- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 19

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS
(NELAC 5.5.3)

19.1 OVERVIEW

TestAmerica Irvine is a 45,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

19.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may effect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include temperature and barometric pressure. These are monitored in relevant testing areas during the testing period.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels (refer to Section 12).

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

19.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic analysis is performed in a separate room provided with positive air pressure.
- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

19.4 FLOOR PLAN

A floor plan can be found in Appendix 3.

19.5 BUILDING SECURITY

Building electronic keys are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of TestAmerica Irvine. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

Signs are posted in the laboratory designating employee only areas - "Authorized employees beyond this point".

SECTION 20.0

TEST METHODS AND METHOD VALIDATION
(NELAC 5.5.4)

20.1 OVERVIEW

TestAmerica Irvine uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

20.2 STANDARD OPERATING PROCEDURES (SOPs)

TestAmerica Irvine maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory (refer to Section 6 on Document Control):

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for preparation, review, revision and control are incorporated by reference to SOPs: **CW-Q-S-002** (Writing a Standard Operating Procedure (SOP) and SOP **IR-QA-DOC** (Document Control and Review)
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

20.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP. Refer to the corporate SOP CW-Q-S-002 "Writing a Standard Operating Procedure" for content and requirements of technical and non-technical SOPs.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from

the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

20.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc..), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

20.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

In general, TestAmerica Irvine follows procedures from the referenced methods shown below in 20.3.1.4.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

20.4.1.1 The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*

- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

20.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

20.4.2.1 A demonstration of capability is performed whenever there is a change in instrument type, method or personnel.

20.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures (refer to Section 15, Control of Records).

20.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method).
- The reporting limit is set at or above the first standard of the curve for the analyte.
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*
- Refer to Section 12 (Control of Non-Conforming Work).

20.4.3 Initial Demonstration of Capability (IDOC) Procedures

The laboratory's SOP IR-QA-TRAIN (Training and Documentation) describes in detail the process by which IDOCs are prepared, performed, evaluated, and documented.

20.4.3.1 The following criteria are to be met for any IDOC:

- The spiking standard used must be prepared independently from those used in instrument calibration.
- The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.
- At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).
- Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.
- When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.
- Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated

acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

20.4.3.2 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 20.4.3.3 above.
- Beginning with 20.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 20.4.3.1 above.

A certification statement (see Figure 20-1) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

20.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP/Methods Manual (Section 20.2) and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method. The information included in the checklist below (Figure 20-2) is needed before samples are accepted for analysis by a new method.

20.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled. (From 2003 NELAC Standard)

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

20.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

20.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

20.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. The laboratory determinations of MDLs are described in Section 20.6.

20.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

20.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

20.6.1.5 Determination of Range

Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

20.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

20.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

20.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

20.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 20.7.10). The analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

20.7.1 MDL's are initially performed for each individual instrument and non-microbiological method analysis. Unless there are requirements to the contrary, the laboratory will use the highest calculated MDL for all instruments used for a given method as the MDL for reporting purposes. This MDL is not required for methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report values to the MDL. Titration and gravimetric methods where there is no additional preparation involved, the MDL is based on the lowest discernable unit of measure that can be observed.

20.7.2 MDL's must be run against acceptable instrument QC, including ICV's and Tunes. This is to insure that the instrument is in proper working condition and falsely high or low MDL's are not calculated.

20.7.3 Use only clean matrix which is free of target analytes (e.g.: Laboratory reagent water, Ottawa Sand) unless a project specific MDL is required in a field sample matrix.

20.7.4 The Reporting Limit (also may be referred to as Limit of Quantitation or LOQ) should generally be between 2 and 5 times the MDL. If the MDL is being performed during method development, use this guideline to determine the Reporting Limit for the analysis.

20.7.5 If a sample is diluted, the reported MDL is adjusted according to the dilution factor.

20.7.6 The calculated MDL cannot be greater than the spike amount.

20.7.7 If the most recent calculated MDL does not permit qualitative identification of the analyte then the laboratory may use technical judgment for establishing the MDL (e.g., calculate what level would give a qualitative ID, compare with IDL (20.7), spike at a level where qualitative ID is determined and assign that value as MDL, minimum sensitivity requirements, Standard deviation of method blanks over time, etc.). These alternate verification procedures are documented in the laboratory's MDL.SOP (Determination of Method Detection Limits).

20.7.8 Each of the replicate spikes must be qualitatively identifiable (e.g., appear in both columns for dual column methods, characteristic ions for GCMS mass spectra, etc). Manual integrations to force the baseline for detection are not allowed.

20.7.9 The initial MDL is calculated as follows:

$$MDL = t_{(n-1,\ 1-a\ =\ 0.99)} \times (\text{Standard Deviation of replicates})$$

where $t_{(n-1,\ 1-a\ =\ 0.99)}$ = 3.143 for seven replicates. (2.998 for eight)

20.7.10 Subsequent to the initial MDL determination, periodic MDL verification, confirmation or determinations may be performed by the procedure in 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices (e.g., method blanks over time, single standard spikes that have been subjected to applicable sample prep processes, etc.). The procedures utilized is documented in the laboratory SOP MDL.SOP (Determination of Method Detection Limts).

20.7.11 Because of the inherent variability in results outside of the calibration range, TestAmerica does not recommend the reporting of results below the lowest calibration point in a curve; however, it is recognized that some projects and agencies require the reporting of results below the RL. Any result that falls between the MDL and the Reporting limit, when reported, will be qualified as an estimated value.

20.7.12 Detections reported down to the MDL must be qualitatively identified.

20.7.13 MDLs and Reporting limits are adjusted in LIMs based on moisture content and sample aliquot size.

20.8 INSTRUMENT DETECTION LIMITS (IDL)

20.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

20.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

20.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

20.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

20.9.1 Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and 1-4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified or see section 20.6.7 for other options. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established (See 20.6.7). MDLs must be verified at least annually if an annual MDL study is not performed.

20.9.2 When a Reporting limit is established, it must be initially verified by the analysis of a low level standard or QC sample (LCS at 1-2 the reporting limit) and annually thereafter. Unless there are requirements to the contrary the acceptance criteria is ± 50%. The annual requirement is waved for methods that have an annually verified MDL.

20.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes.

For GC, HPLC and IC methods, there must be sufficient separation between analyte peaks so as to not misidentify analytes. In the mid-level standard, the distance between the valley and peak height cannot be any less than 25% of the sum of the peak heights of the analytes. This also applies to GCMS in the case where the two compounds share the same quantitation ion.

Note: Some analytes do not separate sufficiently to be able to identify or quantitate them as separate analytes (e.g. m-xylene and p-xylene) and are quantitated and reported as a single analyte (e.g. m,p-xylenes).

Once the analyst has determined that the instrument is in optimum working condition through calibration and calibration verification procedures, he or she uses a mid-range calibration or calibration verification standard to establish the retention times for each of the individual analytes in a method. The analyst makes three injections of the same standard over a 72-hour (24 hr period for 300.0) period, tabulating the retention times for each analyte for each of the three injections. The width of retention time window is normally the average absolute retention time ± 3 Standard Deviations. A peak outside the retention time window will not be identified by the computer as a positive match of the analyte of interest.

It is possible for the statistically calculated RT window to be too tight and need to be adjusted based on analyst experience. In these instances method default retention time windows may be used (e.g., for 8000 series methods a default of 0.03 minutes may be used, and EPA CLP 0.05

minutes is used). The same concept is applied when any peak outside of that window will not be identified by the computer as a positive match.

The calibration verification standard at the beginning of a run may be used to adjust the RT for an analyte. This is essentially re-centering the window but the size of the window remains the same. The RTs are verified when all analytes are within their RT windows and are properly identified.

20.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks and specific electrode response factors.

20.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

20.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

20.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

20.12.3 The uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

20.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and

the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 +/- 0.5 mg/l.

20.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g. 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

20.13 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

20.13.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in SOP COMPSECU.SOP (Computer Security). The laboratory is currently running the Element which is a 3rd party LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes SQL which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

20.13.1.1 Maintain the Database Integrity

Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

  Note: "Commercial off-the-shelf software in use within the designed application range is considered to be sufficiently validated." *From NELAC 2003 Standard.* However, laboratory specific configurations or modifications are validated prior to use.

- In order to assure accuracy, all data entered or transferred into the LIMS data system goes through a minimum of two levels of review.
- The QA department performs random data audits to ensure the correct information has been reported.
- Changes to reports are documented using the non-conformance/corrective action database. Changed report files are named "revision_a", "revision_b", etc to clearly differentiate them from the originally reported file.

- Analytical data file security is provided through three policies.
  - The first policy forbids unauthorized personnel from using laboratory data acquisition computers.
  - The second policy is the implementation of network passwords and login names that restrict directory access.

- The third layer is maintained through the LIMS and includes the use of username/password combinations to gain access to the LIMS system, the fact that all data in the LIMS is associated with the user to added/reviewed the data, and the restriction of review authority of data.

- All software installations will be in accordance with any relevant copyright licensing regulations.
- All software installed on any computer within the laboratory must be approved by the Information Technology Department regional support technician assigned to the laboratory Shrink-wrapped or otherwise sealed OEM software that is directly related to instrument usage does not need approval but the Information Technology department must be notified of the installation.
- Anti-virus software shall be installed on all servers and workstations. The anti-virus software shall be configured to check for virus signature file and program updates on a daily basis and these updates will be pushed to all servers and workstations. The anti-virus software will be configured to clean any virus-infected file if possible, otherwise the file will be deleted. Disks and CDs brought from any outside source that are not OEM software must be scanned for viruses before being accessed.

- **Interlab LIMS Permissions Policy**
  - PURPOSE - The purpose of this policy is to provide a mechanism for maintaining the integrity of information contained in each laboratory's LIMS while providing the necessary access for information sharing to staff at other laboratory facilities.
  - DEFINITIONS - Host Laboratory: The laboratory facility that 'owns' the LIMS system or 'hosts' a project/job.
  - POLICIES

    (a) All permissions for the laboratory's LIMS system must only be granted by a representative of that laboratory.
    - If someone outside of the host lab needs permissions for Project Management or other uses, they must go through the Lab Director or his/her designated representative.
    - Permissions must never be granted without the knowledge of the host laboratory.

    (b) Only laboratory analytical or QA staff from the home laboratory may have edit permissions for laboratory analysis data.

    (c) Any changes made in laboratory's LIMS system:
    - Must be documented and traceable.
    - If made by staff of an affiliate lab, written permission from the home lab to make the changes (email approval is sufficient) is required.
    - No corrections may be made in another laboratories system without their knowledge.

    (d) Data qualifiers in laboratory reports must only be corrected, edited, etc. by the staff at the host laboratory.

    (e) Full analytical data "View" only permissions may be granted to outside Project Management and Sales staff. ~~Query~~ Search permissions may also be granted so status may be checked.

    (f) All qualifiers must be approved by QA staff before adding to standard reference (static) tables.

    (g) Please contact Corporate QA or IT staff if you have any questions regarding implementation or interpretation of this policy.

20.13.1.2 **Ensure Information Availability:** Protection against loss of information or service through scheduled back-ups, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

- Insured by timely backup procedures on reliable backup media, stable file server network architecture, and UPS protection
- UPS Protection:
  - Each fileserver is protected by an appropriate power protection/backup unit. In the event of a power outage, there is approximately 15-30 minutes of up-time for the servers prior to shutdown. This allows for proper shutdown procedures to be followed with the fileservers.
- File Server Architecture
  - All files are maintained on multiple Windows 2000 or newer servers which are secured physically in the Information Technology office. Access to these servers is limited to members of the Information Technology staff.
  - All supporting software is maintained for at least 5 years from the last raw data generated using that software. [Length of time is dependent on local regulations or client requirements (e.g., OVAP requires 10 years).]
- System Back-up Overview and Procedures
  - Data from both servers and instrument attached PC's are backed up and purged in compliance with the corporate back-up policy.
  - A Maintenance Plan has been defined to create a daily archive of all data within the LIMS database to a backup location. This backup is initiated automatically by either the database or back-up system.
  - Backup tapes will be stored in compliance with the corporate Data Backup Policy. Backup verifications are carried out in accordance with the corporate Data Backup Policy.
  - Instrument data back-ups are verified on a periodic basis by the QA department when performing electronic data audits. The audit takes place on data that has been moved to a back-up location ensuring that it has been moved.

20.13.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls, and encryption of when electronically transmitting data.

- All servers are located in a secure area of the IT department offices. Access to the servers is limited to IT staff (Desktop Support, Director of LIMS support, Database administrator) and Lab Director.
- The company website contains SSL (Secure Socket Layer) encryption for secure website sessions and data transfers.
- The reporting portion of the LIMS system requires a project manager to enter their unique password anytime they create a report that displays a signature on it (.PDF).
- Electronic documents such as PDF files and electronic data deliverables will be made available to clients via the secure web site. The logon page for this web site contains an agreement that the customer must accept before they will be logged on which states that the customer agrees not to alter any electronic data made available to them.

- If electronic documents are made available outside of the web site, the customer must sign an agreement in advance that states they will not alter the data in any way.

20.13.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to entering the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

20.13.2.1 All raw data must be retained in the daily run sequence folder, computer file (if appropriate), and/or logbook. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

20.13.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. The units "mg/l" and "mg/kg" are the same as "parts per million (ppm)". The units "µg/l" and "µg/kg" are the same as "parts per billion (ppb)." For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%.

- Several environmental methods, such as color, turbidity, conductivity, use very specific, non-concentration units to report results (e.g., NTU, umhos/cm etc).
- Occasionally, the client requests that results be reported in units which take into account the measured flow of water or air during the collection of the sample. When they provide this information, the calculations can be performed and reported.

20.13.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant

figures. In general, client sample results are reported to 2 significant figures and QC samples are reported to 3 significant figures on the final report.

20.13.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

20.13.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

20.13.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 13.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Director and QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

20.13.4 Review / Verification Procedures

Review procedures are outlined in several SOPs (LOGIN.SOP [Sample Control], DATAREV.SOP [General Data Review], PMDATA.SOP [Project Management Data Reporting, Validation and Distribution]) to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data. (**CA-Q-S-002**, Acceptable Manual Integration Practices) The general review concepts are discussed below, more specific information can be found in the SOPs.

20.13.4.1 The data review process at TestAmerica Irvine starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Sample Control Supervisor reviews the transaction of the chain-of-custody forms and the inputted

information. The Project Managers perform final review of the chain-of-custody forms and inputted information.

20.13.4.2 The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add data qualifiers if applicable (see Appendix 7 for list of common data qualifiers). To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Approximately 15% of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. Manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

20.13.4.3 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Manager, Department Manager for further investigation. Corrective action is initiated whenever necessary.

20.13.4.4 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

20.13.4.5 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The following are some examples of chemical relationships that are reviewed (if data is available):

- Total Results are $\geq$ Dissolved results (e.g. metals)

- Total Solids (TS) ≥ TDS or TSS
- TKN ≥ Ammonia
- Total Phosphorus ≥ Orthophosphate
- COD ≥ TOC
- Total cyanide ≥ Amenable Cyanide
- TDS ≥ individual anions

20.13.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. (*Also see section 26 on Reporting Results*). The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

20.13.4.7 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 20-3.

20.13.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines.

20.13.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

20.13.5.2 Analysts shall not increase or decrease peak areas to for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

20.13.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

20.13.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Table 20-1
Laboratory Method SOPs by Department and Method Reference

DEPARTMENT	Method	TITLE	FILENAME
Administrative	Computer Security	COMPUTER SECURITY	COMPSECU.SOP
Administrative	Power Outage	POWER OUTAGES	POWEROUT.SOP
Administrative	Software	SOFTWARE MAINTENANCE	SOFTWARE.SOP
Extractions	CADHS LUFT Diesel	DIESEL EXTRACTION FOR SOIL, CA LUFT METHOD	DHSDIESEL.SOP
Extractions	EPA 3510C/EPA 625	EPA METHOD 3510C (BNA EXTRACTION BY SEPARATORY FUNNEL)	3510C_BNA.SOP
Extractions	EPA 3510C Diesel	EPA METHOD 3510C (DIESEL EXTRACTION FOR WATER)	3510_D.SOP
Extractions	EPA 3510C Pest/PCB	EPA METHOD 3510C (ORGANOCHLORINE PESTICIDES AND PCBS EXTRACTION FOR WATER)	3510_PR9.SOP
Extractions	EPA 3520C/EPA 625	EPA METHOD 3520C AND EPA METHOD 625 (CONTINUOUS LIQUID-LIQUID EXTRACTION)	3520C.SOP
Extractions	EPA 3545 Pest/PCB	EPA METHOD 3545 (PRESSURIZED FLUID EXTRACTION [PFE], PESTICIDE AND PCB EXTRACTION FOR SOIL)	3545_P.SOP
Extractions	EPA 3545 Semi-volatiles	EPA METHOD 3545 (PRESSURIZED FLUID EXTRACTION [PFE],SEMI-VOLATILE EXTRACTION FOR SOIL	3545_SV.SOP
Extractions	Na2SO4	PREPARATION OF SODIUM SULFATE FOR EXTRACTIONS	NA2SO4.SOP
GC-BTEX	EPA 8015/8020/CARB 410A	EPA METHOD 8015/8020, MODIFIED FOR AIR AND CARB METHOD 410A (BTEX, MTBE AND FUEL HYDROCARBONS AS GASOLINE)	8015AIR.SOP
GC-BTEX	EPA 8015B/8021B	GASOLINE RANGE ORGANICS (GRO) / BTEX AND MTBE	8015G.SOP
GC-BTEX	Mineral Spirits	GRO/BTEX/MTBE BY GC, ADDENDUM FOR DETERMINATION OF MINERAL SPIRITS (C8-C14) (EPA METHOD 8015B MOD.)	8015minsprt.SOP
GC-SEMI	EPA 8015B Diesel	EPA METHOD 8015B AND MODIFIED FOR DHS LUFT (TOTAL PETROLOLEUM HYDROCARBONS AS DIESEL)	8015D.SOP
GC-SEMI	EPA 8082/608	EPA METHOD 8082/608 (POLYCHLORINATED BIPHENYLS (PCBS) BY GC)	PCBs.SOP
GC-SEMI	EPA 8081A/608	ORGANOCHLORINE PESTICIDES BY GC (EPA METHODS 608 & 8081A)	PESTICIDES.SOP
GC-SEMI	EPA 8081A/608	ORGANOCHLORINE PESTICIDES BY GC (EPA METHODS 608 & 8081A) - Change Form ID - CF1	PESTICIDES.SOP-CF1
GCMS-SEMI	EPA 8270C MOD	1,4-DIOXANE BY 8270C MODIFIED SCAN MODE	14DIOX_8270C.SOP
GCMS-SEMI	827OC MOD	ADDENDUM FOR THE DETERMINATION OF DDT, DDD, DDE AND CHLOROBENZENE IN WATER AND METHYLENE CHLORIDE SOIL EXTRACTS	8270_DDT.SOP
GCMS-SEMI	Chloroacetaldeydes by GCMS	CHLORAL HYDRATE BY EPA 8270C SELECTIVE ION MONITORING (SIM) MODE	ChloralHydrate_8270Cr2.SOP
GCMS-SEMI	EPA 8270C/625	EPA METHOD 8270C (SEMI-VOLATILE ORGANIC COMPOUNDS)/EPA METHOD 625 (BASE/NEUTRALS AND ACIDS)	GCMS-SVOA.SOP
GCMS-SEMI	EPA 1625C MOD	NITROSAMINES BY GC/MS USING CHEMICAL IONIZATION (EPA 1625C MODIFIED)	IR-MSS-NITROSA
GCMS-VOL	EPA 8260B SIM	1,2,3-TRICHLOROPROPANE BY GAS CHROMATOGRAPHY/MASS SPECTOMETRY (GC/MS) SIM (SRL 524M-TCP, EPA 8260B SIM)	123TCP_R1.SOP
GCMS-VOL	EPA 8260B	EPA METHOD 8260B/624 (VOLATILE ORGANIC COMPOUNDS)	GCMS_VOA.SOP
GCMS-VOL	TPH by GCMS	TPH BY GCMS	GCMSTPH.SOP
GCMS-VOL	EPA 8260B MOD	VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) ADDENDUM FOR DETERMINATION OF 1,4-DIOXANE BY EPA 8260B MODIFIED	14DIOX.SOP
GCMS-VOL	EPA 5030B & 5035A	VOLATILE ORGANIC PREPARATION (EPA 5030B & 5035A)	IR-MSV-PREP
Health & Safety	Glass crusher	Glass Crusher	GLASSCR.SOP

DEPARTMENT	Method	TITLE	FILENAME
Health & Safety	Plastic shredder	PLASTIC SHREDDER	PLASTSH.SOP
Health & Safety	Safety Manual	SAFETY MANUAL & CHEMICAL HYGIENE PLAN	SMCHP.DOC
INORGANIC PREP	EPA 3050B	ACID DIGESTION FOR TOTAL METALS BY GFAA AND ICP IN SOIL (EPA METHOD 3050B)	3050B.SOP
INORGANIC PREP	EPA 3020A	ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS BY GFAA (EPA METHOD 3020A)	3020A.SOP
INORGANIC PREP	EPA 3010A	ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS BY ICP (EPA METHOD 3010A)	3010A.SOP
INORGANIC PREP	EPA 200.2/3005A	Acid Digestion of Water for Total Recoveralbe or Dissolved Metals by ICP and ICPMS	METPREP-W.SOP
INORGANIC PREP	EPA 1010	EPA METHOD 1010 (PENSKY-MARTENS CLOSED-CUP METHOD FOR DETERMINING IGNITABILITY)	1010.SOP
INORGANIC PREP	EPA 150.1/9040/9045/SM 4500H,B	EPA METHOD 150.1/ 9040B/ 9045C (ELECTROMETRIC pH)	150_1.SOP
INORGANIC PREP	SM 2120B	EPA METHOD 2120B (COLOR, COLORIMETRIC-PLATINUM-COBALT)	2120B.SOP
INORGANIC PREP	EPA 413.1	EPA METHOD 413.1 (TOTAL RECOVERABLE OIL AND GREASE FOR WATER)	413_1.SOP
INORGANIC PREP	EPA 413.2	EPA METHOD 413.2 (TOTAL RECOVERABLE OIL AND GREASE FOR WATER)	413_2.SOP
INORGANIC PREP	EPA 418.1	EPA METHOD 418.1 (TOTAL RECOVERABLE PETROLEUM HYDROCARBONS)	418_1.SOP
INORGANIC PREP	SM 3500Fe-D	FERROUS IRON BY SM 3500Fe-D	3500Fe_D.SOP
INORGANIC PREP	Glass Washing	GLASSWARE CLEANING	GLASS_E.SOP
INORGANIC PREP	EPA 1664A	GRAVIMETRIC DETERMINATION OF N-HEXANE EXTRACTABLE MATERIAL AND SILICA GEL TREATED N-HEXANE EXTRACTABLE MATERIAL IN WATER	1664A.SOP
INORGANIC PREP	Ignitability	IGNITABILITY IN SOIL	IGNITE.SOP
INORGANIC PREP	EPA 160.5	SETTLEABLE MATTER (EPA METHOD 160.5 / SM2540F)	IR-WET-SETT
INORGANIC PREP	SM 2710F	SPECIFIC GRAVITY BY MASS RATIO (SM2710F)	2710F.SOP
INORGANIC PREP	SM 2580B	STANDARD METHOD 2580B (OXIDATION REDUCTION POTENTIAL)	ORP.SOP
INORGANIC PREP	STLC TITLE 22, SECTION 66261.126, APPENDIX II)	STLC/WET EXTRACTION (TITLE 22, SECTION 66261.126, APPENDIX II)	STLC.SOP
INORGANIC PREP	EPA 1311/1312	TCLP & SPLP (EPA METHOD 1311 & 1312)	1311_1312.SOP
INORGANIC PREP	SM 2150B & EPA 140.1	THRESHOLD ODOR (SM 2150B & EPA 140.1)	IR-WET-ODOR
INORGANIC PREP	EPA 180.1	TURBIDITY, NEPHELOMETRIC (EPA METHOD 180.1 AND STANDARD METHOD 2130B)	180_1.SOP
METALS	EPA 200.9	DETERMINATION OF TRACE ELEMENTS BY STABILIZED TEMPERATURE GRAPHITE FURNACE AA (EPA METHOD 200.9 & STANDARD METHOD 3113)	200_9.SOP
METALS	EPA 9081A	EPA METHOD 9081A CATION-EXCHANGE CAPACITY OF SOILS (SODIUM ACETATE)	9081A.SOP
METALS	EPA 6010B/EPA 200.7	ICP METALS ANALYSES (EPA METHOD 6010B, EPA METHOD 200.7)	ICP.SOP
METALS	EPA 245.1/7470A/7471A	MERCURY, COLD-VAPOR ATOMIC ABSORPTION SPECTROMETRY (EPA METHODs 245.1/7470A/7471)	MERCURY.SOP
METALS	EPA 200.8	METALS BY ICP/MS (EPA METHOD 200.8)	200_8.SOP
METALS	EPA 6020	METALS BY ICP/MS (EPA METHOD 6020)	6020.SOP
METALS	CA DTSC 939-M	ORGANIC LEAD BY GRAPHITE FURNACE AA (CA DTSC 939-M)	ORG_PB_GFAA.SOP
PM	Data packages	DATA PACKAGE GENERATION	DATAPACK
PM	EDFs	EDF (ELECTRONIC DATA FORMAT)	EDF.SOP
PM	Client/Project set-up	PROJECT MANAGEMENT--CLIENT/PROJECT SET-UP	PMCLIENT.SOP
PM	Client communication	PROJECT MANAGEMENT--COMMUNICATION AND DOCUMENTATION	PMDOC.SOP

DEPARTMENT	Method	TITLE	FILENAME
PM	Data reporting	PROJECT MANAGEMENT--DATA REPORTING, VALIDATION AND DISTRIBUTION	PMDATA.SOP
PM	WIP packages	WELL INVESTIGATION PROGRAM (WIP) Package Generation	WIP.SOP
QA	Balances	BALANCE CALIBRATION VERIFICATION AND DOCUMENTATION	BAL.SOP
QA	BP GCLN	BP GCLN Technical Requirements	BPREQS.SOP
QA	Lot testing	CONTAINER AND REAGENT VERIFICATION BY LOT TESTING	LOTTEST.SOP
QA	Control Limits	CONTROL CHARTS AND STATISTICAL PROCESS CONTROL	CNTRLLIM.SOP
QA	Corrective Actions	CORRECTIVE ACTIONS	CAR.SOP
QA	Data Integrity	DATA INTEGRITY AND BUSINESS ETHICS PLAN	DIBEP.SOP
QA	Ethics Policy	DATA INTEGRITY AND ETHICAL PRACTICES POLICY AND PROCEDURE	DMA_ETHICS.SOP
QA	MDLs	DETERMINATION OF METHOD DETECTION LIMITS	MDL.SOP
QA	Documents	DOCUMENT CONTROL	DOCCNTRL.SOP
QA	ET Edwards	EARTH TECH/EDWARDS AFB PROJECT REQUIREMENTS	IR-QA-ETEDW.SOP
QA	Data Review	GENERAL DATA REVIEW	DATAREV.SOP
QA	ICOC	LEGAL CUSTODY PROCEDURES	LEGALCOC.SOP
QA	Logbooks	LOGBOOK DOCUMENTATION	LOGBOOK.SOP
QA	Manual Integration	MANUAL INTEGRATION AND DATA INTEGRITY	MANINT.SOP
QA	Pipets	PIPET CALIBRATION	PIP.SOP
QA	QA Manual	QUALITY ASSURANCE MANUAL	QAM
QA	QA Department	QUALITY ASSURANCE DEPARTMENT	QADR5.SOP
QA	Reagents and Standards	REAGENT AND STANDARD CONTROL AND DOCUMENTATION	STDCTRL.SOP
QA	Archiving	RECORD ARCHIVING	ARCHIV.SOP
QA	Storage Blanks	REFRIGERATOR STORAGE BLANKS	REFBLK.SOP
QA	Sig Figs	SIGNIFICANT FIGURES	SIGFIGS.SOP
QA	Subsampling	SUBSAMPLING	SUBSAMP.SOP
QA	Thermometers	THERMOMETER CALIBRATION, TEMPERATURE MONITORING, AND DOCUMENTATION	THERMA.SOP
QA	Training	TRAINING AND DOCUMENTATION	TRAINING.SOP
QA	Qualifiers	USE OF DATA QUALIFIERS	DATAQUAL.SOP
Sample Control	Bottle Prep	BOTTLE PRESERVATION	BTLPRP.SOP
Sample Control	Courier	COURIER	COURIER.SOP
Sample Control	Field Sampling	FIELD SAMPLING	FIELD.SOP
Sample Control	Manual Entry	MANUAL ENTRY OF SAMPLES FOR SAMPLE CONTROL	MANUALOG.SOP
Sample Control	Sample Control	SAMPLE CONTROL	LOGIN.SOP
WETCHEM	EPA 305.1	ACIDITY, TITRIMETRIC (EPA METHOD 305.1)	305_1.SOP
WETCHEM	EPA 3060A	ALKALINE DIGESTION PROCEDURE FOR HEXAVALENT CHROMIUM IN SOILS	3060A.SOP
WETCHEM	EPA 310.1/SM 2320B	ALKALINITY BY SM2320B, EPA METHOD 310.1	2320B.SOP
WETCHEM	EPA 350.3/SM 4500 NH3	AMMONIA POTENTIOMETRIC, ION SELECTIVE ELECTRODE	350_3r6.SOP
WETCHEM	EPA 405.1/SM 5210B	BIOCHEMICAL OXYGEN DEMAND / CARBONACEOUS BIOLOGICAL OXYGEN DEMAND (EPA METHOD 405.1/SM 5210B)	405_1.SOP
WETCHEM	EPA 7199/218.6	Determination of Hexavalent Chromium by Ion Chromatography--EPA Methods 7199 and 218.6	Cr6IC.SOP
WETCHEM	EPA 314.0	Determination of Perchlorate by Ion Chromatography--EPA 314.0	314_0.SOP

DEPARTMENT	Method	TITLE	FILENAME
WETCHEM	EPA 314.0 Modified	EPA 314.0 MOD. (DETERMINATION OF 4-CHLOROBENZENESULFONIC ACID (PCBSA) BY ION CHROMATOGRAPHY)	PCBSA.SOP
WETCHEM	EPA 160.2/SM 2540D	EPA METHOD 160.2/SM 2540D (TOTAL SUSPENDED SOLIDS; NON-FILTERABLE RESIDUE)	160_2.SOP
WETCHEM	EPA 160.3/SM 2540B	EPA METHOD 160.3 (TOTAL SOLIDS / PERCENT SOLIDS / PERCENT MOISTURE, GRAVIMETRIC, DRIED AT 103-105 C)	160_3.SOP
WETCHEM	EPA 160.4/SM 2540E	EPA METHOD 160.4/SM2540E (FIXED AND VOLATILES RESIDUE IN WATERS)	IR-WET-TVS
WETCHEM	EPA 300.0/9056	EPA METHOD 300.0 and EPA SW9056 (THE DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY)	300_0.SOP
WETCHEM	EPA 300.1	EPA METHOD 300.1 (THE DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY)	300_1.SOP
WETCHEM	EPA 330.5	EPA METHOD 330.5 (RESIDUAL CHLORINE)	330_5.SOP
WETCHEM	EPA 340.2/SM 4500F	EPA METHOD 340.2/SM 4500F (FLUORIDE BY POTENTIOMETRIC, ION SELECTIVE ELECTRODE)	340_2.SOP
WETCHEM	EPA 360.1/SM 4500O-G	EPA METHOD 360.1 / STANDARD METHOD 4500-O-G (DISSOLVED OXYGEN)	4500_OG.SOP
WETCHEM	EPA 365.3	EPA METHOD 365.3 (TOTAL PHOSPHORUS)	365_3.SOP
WETCHEM	EPA 410.4	EPA METHOD 410.4 (CHEMICAL OXYGEN DEMAND)	410_4.SOP
WETCHEM	EPA 415.1/9060/SM 5310B	EPA METHOD 415.1/SM 5310B OR EPA METHOD SW 9060 (TOTAL ORGANIC CARBON)	IR-WET-TOC
WETCHEM	EPA 420.1/9065	EPA METHOD 420.1/9065 (PHENOLICS, TOTAL RECOVERABLE)	420_1.SOP
WETCHEM	SM 5540C	EPA METHOD 5540C (ANION SURFACTANTS AS METHYLENE BLUE ACTIVE SUBSTANCES)	5540C.SOP
WETCHEM	EPA 7196A/SM 3500CR-D/EPA 3060A	EPA METHOD 7196A/STANDARD METHODS 3500-CR D (HEXAVALENT CHROMIUM, COLORIMETRIC + ALKALINE DIGEST (EPA 3060A)	7196A.SOP
WETCHEM	EPA 9030/9034/SM 4500S-F	EPA METHOD 9030/9034 / SM 4500S-F - ACID SOLUBLE/INSOLUBLE SULFIDES	9030_34.SOP
WETCHEM	EPA 9010B/9014/335.2	EPA METHODS 9010B, 9014 AND EPA 335.2 (TOTAL CYANIDE IN SOIL AND WATER)	9010_14.SOP
WETCHEM	EPA 130.2/SM 2340C	HARDNESS BY TITRATION EPA 130.2/SM2340C	2340c.SOP
WETCHEM	Various	Inorganic Calculations for Ion Balance, Langlier, Aggressive Index, Hardness, Unionized Sulfide, Larson-Skold Index, Sodium Absorption Ratio, Salinity	INORG_CALC.SOP
WETCHEM	LACSD 258	MERCAPTANS, TOTALS (LACSD 258)	258.SOP
WETCHEM	EPA 350.2/SM4500NH3 E	NITROGEN AMMONIA (TITRIMETRIC) (EPA METHOD 350.2/SM4500-NH3-B,E)	350_2r2.SOP
WETCHEM	EPA 120.1/SM 2510B	SPECIFIC ELECTRICAL CONDUCTANCE (EPA METHOD 120.1 / STANDARD METHOD 2510B)	120_1.SOP
WETCHEM	SM 2540G	STANDARD METHOD 2540G (TOTAL FIXED AND VOLATILE SOLIDS IN SOLIDS AND SEMISOLIDS)	2540G.SOP
WETCHEM	SM 4500CN-G	STANDARD METHOD 4500-CN-G/EPA 335.1/9010B (CYANIDES, AMENABLE TO CHLORINATION)	4500_CNG.SOP
WETCHEM	SM 4500CN-B,C,E	STANDARD METHOD 4500-CN~ -B,C,E (CYANIDES, TOTAL)	4500_CN.SOP
WETCHEM	SM 4500CO2	STANDARD METHOD 4500-CO2 (TITRIMETRIC METHOD FOR FREE CARBON DIOXIDE)	4500_CO2.SOP
WETCHEM	SM 4500CN-I	STANDARD METHODS 4500-CN, I - WEAK ACID DISSOCIABLE CYANIDE	4500_CNI.SOP
WETCHEM	EPA 376.2/SM 4500S2-	SULFIDE, COLORIMETRIC, METHYLENE BLUE (STANDARD METHOD 4500 S2-, EPA 376.2)	4500_S.SOP
WETCHEM	LACSD 253B	THIOSULFATE BY TITRATION (LACSD 253B)	S2O3.SOP
WETCHEM	SM5310C	TOTAL AND DISSOLVED ORGANIC CARBON (STANDARD METHOD 5310C)	5310C.SOP
WETCHEM	EPA 160.1/SM 2540C	TOTAL DISSOLVED SOLIDS, FILTERABLE RESIDUE (EPA METHOD 160.1/SM2540C)	IR-WET-TDS

DEPARTMENT	Method	TITLE	FILENAME
WETCHEM	SM4500-Norg-C	TOTAL KJELDAHL NITROGEN	4500NORG_C.SOP

Figure 20-1a.
Example - Demonstration of Capability Checklist

TestAmerica Irvine
Demonstration of Capability Checklist
Initial / Annual
(Circle one)

Employee: _____ Procedure(s): _____

Job Title: _____ Matrix: _____

Department: _____ SOP Name/Revision: _____

	Task	**Initials / Date Completed**
	Initial DOC Only	
1	Employee has read and understands the published procedure(s). (i.e. pH published methods: EPA 9040B, EPA 150.1 & SM 4500)	_____/_____
2	Employee has read, understands and agrees to follow the applicable SOP(s) without deviation.	_____/_____
3	Using the SOP as a ***step-by-step*** reference, the trainer has demonstrated the entire procedure to the Employee. *If any inaccuracies or contradictions in the SOP are discovered at this time, notify the area Supervisor and the QA Manager before proceeding further.*	_____/_____
4	Employee has performed the procedure under the direct supervision of an experienced staff member. **(including standard and reagent preparation and calibration where applicable)**	_____/_____
5	Employee has independently performed the procedure and results have been reviewed and confirmed by experienced staff member.	_____/_____
	QA only (*Note when the training took place.*)	
6	Trainer has completed a DOC for this method.	_____/_____
7	Trainer has read the Training SOP.	_____/_____
8	Employee has been trained on the Manual Integration and Data Integrity SOP (MANINT.SOP). Analysts only	_____/_____
7	Employee has been trained on Ethics and Data Integrity.	_____/_____
8	Employee has demonstrated capability by generating acceptable results on: _____ (4 LCS replicates, PT sample, Blind QC, etc.)	_____/_____

The employee named above has successfully demonstrated proficiency to perform the above mentioned procedure, maintain applicable QA/QC requirements, and report results on his or her own.

Employee Signature:_____ Date:_____

Trainer Signature (*if applicable*): _____ Date:_____

Supervisory Signature:_____ Date:_____

Lab Director/QA approval:_____ Date:_____

G:\Depts\QUALITY\TRAINING\CHKLIST7.DOC
rev.7, 10/17/07

Figure 20-1b.
Example - Demonstration of Capability Document

DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

Date: **Page 25 of 284**
Laboratory Name:
Laboratory Address:
Analyst(s) Name(s):

Matrix:
SOP# and Rev#:
Parameter:

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete, and self explanatory.[1]

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Technical Director's Name and Title	Signature	Date
Quality Assurance Manager	Signature	Date

[1] True: Consistent with supporting data.
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing.
Self-Explanatory: Date properly labeled and stored so that the results are clear and require no additional explanation.

Figure 20-2.

Example - New Method / Additional Analyte Checklist

New Method / Additional Analyte Checklist

The following items are **required** to be completed prior to the acceptance of client samples. Fill in any blanks that do not apply with "NA". Provide associated instrument QC when samples or QC samples are analyzed (includes run log).

New Method _____ Added Analytes _____

1_____ Standard Operating Procedure
- Note: For additional analytes, a **ROMD [or whatever an internal communication memo is named in your lab]** can be used to add the analytes, include RL and matrix.
  - _____ Analysis SOP
  - _____ Preparation SOP
  - _____ SOP for any other relevant process
  - _____ Pages from any applicable logbooks (instrument, standards, etc)

2_____Evaluation of Selectivity. As applicable: e.g. Retention Time Window Study, second column confirmation, Interelement correction checks, spectral or fluorescence profiles, etc.

3_____ Initial Calibration Curve (Include Tune verification or similar (e.g. degradation checks) if applicable)

4_____ Method Detection Limit (MDL) Study (summary and raw data)
- _____ Water
- _____ Soil
- _____ Other

5_____ Real Sample and MS, MSD ***(CA ELAP Requirement)***
- Tap Water for water only methods
- Local Soil sample for SW-846 methods (if applying for soil or soil/water)
- Local water sample may be used in lieu of tap water if it is a non- drinking water method
- Does not have to contain the target analytes

6_____ Reporting Limit Verification standard
- Spike a blank matrix at the RL and process through the entire method. MDL study should be able to be used if recovery is good. Note the spike level(s) and recovery(~~y~~ies)

7_____ Demonstration of Capability (DOC) per analyst (Precision and Accuracy (P&A) verification)
- 4 LCS for each matrix – most acceptance criteria are in the methods. The MDL study may be used if DOC criteria are met.
- Non-Standard methods – 3 x (1 LCS at LOQ-25%, 50%, 75% of the calibration range + Blank) prepared each day. (see NELAC Chpt 5, appendix C.3.3 (b))

8_____ Acceptable PT sample(s) if available

Notes: PT sample required for all new methods
PT sample required for all new analytes under NELAP

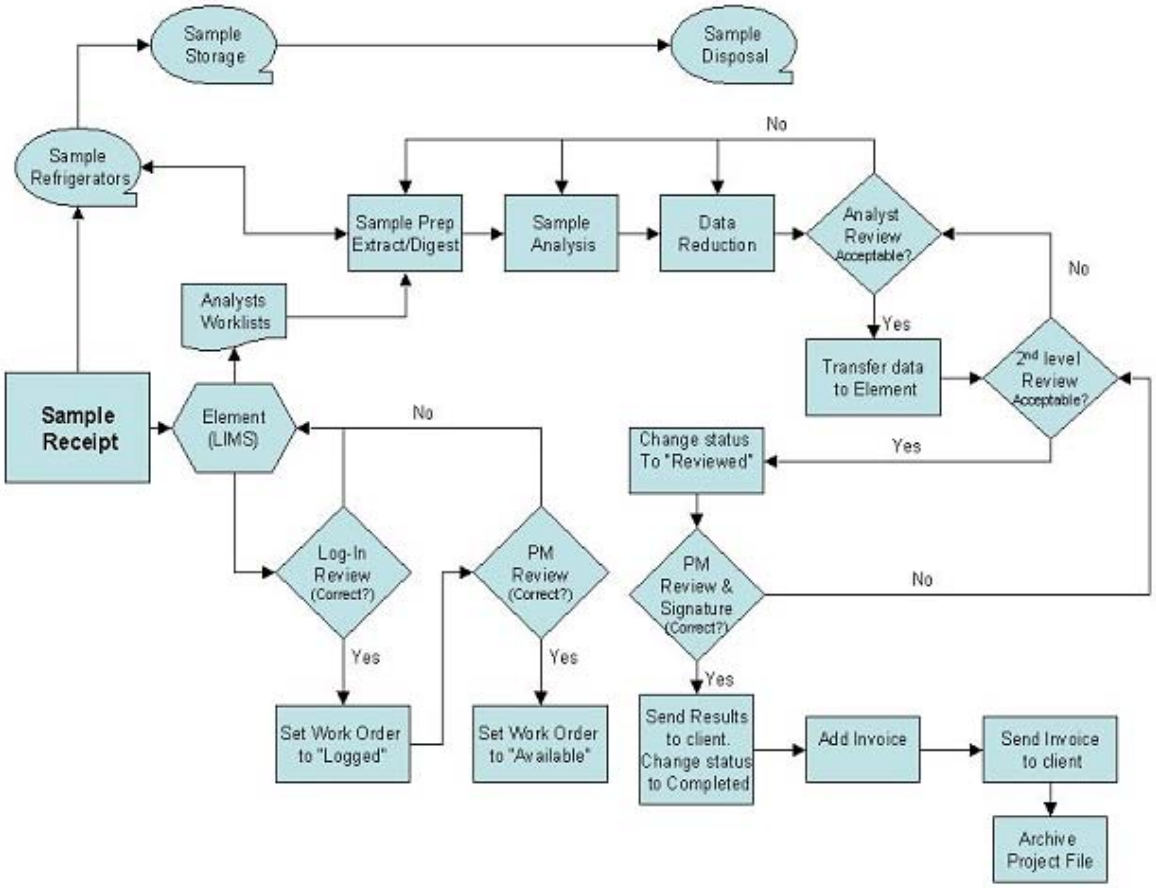
Submitted by _____ Date _____

9_____ Certification/Approval from Regulatory Agency where available.

QA Review / Acceptance _____ Date _____

Figure 20-3.

Work Flow



SECTION 21

EQUIPMENT (AND CALIBRATIONS
(NELAC 5.5.5)

21.1 OVERVIEW

TestAmerica purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs and are summarized in Appendix 4 of the QA manual. A list of laboratory equipment and instrumentation is presented in Table 21-1.

Equipment is only operated by authorized and trained personnel. Manufacturer instructions for equipment use are readily accessible to all appropriate laboratory personnel.

21.2 PREVENTIVE MAINTENANCE

21.2.1 TestAmerica Irvine follows a well-defined program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

21.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

21.2.2.1 Calibrations, routine maintenance, and adjustments are part of the analysts' and Department Managers' responsibilities. However, service contracts may be in place for some instruments to cover any major repairs.

21.2.2.2 High purity gases, reagents, and spare parts are kept on hand to minimize repair time and optimize instrument performance.

21.2.3 Table 21-2 summarizes the schedule for routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

21.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

21.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

21.2.4.2 Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.).

21.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled-in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

21.2.5 In addition, the maintenance records contain:

- The identification of the instrument/equipment (instrument's Serial Number and Model Number)
- The date the instrument/equipment was put into use.
- If available, the condition when the instrument was received (e.g. new, used, reconditioned).
- Routine maintenance procedures and frequency or a reference to their location in the method SOP(s).

21.2.6 If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses (refer to Sections 12 and 13).

21.2.7 In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted using the procedures outlined in Section 8.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

21.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

21.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains “calibration only” ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. The laboratory SOP BAL.SOP (Balance Calibration, Verification and Documentation) covers these procedures in greater detail.

21.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to $\pm$ 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

21.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer has increments of 0.2 ºC, and has a range applicable to all method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the laboratory's SOP THERMA.SOP (Thermometer Calibration/Temperature Monitoring and Documentation).

21.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

21.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are checked for accuracy at least quarterly. Glass micro-syringes with volumes of 500 µL or greater are checked for accuracy every six months.

The laboratory maintains a sufficient inventory of autopipettors, and dilutors of differing capacities that fulfill all method requirements.

These devices are given unique identification numbers, and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis (every six months for applicable syringes).

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any

quantitative measurements. See PIP.SOP (Pipet Calibration) for more details on pipettor, syringe, and dispenser calibration procedures.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

21.3.6 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated monthly by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 10%, the procedure is repeated and if the result is still greater than 10%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

21.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.
If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 13).

Note: Instruments are calibrated initially and as needed after that and at least annually.

21.4.1 CALIBRATION STANDARDS

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. However, the general procedures are described below.

21.4.1.1 For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods. If a reference or mandated method does not specify the number of calibration standards, the minimum number is three, not including blanks or a zero standard. All of the standard solutions are prepared using Class A volumetric glassware, calibrated pipettes, and/or microsyringes and appropriate laboratory quality solvents and stock standards.

21.4.1.2 Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to NIST whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. The laboratory uses its LIMS to document the following standard information: department, concentration, date of receipt, date of standard preparation, expiration date, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.

21.4.1.3 The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

21.4.1.4 The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The lowest calibration standard must be at or below the reporting limit. The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

21.4.1.5 Given the number of target compounds addressed by some of the organic methods, it may be necessary to prepare several sets of calibration standards, each set consisting of the appropriate number of solutions at different concentrations. The initial calibration will then involve the analysis of each of these sets of the appropriate number of standards.

21.4.1.6 All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

21.4.2 CALIBRATION FOR ORGANIC METHODS (GC, HPLC, GC/MS)

21.4.2.1 Many of the organic analytical methods utilize an internal standard calibration (GCMS and some GC). Because of the complex nature of the multipeak chromatograms produced by the method, some instruments necessitate the use of external standard calibration (most GC and HPLC). Surrogate compounds are included in the calibration processes for all appropriate organic analyses. For more details on the calibration types listed below, refer to SOP No. CA-Q-S-005, Calibration Curves.

21.4.2.2 Once the operating parameters have been established according to the method, each instrument is calibrated for the appropriate method. The analyst prepares five or more standard solutions at various concentrations containing all of the analytes of interest, internal standards, and surrogates that are appropriate for the method. Note: There are a several EPA methods that have different requirements and are exceptions (e.g. EPA 547) where a minimum of 3 calibration standards are prepared and analyzed.

21.4.2.3 The standard solutions are introduced into the instrument in the same manner as samples are; whether it be by direct injection, by headspace analysis, or by purge and trap. The calibration factor (CF) for methods that use external standards, and the response factor (RF) for methods that use internal standards are calculated for the five standards.

- External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the response to the amount of analyte in the calibration standard is defined as the Calibration factor (CF).

- Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. The use of specific internal standards is available in the method SOP.

Whichever internal standards are employed, the analyst needs to demonstrate that the measurement of the internal standard is not affected by method analytes and surrogates or by matrix interferences. In general, internal standard calibration is not as useful for GC and HPLC methods with non-MS detectors because of the inability to chromatographically resolve many internal standards from the target compounds. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

When preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes will vary. The internal standard solution will contain one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution (e.g., not all internal standards need to be at the same concentration in this solution). The mass of each internal standard added to each sample extract immediately prior to injection into the instrument or to each sample prior to purging must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts should be such that minimal dilution of the extract occurs (e.g., 10 uL of solution added to a 1 mL final extract results in only a negligible 1% change in the final extract volume which can be ignored in the calculations).

An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard should produce an instrument response (e.g., area counts) that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This should result in a minimum response factor of approximately 0.01 for the least responsive target compound. Refer to SOP No. CA-Q-S-005, Calibration Curves, for specific calculations.

21.4.2.4 Policies regarding the use of calibration standard results for creating the calibration curve are as follows:

- A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting level must be elevated to be the lowest calibration standard used for calibration.

- The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.
- Mid-calibration standards may not be excluded unless an obvious reason is found, i.e., cracked vial, incorrectly made, etc. The failed standard should be re-run immediately and inserted into the initial calibration. If not useful, recalibration is required.

21.4.2.5 Percent RSD Corrective Action

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

21.4.2.5.1 The first step is generally to check the instrument operating conditions. This option will apply in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance, changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.

21.4.2.5.2 If the RSD for any analyte is greater than the applicable acceptance criteria in the applicable analytical method (see also Appendix 4), the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

21.4.2.5.3 A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

Note: When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the method quantitation limit is at least as low as the regulatory limit or action level.

21.4.2.6 Alternatively, the least squares regression may be used to determine linearity. A five point line must result in a correlation coefficient (r) of 0.990 or better using the least squares method to be considered acceptable. In many cases it may be preferred that the curves be forced through zero (not to be confused with including the origin as an additional data point, which is not allowed). **Note:** EPA method 8000B does not allow forcing through zero however the agency has revaluated this position and has since changed this stance to allow forcing through zero. In addition, from EPA Method 8000C: "However, the use of a linear regression or forcing the regression through zero may NOT be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards.").

21.4.2.7 Instead of a linear curve model (either Average RF or least squares regression), a second order curve (Quadratic) may be used (and preferred) as long as it contains at least six data points. As a rule of thumb, if there is a consistent trend in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (COD or r^2) for the quadratic curve must be at least 0.99 for it to be considered acceptable. For more details on the calculations see Calibration Curve SOP CA-Q-S-005. Some limitations on the use of Quadratic Curve fits:

21.4.2.7.1 Care MUST be exercised to assure that the results from this equation are real, positive, and fit the range of the initial calibration.

21.4.2.7.2 They **may not** be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).

21.4.2.7.3 They **may not** be used to compensate for detector saturation. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve and try a 1st order fit or average RF.

21.4.3 Calibration for Inorganic Analyses

EPA Method 7000 from EPA SW-846 is a general introduction to the quality control requirements for metals analysis. For inorganic methods, quality control measures set out in the individual methods and in the *Standard Methods for the Examination of Water and Wastewater* (20th Edition) may also be included. Standard Operating Procedures for the analysis and the quality control documentation measures are kept in each department's SOP binder.

In general, inorganic instrumentation is calibrated with external standards. Some exceptions would be Inductively Coupled Plasma (ICP), Inductively Coupled Plasma Mass Spec (ICPMS), and Ion Chromatography Mass Spec (ICMS). These analyses may use an internal standard to compensate for viscosity or other matrix effects. While the calibration procedures are much the same for inorganics as they are for organics, CF's or RF's are not used. The calibration model in 21.4.2.6 is generally used for most methods, however in some instances the model from section 21.4.2.7 may be used. A correlation coefficient (r) of 0.995 or greater must be used to accept a calibration curve generated for an inorganic procedure. Correlation coefficients are determined by hand-held scientific calculators or by computer programs [state what your lab uses] and documented as part of the calibration raw data. Coefficients of calibration curves used for quantitation must be documented as part of the raw data. Curves are not allowed to be stored in calculator memories and must be written on the raw data for the purposes of data validation.

21.4.3.1 "Calibrations" for titrimetric analyses are performed by standardizing the titrants against a primary standard solution. See specific methods in *Standard Methods for the Examination of Water and Wastewater* (20th Edition) for more information.

21.4.3.2 Spreadsheets that are used for general chemistry calculations must have all cells containing calculations locked to prevent accidental changes to the calculations.

21.4.3.3 Instrument technologies (e.g. ICP) with validated techniques from the instrument manufacturer or other methods using a zero point and single point calibration require the following:

21.4.3.3.1 The instrument is calibrated using a zero point and a single point calibration standard.

21.4.3.3.2 The linear range is established by analyzing a series of standards, one at the reporting limit (RL).

21.4.3.3.3 Sample results within the established linear range do not need to be qualified.

21.4.3.3.4 The zero point and single standard is run daily with each analytical batch.

21.4.3.3.5 A standard at the RL is analyzed daily with each analytical batch and must meet established acceptance criteria.

21.4.3.3.6 The linearity is verified at a frequency established by the manufacturer or method.

21.4.4 Calibration Verification

The calibration relationship established during the initial calibration must be verified at periodic intervals as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration, and is not appropriate nor permitted in SW-846 chromatographic procedures for trace environmental analyses.

21.4.4.1 Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.

21.4.4.2 A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples.

21.4.4.3 The acceptance limits for calibration verifications can be found in each method SOP. As a rule of thumb: GCMS $\pm$ 20%, GC and HPLC $\pm$ 15%, Inorganics: $\pm$ 10 or 15%. Actual methods may have wider or tighter limits; see the method SOP for specifics.

21.4.4.4 If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF, RF or % drift values from the initial calibration to quantitate sample results.

21.4.4.5 If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new initial instrument calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

21.4.4.5.1 When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

21.4.4.5.2 When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

21.4.4.6 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

The Percent Difference is calculated as follows:

$$\% \text{ Difference} = \frac{(CF(v) \text{ or } RF(v)) - (\text{Avg. CF or RF})}{(\text{Avg. CF or RF})} \times 100$$

Where: CF(v) or RF(v) = CF or RF from verification standard

Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

$$\% \text{ Drift} = \frac{\text{Result} - \text{True Value}}{\text{True Value}} \times 100$$

The Percent Recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100$$

21.4.4.7 Verification of a Non-Linear Calibration

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations described in 21.4.4.6 above.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the laboratory's SOP for the specific method.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

21.5 POLICY ON TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it will not be reported as a TIC. If the compound is reported on the same form as true TICs, it must be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. See SOPs IR-MSV-8260 and IR-MSS-8270 for guidelines on making tentative identifications

21.5.1 The following guidelines for making tentative identifications are taken from EPA SW846 III edition, method 8260B.

21.5.1.1.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

21.5.1.1.2 The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

21.5.1.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

21.5.1.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

21.5.1.1.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

21.5.1.1.6 The concentration of any non-target analytes identified in the sample (see above) should be estimated. The same formulae as calibrated analytes should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

21.5.1.1.7 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

21.5.1.2 For general reporting if TICs are requested, the ten (10), largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard will be termed “Tentatively Identified Compounds” (TICs). More or fewer TICs may be identified based on client requirements.

21.5.1.3 TIC Reporting Limits

In general Reporting limits cannot be specified because of the unknown nature of the TIC. Any reporting limit that is reported can only be evaluated as an estimate as the quantitation is based on the assumption that the TIC responds exactly as the IS responds which is most likely not the case. In general, it is not recommended to set a Reporting limit at too low of a concentration as it gives a false impression.

TICs that meet the above identification criteria (Section 21.5.1) at 10% area of the IS: The RL would be 10% of the concentration of the internal standard used for quantitation. (e.g. 2.5 ug/L for 8260B, 4.0 ug/L for 8270C). In general, if the 10% area criteria is not met, the TIC RLs should be set at a level approximately 5x the level of the poorest performer in the analysis.

If a compound meets the TIC criteria, the reporting limit will reflect the ratio between the TIC and the IS or 5x the level of the poorest performer whichever is lower.

21.6 POLICY ON GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

21.6.1 The concentration of the BFB or DFTPP must be at or below the concentrations that are referenced in the analytical methods. Part of the purpose of the tune is to demonstrate sensitivity and analyzing solutions at higher concentrations does not support this purpose. Tune failures may be due to saturation and a lower BFB/DFTPP concentration may be warranted.

21.6.2 Tune evaluations usually utilize the "Autofind" function and are set up to look at the apex +/- 1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction cannot include any part of the target peak.

21.6.3 Other Options or if Auto Tune Fails:

21.6.3.1 Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. In this case, manually identify and average the apex peak +/- 1 scan and background correct as in 21.6.4 above. This is consistent with EPA 8260 and 8270.

21.6.3.2 Or the scan across the peak at one half peak height may be averaged and background corrected. This is consistent with Standard Methods 6200, EPA 624 and EPA 625.

21.6.3.3 Adjustments such as adjustments to the repeller and ion focus lenses, adjusting the EM Voltage, etc. may be made prior to tune verification as long as all of the subsequent injections in the 12 hour tune cycle are analyzed under the same MS tune settings and it is documented in the run sequence log and/or maintenance log that an adjustment was made. Excessive adjusting (more than 2 tries) without clear documentation is not allowed. Necessary maintenance is performed and documented in instrument log.

21.6.3.4 A single scan at the Apex (only) may also be used for the evaluation of the tune. For SW 846 and EPA 600 series methods, background correction is still required.

21.6.3.5 Cleaning the source or other maintenance may be performed and then follow steps for tune evaluation above. Note: If significant maintenance was performed, see methods 8000B or 8000C then the instrument may require recalibration prior to proceeding.

21.6.4 Tune evaluation printouts must include the chromatogram and spectra as well as the Tune evaluation information. In addition, the verifications must be sent directly to the printer or pdf file (no screen prints for DFTPP or BFB tunes). This ability should be built into the instrument software.

21.6.5 Since the limits are expressed in whole percentages, the results may be rounded to whole percentage before comparing to criteria when assessing the tune verification against the tune requirements. However, the comparison to the criteria is usually done automatically by the software and if the printout says “Fail” then there would have to be documentation of the hand calculation on the raw data and comparison to the criteria if the lab intends to still accept the tune. In most cases the analyst is better off performing an adjustment and rerunning the tune standard.

21.6.6 All MS tune settings must remain constant between running the tune check and all other samples. It is recommended that a separate tune method not be used, however a separate method may be used as long as the MS conditions between the methods are the same as the sample analysis method and tracked so any changes that are made to the analysis method are also made to the tune method.

Table 21-1. Laboratory Equipment and Instrumentation

Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Accelerated Solvent Extractor	Dionex	ASE 200	96040278	2000	NEW
Accelerated Solvent Extractor	Dionex	ASE 200	120362	2001	NEW
Accelerated Solvent Extractor	Dionex	ASE 200	97040463	2001	NEW
Accelerated Solvent Extractor	Dionex	ASE 200	96090216	2001	NEW
Accelerated Solvent Extractor	Dionex	ASE 200	99120782	2002	NEW
Accelerated Solvent Extractor	Dionex	ASE 200E	07090745	2007	NEW
Accelerated Solvent Extractor	Dionex	ASE 200E	07090746	2007	NEW
Air Concentrator	Entech	2000		1993	NEW
Ammonia Probe	Orion	96-12			Footnote 1
Atomic Absorption Spectrophotometer	Perkin Elmer	SIMAA 6000	5016	1995	NEW
Auto sampler	Dionex	AS40	98050117	2007	NEW
Auto Sampler (Archon)	O.I. Analytical	4552	12243	2001	NEW
Auto Sampler (Archon)	Varian	Archon	14636	2006	NEW
Auto Sampler (Archon)	Varian	Archon	14633	2006	NEW
Auto Sampler (Archon)	Varian	Archon	14634	2006	NEW
Auto Sampler (Archon)	Varian	Archon	14632	2006	NEW
Auto Sampler (Archon)	Varian	Archon	13171	2006	NEW
Auto Sampler (Archon)	Varian	Archon	14638	2006	NEW
Auto Sampler (Archon)	O.I. Analytical	4552	14418	2004	NEW
Auto Sampler (Archon)	Varian	Archon	14407	2006	NEW
Auto Sampler (Archon)	O.I. Analytical	4552	14417	2006	NEW
Auto Sampler (Archon)	Varian	Archon	14418	2006	NEW
Auto Sampler (Archon)	Varian	Archon	14195	2006	NEW
Auto Sampler (Archon)	Varian	Archon	13388	2006	NEW

Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Auto Sampler (Archon)	Archon		14411	2006	NEW
Auto Sampler (Archon)	Varian	Archon	14492	2006	NEW
Auto Sampler (Archon)	Varian	Archon	14637	2006	NEW
Auto Sampler (Archon)	Varian	Archon	14639	2006	NEW
Auto Sampler (Archon)	Varian	Archon	13389	2006	NEW
Auto Sampler (DPM)	O.I. Analytical	MPM 16		1993	NEW
Auto Sampler (DPM)	O.I. Analytical	MPM 16		1997	NEW
Auto Sampler (DPM)	O.I. Analytical	MPM/DPM 16		1993	NEW
Auto Sampler (DPM)	O.I. Analytical	MPM 16		1992	NEW
Auto Sampler (DPM)	O.I. Analytical	MPM-16		1993	NEW
Auto Sampler (DPM)	O.I. Analytical	DPM 16		2003	NEW
Auto Sampler (DPM)	O.I. Analytical	MPM 16			Footnote 1
Auto Sampler for GC	Hewlett Packard	7673A			Footnote 1
Auto Sampler for GC	Hewlett Packard	7673B			Footnote 1
Auto Sampler for GC	Hewlett Packard	7673B			Footnote 1
Auto Sampler for GC	Hewlett Packard	7673A			Footnote 1
Auto Sampler for GC	LEAP				
Auto Sampler for GC	Hewlett Packard	7673B			Footnote 1
Auto Sampler for GC	Agilent	7683			Footnote 1
Auto Sampler for GC	Hewlett Packard	18596M			Footnote 1
Auto Sampler for GC	Agilent	7683			Footnote 1
Auto Sampler for GC	Hewlett Packard	7673			Footnote 1
Auto Sampler for GC	Hewlett Packard	7673			Footnote 1
Auto Sampler for GC	Hewlett Packard	7673B		1993	NEW
Auto Sampler for GC	Hewlett Packard	7673B		1995	NEW

Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Auto Sampler for GC	Hewlett Packard	7673B		1993	NEW
Auto Sampler for GC	Agilent	7683		2003	NEW
Auto Sampler for GC	Agilent	7683		2005	NEW
Auto Sampler for GC	Hewlett Packard	7673B		1993	NEW
Auto Sampler for GC	Agilent	7683B	CN63340749	2006	NEW
Auto Sampler for GC	Hewlett Packard	18593B	3120A26939	1992	NEW
Auto Sampler for GC	Agilent	7683	CN42637490		Footnote 1
Auto Sampler for GC	Agilent	G2614A	CN55237971		Footnote 1
Auto Sampler for IC	Dionex	AS			Footnote 1
Auto Sampler for IC	Dionex	AS	96060542		Footnote 1
Auto Sampler for IC	Dionex	AS	3080145		Footnote 1
Auto Sampler for IC	Dionex	AS	3080145		Footnote 1
Auto Sampler for IC	Dionex	AS50	0411004Y	2002	NEW
Auto Sampler for IC	Dionex	AS50	99010302	2005	NEW
Auto Sampler for IC	Dionex	AS40	932811		Footnote 1
Auto Sampler for IC	Dionex	AS40	06110242	2007	NEW
Auto Sampler for IC	Dionex	AS50	00100242		Footnote 1
Auto Sampler for Metals	Perkin Elmer	AS-72	1464	1995	NEW
Auto Sampler for Metals	Perkin Elmer	CETAC	060019ASX	2001	NEW
Auto Sampler for Metals	Perkin Elmer	AS 91	913S3040101	1997	NEW
Auto Sampler for Metals	Perkin Elmer	AS 93	1075	2002	NEW
Auto Sampler for Metals	Perkin Elmer	AS 90	3380	1995	NEW
Auto Sampler for Metals	Perkin Elmer	CETAC	080002ADX	2004	NEW
Auto Sampler for Metals	Perkin Elmer	AS 91	6060	1995	NEW
Auto Sampler for Metals	Perkin Elmer	AS 91	3023	2006	NEW