless agreed to in the QAPP or client contract.

Method Blanks
The method blank is an internally generated sample of reagent grade water or analytical reagents that is treated in the same manner as the corresponding field samples. Method blanks are performed at a frequency of 1 per batch of 20 or fewer samples per matrix type per sample extraction or preparation test method. The results of these samples are used to determine analytical batch acceptance.

Laboratory Control Sample
Laboratory control samples (LCS or QC Check Sample) are analyzed at a minimum of 1 per batch of 20 or fewer samples per matrix type per sample extraction or preparation method. The results of these samples are used to determine analytical batch acceptance.

Matrix Spike
The purpose of matrix spikes (MS) are to determine method performance in a specific type of matrix. MSs are performed at a frequency of one in 20 samples per matrix type per sample preparation method. The percent recovery for the spiking compounds is calculated. Poor performance in a matrix spike generally indicates a problem with the sample composition, and not the laboratory analysis, and is reported to assist in data assessment.

Initial and Continuing Calibration Verification

Initial calibration verification (ICV) and continuing calibration verification (CCV) are used to demonstrate the validly of the calibration curve and working standards used to create it. The ICV is composed of a second source standard and the CCV can be either second source or same mid-point standard as calibration. ICV/ CCVs are utilized at the start of an analytical run, at least every 20 samples, and at the end of the analytical day. The results of these samples are used to determine analytical batch acceptance.

Initial and Continuing Calibration Blanks

Initial calibration blanks (ICB) and continuing calibration blanks (CCB) are used to determine if system contamination, carryover, or baseline drift had occurred during an analytical run. An ICB is run following the ICV and a CCB is run after each CCV. The results of these samples are used to determine analytical batch acceptance.

Laboratory Duplicates

Duplicate analyses are performed to evaluate the precision of the method. Results of the duplicate analyses are used to determine the relative percent difference (RPD) between replicate samples. Laboratory Duplicates are analyzed as part of an analytical run at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation test method. Poor performance in the duplicates generally indicates a problem with the sample composition and homogeneity and is reported to assist in data assessment.

7.2 INSTRUMENT SPECIFIC QUALITY CONTROL CHECKS

Analysis Group	QC Check	Frequency Performed	Quality Assurance Target [a]	Range
ICP-OES	Instrument Blank	Initial, every run		
	Initial Calibration Blank (ICB)	Initial, every run		
	Initial Calibration Verification (ICV)	≥ 5% of samples	A	mid-high
	Interference Check Sample (ICS)	Initial, every run		high
	Continuing Calibration Blank (CCB)	≥ 10% of samples		
	Continuing Calibration Verification (CCV)	≥ 10% of samples	A	mid-high
	Preparation Blank	≥ 5% of samples		
	Laboratory Control Sample (LCS)	≥ 5% of samples	A	mid
	Duplicate Sample	≥ 5% of samples	P	
	Matrix Spike (SPK)	≥ 5% of samples	A	mid
	Matrix Spike Duplicate	per client request	A, P	mid
	Serial Dilution	≥ 5% of samples or every analysis set	A	
	Post Digestion Spike	≥ 5% of samples	A	mid
	QC Check Sample (PE)	≥ Once annually	A	low-high
GFAAS	Instrument Blank	Initial, every run		
	Initial Calibration Blank (ICB)	Initial, every run		
	Initial Calibration Verification (ICV)	≥ 5% of samples	A	mid-high
	Continuing Calibration Blank (CCB)	≥ 10% of samples		
	Continuing Calibration Verification (CCV)	≥ 10% of samples	A	mid-high
	Preparation Blank	≥ 5% of samples		
	Laboratory Control Sample (LCS)	≥ 5% of samples	A	mid
	Duplicate Sample	≥ 5% of samples	P	
	Matrix Spike (SPK)	≥ 5% of samples	A	mid
	Matrix Spike Duplicate	per client request	A, P	mid
	Post Digestion Spike	≥ 20% of samples	A	mid
	QC Check Sample (PE)	≥ Once annually	A	low-high

[a] A = accuracy; P = precision

Analysis Group	QC Check	Frequency Performed	Quality Assurance Target	Range
CVAA, Hg	Instrument Blank	Initial, every run		
	Initial Calibration Blank (ICB)	Initial, every run		
	Initial Calibration Verification (ICV)	≥ 5% of samples	A	mid-high
	Continuing Calibration Blank (CCB)	≥ 10% of samples		
	Continuing Calibration Verification (CCV)	≥ 10% of samples	A	mid-high
	Preparation Blank	≥ 5% of samples		
	Laboratory Control Sample (LCS)	≥ 5% of samples	A	mid
	Duplicate Sample	≥ 5% of samples	P	
	Matrix Spike (SPK)	≥ 5% of samples	A	mid
	Matrix Spike Duplicate	per client request	A, P	mid
	Post Digestion Spike	≥ 5% of samples	A	mid
	QC Check Sample (PE)	≥ Once annually	A	low-high
ICP-MS	Instrument Blank	Initial, every run		
	Initial Calibration Blank (ICB)	Initial, every run		
	Initial Calibration Verification (ICV)	≥ 5% of samples	A	mid-high
	Interference Check Sample (ICS)	Initial, every run		high
	Continuing Calibration Blank (CCB)	≥ 10% of samples		
	Continuing Calibration Verification (CCV)	≥ 10% of samples	A	mid-high
	Preparation Blank	≥ 5% of samples		
	Laboratory Control Sample (LCS)	≥ 5% of samples	A	mid
	Duplicate Sample	≥ 5% of samples	P	
	Matrix Spike (SPK)	≥ 5% of samples	A	mid
	Matrix Spike Duplicate	per client request	A, P	mid
	Post Digestion Spike	≥ 5% of samples	A	mid
	QC Check Sample (PE)	≥ Once annually	A	low-high
Flow Analyzer	Initial and Continuing Calibration Blank	Initial & every 10 samples		
	Initial and Continuing Calibration Verification	Initial & every 10 samples	A	mid
	Matrix Spike	Every 10 samples	A	mid
	Duplicate Sample	Every 10 samples	P	
Chromatography	Duplicate Sample	Every 10 samples	P	
	Initial and Continuing Calibration Verification	Initial & every 10 samples	A	mid
	Matrix Spike	Every 10 samples	A	mid
	Duplicate Sample	Every 10 samples	P	

Analysis Group	QC Check	Frequency Performed	Quality Assurance Target	Range
Total Organic Carbon (TOC)	Initial and Continuing Calibration Blank	Initial & every 10 samples		
	Initial and Continuing Calibration Verification	Initial & every 10 samples	A	
	Matrix Spike	Every 10 samples	A	
	Duplicate Sample	Every 10 samples	P	
Carbon/ Hydrogen/ Nitrogen (CHN)	Initial and Continuing Calibration Blank	Initial & every 10 samples		
	Initial and Continuing Calibration Verification	Initial & every 10 samples	A	
GC/MS/MS	Instrument Blank	Initial, every run		
	Initial Calibration Blank (ICB)	Initial, every run		
	Initial Calibration Verification (ICV)	≥ 5% of samples	A	mid-high
	Continuing Calibration Blank (CCB)	≥ 10% of samples		
	Continuing Calibration Verification (CCV)	≥ 10% of samples	A	mid-high
	Preparation Blank	≥ 5% of samples		
	Laboratory Control Sample (LCS)	≥ 5% of samples	A	mid
	Duplicate Sample	≥ 5% of samples	P	
	Matrix Spike (SPK)	≥ 5% of samples	A	mid
	Matrix Spike Duplicate	per client request	A, P	mid
	Standard Reference Material (SRM)	≥ 5% of samples	A, P	
UPLC/MS/MS	Instrument Blank	Initial, every run		
	Initial Calibration Blank (ICB)	Initial, every run		
	Initial Calibration Verification (ICV)	≥ 5% of samples	A	mid-high
	Continuing Calibration Blank (CCB)	≥ 10% of samples		
	Continuing Calibration Verification (CCV)	≥ 10% of samples	A	mid-high
	Preparation Blank	≥ 5% of samples		
	Laboratory Control Sample (LCS)	≥ 5% of samples	A	mid
	Duplicate Sample	≥ 5% of samples	P	
	Matrix Spike (SPK)	≥ 5% of samples	A	mid
	Matrix Spike Duplicate	per client request	A, P	mid
	Standard Reference Material (SRM)	≥ 5% of samples	A, P	

7.2 INSTRUMENT CALIBRATIONS

With a new run for each instrument, a new calibration is performed. The acceptance criteria for an acceptable curve will have a correlation coefficient >0.995. Data may still be reported if below this limit, however this must be noted to the client.

In the nutrients laboratory with the flow injection analyzers it may be determined that a point on a calibration curve is an outlier. Although this is a rare occasion, it may be determined that a point should be deleted from the calibration curve. Only one point on one calibration curve may be deleted, and once the point is deleted, the correlation coefficient must yield >0.997 or greater. When this occurs, the deleted standard is remade and rerun at the end of the analysis to verify that the standard was made incorrectly, or instrument problems precluded an accurate sample analysis.

The UPLC/MS/MS, GC/MS/MS, Shimadzu Total Organic Carbon analyzer, Dionex Ion Chromatograph, Turbidimeter, alkalinity titrator, and the Perkin Elmer CHN analyzer will hold their calibrations over a period of time and do not need to be re-calibrated every day. Once the start-up procedure has been performed, a second source QC sample is run to determine if the instrument is still within instrument specific acceptable limits (e.g. 85-115% recovery). If the QC fails, a new calibration must be performed or maintenance may be needed on the instrument. Data may still be reported if below this limit, however issues must be noted to the client.

Refer to each applicable SOP for any method specific requirements.

7.3 METHOD DETECTION LIMIT STUDIES

Method Detection Limits (MDLs) are set such that the risk of reporting a false positive is less than 1%. MDL studies are part of our initial demonstration of capability and are performed on an annual basis for most analytes and matrices as described in by 40 CFR Part 136, Appendix A. The method detection limit is determined for the compounds of interest in each method in laboratory reagent water or Ottawa sand. Seven or more low-level (3 to 5 times the anticipated MDL) spikes are processed exactly like samples. The MDL is determined as the standard deviation of the seven or more replicates.

MDLs are determined on an annual basis for each method and sample matrix and are archived with the Laboratory Supervisors and the Laboratory Director.

CESE can perform project specific MDL studies if existing studies need to be conducted on a more frequent basis.

7.4 DEMONSTRATION OF METHOD CAPABILITY

Prior to acceptance and use of any method, satisfactory initial demonstration of capability (IDC) is required. This initial demonstration of method performance is performed each time there is a significant change in instrument type, personnel or test method. The process is described in Appendix A.

7.5 CONTROL CHARTING

The performance of analytical instrumentation and human operators can be monitored using control charts. Control charts are used to record the results of quantitative QC checks such as blanks, calibration checks, and laboratory duplicates. Control charts document instrument and measurement system performance on a regular basis and identify conditions requiring corrective actions on a real time basis. Control charting will be updated and retained on a regular interval for the following tests, for applicable QA parameters: Chlorophyll a (EPA 445.0), Acid Number (ASTM D664), Cloud Point (ASTM D2500), Elemental Analysis of Biodiesel (EN14538 and ASTM D4951), Flash Point (ASTM D93), Free and Total Glycerin (ASTM D6584), Oxidation Stability (EN14112), and Sulfur (ASTM D4294). The control chart tables will be generated for each applicable test and include the LIMS number or order of the associated samples and analysis date. If a test is out of QA tolerance, then corrective action will be initiated per the individual test SOP.

All control charting will be stored on the shared Biodiesel or Nutrients Network Drives. Control charts will be reviewed by the laboratory supervisor and Laboratory Director on a quarterly basis. Control charts will be revised only if the existing limits are no longer appropriate, for example if new instrumentation were purchased and brought on-line, improvements were made in the analytical method, of a change in the value of the QC reference material.

8.0 DATA REDUCTION, REVIEW, REPORTING, AND RECORDS

8.1 DATA REDUCTION AND REVIEW

Data are reviewed and validated to ensure that the analytical data are properly reduced and properly transcribed to the correct reporting format.

CESE has developed a standardized data review and reporting procedure. In summary, all data are reviewed by the analyst and the data report is generated via the LIMS or spreadsheet, which is instrument dependent. These customer specific reports are reviewed to check the agreement of the raw data with reported data. A project narrative is developed for each report, where requested by the client. The report then undergoes peer review by customer service personnel or other appropriate personnel (Laboratory Director) for a 10% data review, including checking the agreement of the raw data versus reported results as well as a verification of any calculations. A project narrative is developed for each report, where requested by the client and the package is emailed to the client. A hard copy package of the report is sent to the client, as requested.

8.2 DATA REDUCTION AND INITIAL REVIEW

Raw data resulting from the instrument analyses of samples are reduced according the laboratory SOPs. Computer programs used for data reduction are verified by manual calculations on a regular basis. All information used in the calculations (e.g., raw data, calibration files, results of standard additions, interference check results, and blank or background-correction protocols) are recorded in order to enable reconstruction of the final result at a later date. Information on the preparation of the sample (e.g., weight or volume of sample used, percent dry weight for solids, extract volume, dilution factor used) is maintained in order to enable reconstruction of the final result at a later date.

Upon finishing the data reduction, the analyst performs the initial review for the following items:

- The sample identifiers (IDs) in the run sequence match the sample IDs on the sample vials in the autosampler tray and the IDs on the on the data summary form match the sample IDs on the chain of custody;
- All samples were analyzed by the method required on the chain of custody;
- All samples were analyzed within holding time;
- Sample run batch QC:
    - The instrument blank passes the QC criteria;
    - The ICV and CCV passes the QC criteria;
    - All samples are analyzed within instrument tune time or the CCV was run after every twenty samples and passes the QC requirements.
- One LCS was extracted per batch and the recoveries of the LCS pass the QC criteria;
- One method blank was extracted per sample batch and there was no target compound detected at greater than or equal to 3 times the MDL/ PQL (dependent upon method);
- The MS and MSD were extracted per client's requirement and the associated recoveries and RPD pass QC criteria;
- The concentrations of all target compounds are within the calibration range or validated with a method specific linearity study.

The analyst denotes any noncompliance and initiates corrective action, if required. The responsible analyst signs all data, records, and associated documents.

8.3 SECONDARY DATA REVIEW

All data are reviewed by a second analyst or supervisor according to laboratory procedures to check that calculations are correct and to detect transcription errors. The items listed in 8.2 are double-checked. Errors detected in the review process are referred to the analyst(s) for corrective action. The quality checklist is initiated and dated by the analyst, peer reviewer or Laboratory Director and is submitted with each raw data packet. After the data have been reviewed, the reviewer informs the Laboratory Director that the data can be reported.

As described in Section 8.2, the results of all quality control sample analyses are reviewed and evaluated before data are reported.

8.4 REPORT FORMAT AND CONTENTS

The results of each test, or series of tests, are client specific and are normally provided to each client in a report to include all the information necessary for the interpretation of the results. Where applicable, the data package consists of four sections; cover letter and case narrative, chain of custody, sample results, and QC data. Non compliant data are addressed in the cover letter and case narrative enclosed in each reporting package. These are the elements of the standard data reporting and can be modified based upon data reporting levels as requested by the client.

An analytical report should include:

- Date of receipt of sample, sample collection, preparation, and analysis. If the required holding time for any activity is less than or equal to 48 hours, the time must also be noted;
- Identification of the test method (s) used;
- Name of the analyst performing the analysis;
- Identification of whether data are calculated on a dry weight or wet weight basis;
- Identification of the reporting units;
- The reporting limit and clear identification of numerical results with values below it;
- Matrix;
- In the case of biodiesel, a signature of the Laboratory Director and/ or Manager and date will be on all sample reports.

A data package should include:

- The Chain of Custody and any transfer sheets provided;
- The order number and the total number of pages, with all pages sequentially numbered;
- Sample results;
- All quality control data requested by the client.

A cover letter should include:

- Name and address of laboratory, and phone number with name of contact person;
- Name and address of client;
- Name, title, and signature of Laboratory Director or data reporter;
- Laboratory order identification number;
- Any data discrepancies found;
- Any deviations from, additions to or exclusions from standard operating procedures;
- Any conditions that may have affected the quality of results;

- If pertinent a statement of non-compliance with requirements and/or specifications;
- Any other information requested by the client;
- When the report is complete and printed the Laboratory Director or data reporter reviews the report and signs the cover sheet and any quality control reports.

Material amendments to a test report after issue are made only in the form of a further document, or data transfer including the statement "Supplement to Test Report, order number." Clients are notified promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a report.

8.5 RECORDS

Records provide the direct evidence and support for the necessary technical interpretations, judgments, and discussions concerning laboratory results. These records, particularly those that are anticipated to be used as evidentiary data, provide the historical evidence needed for later reviews and analyses. All records referenced in this section (8.5) are retained for a minimum of five years.

Laboratory records generally consist of bound notebooks with numbered pages, personnel qualification and training forms, equipment maintenance and calibration forms, and chain-of-custody forms. All records are recorded in indelible ink and retained for five years. Records that are stored or generated by computers or personal computers (PCs) have hard copy or write-protected backup copies.

Any documentation errors are corrected by drawing a single line through the error so that it remains legible and is initialed by the responsible individual, along with the date of change. The correction is written adjacent to the error.

Laboratory records include the following:

Standard Operating Procedures

Any revisions to laboratory procedures are written, dated, and distributed to all affected individuals to ensure implementation of changes.

Equipment Maintenance Documentation

Documents detailing the receipt and specification of analytical equipment are retained. A history of the maintenance record of each system serves as an indication of the adequacy of maintenance schedules and parts inventory. As appropriate, the maintenance guidelines of the equipment manufacturer are followed. When maintenance is necessary, it is documented in logbooks. In the case of biodiesel analytical instrumentation the log book will contain the name of the instrument maintained, the dates of last and next maintenance, and person performing the maintenance.

Calibration Records & Traceability of Standards/Reagents

The frequency, conditions, standards, and records reflecting the calibration history of a measurement system are recorded.

Sample Management

A record of all procedures to which a sample is subjected while in the possession of the laboratory is maintained. These include records pertaining to:

- Sample preservation including appropriateness of sample container and compliance with holding time requirement;
- Sample identification, receipt, acceptance or rejection and log-in;
- Sample storage, tracking, and transmittal forms;

Original Data
The raw data and calculated results for all samples are maintained in laboratory notebooks, logs, bench sheets, files or other sample tracking or data entry forms. Instrumental output is stored in a computer file and a hard copy report. Hard copies of the original data are archived by laboratory personnel. These records include:

- Laboratory sample ID code;
- Date of analysis;
- Instrumentation identification and instrument operating conditions/parameters;
- Analysis type and sample preparation information, including sample aliquots processed, cleanup, and separation protocols;
- All manual, automated, or statistical calculations, including all manual integrations;
- Confirmatory analysis data, when required to be performed;
- Review history of sample data; and
- Analyst's signature and second level data review signature.

QC Data
The raw data and calculated results for all QC samples and standards are maintained in the manner described in the preceding paragraph. Documentation allows correlation of sample results with associated QC data. Documentation also includes the source and lot numbers of standards for traceability. QC samples include, but are not limited to, control samples, method blanks, matrix spikes, and matrix spike duplicates.

Correspondence
Correspondence pertinent to a project is kept and placed in the project files.

Final Report
A copy of any report issued and any supporting documentation.

Administrative Records
The following are maintained:

- Personnel qualifications, experience and training records;
- Initial and continuing demonstration of proficiency for each analyst.

8.6 DOCUMENT CONTROL SYSTEM

A document control system is used to ensure that all staff have access to current policies and procedures at all times. Documents which are managed by this system include this Quality Plan and all SOP's. The system consists of a document review, revision and approval system, and document control and distribution. To ensure access the Quality Assurance Plan and SOPs are stored electronically on the CESE Network.

All quality documents (this Manual, SOPs, policies, etc.) are reviewed and approved by the

Laboratory Director, and in some cases the CESE Director. Such documents are revised whenever the activity described changes significantly. All documents are reviewed at least annually.

All quality documents are controlled by the Laboratory Director on the CESE network. Controlled copies are provided to applicable individuals in the laboratory.

8.7 CONFIDENTIALITY

All laboratory results and associated raw data are kept in confidence to the customer who requested the analyses. CESE maintains confidentiality for all information imparted to or derived for the client. Information shall only be released on the written authority of the client. Access to laboratory records and LIMS data is limited to laboratory personnel except with the permission of the Laboratory Director.

Where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, staff will ensure confidentiality is preserved.

9.0 PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

9.1 INTERNAL LABORATORY AUDITS

Internal audits are periodically performed by the Laboratory Director to verify that laboratory operations continue to comply with the requirements of the quality system. Where the audit findings cast doubt on the correctness or validity of the laboratory's results, an immediate corrective action is initiated and any client whose work may have been affected is notified. The senior laboratory staff, has three weeks to provide a written response to the report detailing corrective actions and implementation dates.

The internal system audits include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc.

9.3 THIRD PARTY AUDIT

External audits are conducted by a third party at the discretion of client or as part of CESE's standard laboratory practice. External audits are conducted at the discretion of the client, either prior to award of a contract, or as part of an ongoing laboratory monitoring process. Such audits may include submission of blind performance samples, data packages for complete independent validation, or a complete personal walk-through interview. CESE currently undergoes third party audits as part of the State of Connecticut Laboratory Certification. The Laboratory Director maintains records of each audit and the associated findings.

9.3 MANAGERIAL REVIEW

At least once per year, laboratory management conducts a review of the biodiesel laboratory quality system, and other laboratories where appropriate, to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review will consist of reports from the Laboratory Director and supervisory personnel. Additionally the Managerial Review should take into account the outcome of recent internal audits, assessments by external bodies, the results of proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions and other relevant factors.

10.0 FACILITIES, EQUIPMENT, AND PREVENTATIVE MAINTENANCE

10.1 FACILITIES AND EQUIPMENT

For over fifteen years CESE has been providing research grade analytical chemistry services to our clients in a timeframe that meets their needs. CESE's state-of-the-art laboratories provide a full range of analytical testing services to address the research needs of universities, government and industry. The labs, which include sections for organic, trace metal and nutrient analysis, occupy more than 9,300 square feet of space and are equipped with advanced instrumentation and computers. CESE has a clean room (Class 100 equivalent) for mercury preparation and analysis using a GC/ Atomic Fluorescence Spectrophotometer. CESE has a dedicated sample login facility, walk-in sample storage area and BOD incubator (each 80 ft^2), long-term sample storage areas, sampling equipment and bottle storage areas, fabrication and machine shop, conference room, and various offices. The entire facility is secured by a card-key access system. All major pieces of analytical equipment are listed in Appendix B.

Records are maintained for each major piece of equipment and all reference materials significant to the tests performed. These records include documentation on all routine and non-routine maintenance activities and reference material verifications.
The records include:

- The name of the equipment;
- The manufacturer's name, type identification, and serial number or other unique identification;
- Date received and date placed in service (if available);
- Current location, where appropriate;
- If available, condition when received (e.g. new, used, and/ or reconditioned);
- Copy of the manufacturer's instructions, where available;
- Dates and results of calibrations;
- Details of maintenance carried out to date and planned for the future; and
- History of any damage, malfunction, modification or repair.

10.2 COMPUTERS AND ELECTRONIC DATA SECURITY REQUIREMENTS

Data security has been divided into three categories: access, protection against corruption, and redundancy. Access to data is subject to levels of control. Non-critical data is available throughout the network. Critical data are available to members of predefined groups only. Sensitive and proprietary data is restricted at a user-by-user level. Data are protected from corruption by a strategy of limited access and redundancy. Redundancy takes the form of data backups via computer and secure storage of data in hard copy. All raw analytical data are stored in hardcopy form and on the CESE file server.

All computers and printers, both staff and instrument, are connected via the local area network (LAN). The CESE primary file server is a Dell PowerEdge T320 running Windows Server 2012. The System Administrator controls access and privileges to all shared network devices from this machine. This server is automated to perform data backups to various network attached storage (NAS) devices on a weekly basis. Further, a secondary server (a Dell PowerEdge T410 also running Windows Server 2012) provides the Windows Server Update Service (WSUS), which keeps desktop systems current with applicable security updates. In addition, all desktop systems are equipped with Microsoft System Center 2012 Endpoint Protection, to minimize security issues associated with software viruses, malware, and the like.

CESE maintains a web page via a dedicated server, which is separate from the file server. The web address is http://www.cese.uconn.edu/.

10.3 PREVENTATIVE MAINTENANCE

The instrument operator handles routine instrument preventive maintenance. Repair maintenance is initially diagnosed by an instrumentation technician. The responsibility for preventative maintenance belongs to the Senior Analyst.

Preventive maintenance, such as lubrication, source cleaning, and detector cleaning, is performed according to the procedures delineated in the manufacturer's instrument manual, including the frequency of such maintenance. Precision and accuracy data are examined for trends and excursions beyond control limits to determine evidence of instrument malfunction. Maintenance is performed when an instrument begins to degrade as evidenced by the degradation of peak resolution, shift in calibration curves, decreased ion sensitivity, or failure to meet one or another of the quality control criteria.

Instrument maintenance logbooks are maintained in the laboratory at all times. The logbook contains a complete history of past maintenance, both routine and non-routine. The nature of work performed, the date, and the signature of the person who performed the work are recorded in the logbook. Preventive maintenance is scheduled according to each manufacturer’s recommendation. Keeping adequate supplies of all expendable items minimizes instrument downtime, where expendable means an expected lifetime of less than one year.

10.4 INSPECTION/ ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

Prior to the acceptance of any supplies and consumables, the items are checked for breakage. Any discrepancies in the packing lists are noted. The packing slips are given to the administrative support person for filing.

11.0 CORRECTIVE ACTION

11.1 CORRECTIVE ACTION PROCEDURES

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of control QC performance that can affect data quality. To the extent possible, samples are reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data are to be reported, all samples associated with the failed quality control measure are reported with the appropriate notation in the associated non-conformance letter. Sample results may also be qualified when holding times are not met, improper sample containers and/ or preservatives are used or when other deviations from laboratory standard practices and procedures occur.

Corrective action in the laboratory may occur prior to, during and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/ high pH readings, or potentially high concentration samples may be identified during sample log-in or just prior to analysis. The SOPs specify conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, and automatic re-injection/ reanalysis when certain QC criteria are not met.

Any QC sample result outside of acceptance limits requires corrective action. Once the problem has been identified and addressed, corrective action may include the reanalysis of samples, or appropriately qualifying the results.

The analyst will identify the need for corrective action. The Laboratory Director or his/her designee will approve the required corrective action to be implemented by the laboratory staff.

Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the quality of the laboratory's tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited. Records of the complaint and subsequent actions are maintained.

12.0 SUBCONTRACTING AND SUPPORT SERVICES AND SUPPLIES

12.1 SUBCONTRACTING LABORATORY SERVICES

Clients are advised in writing if any analyses will be subcontracted to another laboratory. Any subcontracted work is placed with another accredited laboratory for the tests to be performed. The following records of all subcontracted analyses are maintained:

- A copy of the subcontracted laboratory's scope of accreditation;
- A copy of the report from the subcontracted laboratory;
- The notice to the client.

In the case of receipt of biodiesel tests from a non BQ-9000 laboratory, CESE will require the receipt of a completed and signed Form BQF-1 with supporting documentation. This documentation will be retained for a minimum of two years.

12.2 OUTSIDE SUPPORT SERVICES AND SUPPLIES

The Center for Environmental Sciences and Engineering only uses those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's tests. Records of all suppliers for support services or supplies required for tests are maintained.

13.0 REFERENCES

Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80, December 29, 1980, Office of Monitoring Systems and Quality Assurance, ORD, U.S. EPA, Washington, DC 20460.

RCRA QAPP Instructions, U. S. EPA Region 5, Revision: April 1998

ASTM D-5283-92.Generation of Environmental Data Related to Waste Management Activities: Quality assurance and Quality Control Planning and Implementation

"American National Standards Specification and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (ANSI/ASQC E-4)", 1994

EPA 2185 - Good Automated Laboratory Practices, 1995

ISO/IEC Guide 25: 1990. General requirements for the competence of calibration and testing laboratories

QA/R-2: EPA Requirements for Quality Management Plans, August, 1994

QA/G-4: Guidance for the Data Quality Objectives Process EPA/600/R-96/055, September, 1994

QA/R-5: EPA Requirements for Quality Assurance Project Plans Draft - November 1997

QA/G-5: Guidance on Quality Assurance Project Plans EPA/600/R-98/018, February, 1998

QA/G-6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November, 1995

QA/G-9: Guidance for the Data Quality Assessment: Practical Methods for Data Analysis EPA/600/R-96/084 , January, 1998

Manual for the Certification of Laboratories Analyzing Drinking Water EPA/570/9-90/008

APPENDIX A: INITIAL DEMONSTRATION OF CAPABILITY

A demonstration of capability (DOC) is made prior to using any test method, and at any time there is a significant change in instrument type, personnel or test method. All demonstrations are documented. A copy of the DOC is retained in a file in the respective laboratory.

An initial demonstration of capability includes a precision and accuracy study, MDL study, and performance evaluation (PE).

Precision and Accuracy Study

The precision and accuracy study consists of:

- Four duplicate samples are prepared by spiking the analytes at the middle level of initial calibration into a clean matrix;
- The samples are analyzed according to the test method either concurrently or over a period of days;
- Using all of the results, the mean recovery and the standard deviation are calculated for each parameter of interest;
- The calculated mean and standard deviation are compared to the corresponding acceptance criteria for precision and accuracy on the test method (if applicable) or in the laboratory generated acceptance criteria (if there are not established mandatory criteria). If any one of the parameters do not meet the acceptance criteria, the performance is considered unacceptable for that parameter.

Method Detection Limit Study

The method detection limit study consists of:

- Seven duplicate samples are prepared by spiking the analytes at the level of 1 to 2 times the low standard or practical quantitation limit (PQL) into a clean matrix;
- The samples are analyzed according to the test method, either concurrently or over a period of days;
- Using all of the results, the mean recovery and the standard deviation are calculated for each parameter of interest;
- The MDL is calculated by multiplying the standard deviation by 3.14. The recovery and calculated MDL are compared to the corresponding acceptance criteria in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

Performance Evaluation

CESE will use the applicable test method to analyze the proficiency test (PT) sample or a standard reference material provided by a certified vendor, if available.

The analysis of actual samples may begin upon the meeting of all criteria required. When one or more of the tested parameters fail at least one of the acceptance criteria, the laboratory will repeat the test for all parameters that failed to meet the criteria.

APPENDIX B: LIST OF INSTRUMENTATION

Organic Analysis

Gas Chromatograph/Mass Spectrometry Systems

Make	GC Model	Detector Model	Sample Type	Service Date
Agilent	6890	Quattro micro	Semi-VOC/Pest/PCB	2011
Agilent	6890	5973	VOC in Liquid and Solid / Semi-VOC	2000

Gas Chromatograph Systems

Make	GC Model	Detector Type	Sample Type	Service Date
Agilent	6890	Dual μECDs	Pesticides/PCB/Herbicides	2000
Hewlett Packard	5890	FID	Biofuel	

High Pressure Liquid Chromatography Systems

Make	Model	Detector Type	Sample Type	Service Date
Waters UPLC	Acquity	MS/MS	Pharmaceuticals, EDCs	2010
		ELSD	Pharaceuticals	2010
		Fluorescence	PAHs, Pharaceuticals	2010
		Photo-Diode Array	Aldehydes/Ketones, PAHs, Pharaceuticals	2010
Perkin Elmer	200	Photo-Diode Array	Aldehydes/Ketones	1995
		Fluorescence	PAHs, Pharaceuticals	1995

Organic Preparation

Chromatography Systems

Make	Model	Type	Detector Type	Service Date
Waters		HPLC	UV	2000
OI	AP-1000	GPC2	UV	2001

Automated Solvent Extraction Systems

Make	Model	Service Date
Dionex	ASE 200	1999

Automated Concentrators

Make	Model	Service Date
Genevac	EZ-2 Plus	2013
Labconco	rapidvap	2001
Labconco	rapidvap	2001

Freeze Dryer

Make	Model	Service Date
Labconco	Freezone	2011

Trace Metal Analysis

Make	Model	Type	Analyses Performed	Service Date
Perkin Elmer	DRC-e with AS90 Autosampler	ICP/MS	Elements	2010
Cetac	LSX-500 Laser Ablation Unit		Solid samples for Elements	2010
Perkin Elmer	Optima 7300 DV	ICP/OES	Elements	2009
Perkin Elmer	Optima 3300XL with AS 91 Autosampler	ICP/OES	Elements	1997

Trace Metal Preparation

Make	Model	Type	Service Date
Milestone	Ethos EZ	Preparation Microwave	2008
Environmental Express	SC196	Hot Block Digestion	2009
Environmental Express	SC100 (3 ea)	Hot Block Digestion	2001

Mercury Analysis

Make	Model	Detector Type	Sample Type	Service Date
Brooks Rand	Model III	CVAFS	Various Matrices for Speciated Mercury	2006
Tekran	2600	CVAFS	Ultra-trace mercury in liquid matrices	2008
Perkin Elmer	FIMS with AS 90 Autosampler	CVAAS	Various Matrices for Mercury	1996
Milestone	DMA 80	AFS	Combustion AFS for trace mercury in solids	2008

Nutrients/ Wet Chemistry Analysis

Make	Model	Type	Analyses Performed	Service Date
Lachat	8500 Quick Chem	FIA	N,P, silica series	2007
Dionex	DX-500	IC	Cations and anions	1998
Shimadzu	TOC-L	TOC Analyzer	Organic and inorganic carbon	2013
Turner	Trilogy	Fluorometer	Chlorophyll and tracer dyes	2008
Metrohm	877	Auto-Titrator	Alkalinity, pH	2011
Accumet	XC40	DO Meter	Dissolved oxygen	2007
Perkin-Elmer	2400	CHN Analyzer	C,H,N	2001

Biofuel Analysis

Make	Model	Type	Analyses Performed	Service Date
Hewlett-Packard	5890	GC-FID	Free and total glycerin	1997
Agilent	6890/5973	GC-MSD	Methanol	2002
Tekmar	7000	S-H injector	Methanol	2000
Kohler	VDA3000	Vacuum Distillation	Distillation Temperature	20098
A2 Technologies	PAL	FT-IR	Biodiesel Blend	2009
Metrohm	743	Rancimat	Oxidation stability	2009
Metrohm	798 MPT Titrino	Auto-Titrator	Total acid number	2009
Spectro	IQ II	ED-XRF	Sulfur and other elements	2009
IEC	HN-SII	Centrifuge	Water and sediment	2009
Fisher	Pensky-Martens	Flash Tester	Flash Point	2002
Perkin Elmer	Optima 3300XL with AS 91 Autosampler	ICP/OES	Elements	1997
Phase Technologies	CPA-T30	Light Scattering	Cloud Point	2006

APPENDIX C: METHOD LIST

Metals Analysis (EPA Methods)

Mercury	Preparation	Analysis
7471	3050B	6010
7470	200.8	6020
245.6	200.7	200.8
245.5		200.7
1630		
1631		

Nutrients Analysis

Ammonia (350.1)
NOx (Nitrate + Nitrite) (353.2)
Orthophosphorus (365.3)
Total Dissolved Nitrogen (353.2)
Total Dissolved Phosphorus (365.3)
Particulate Phosphorus (365.3)
Silica (Dissolved and Biogenic) (370.1)
Nitrite (NO_2) (353.2)
Chemical Oxygen Demand (410.4)
Alkalinity (SM 2320B)
Total Organic Carbon (415.1/ 440)
BOD (5 and 30 day) (405.1)
Particulate Carbon (440)
Particulate Nitrogen (440)
Chlorophyll a (AERP 12)
Total Suspended Solids (160.2)
Total Dissolved Solids (160.1)
Total Solids (160.3)
Turbidity (180.1)
Cations and Anions (300.0)

Biofuel Analysis (ASTM and European Methods)

Flash Point (ASTM D93)
Water and Sediment (ASTM D2709)
Cloud Point (ASTM D2500)
Total Acid Number (ASTM D664)
Cold Soak Filtration (ASTM D7501)
Free and Total Glycerin (ASTM D6584)
Oxidation Stability (EN 14112)
Biodiesel Blend (ASTM D7371 and EN 14078)
Kinematic Viscosity (ASTM D445)
Sulfur (ASTM D5453)
Copper Strip Corrosion (ASTM D130)
Calcium, Magnesium, Sodium, Potassium, Phosphorus (ASTM D4951 and EN 14538)
Methanol (EN 14110)
Distillation, T90 AET (ASTM D86)