S.C. Department of Health and Environmental Control

STATE OF SOUTH CAROLINA CERTIFICATION REQUIREMENTS FOR ORGANIC ANALYSES

In accordance with State Regulation 61-81, certification is required for all laboratories performing analyses to determine the quality of air, drinking water, hazardous waste, solid waste or wastewater required by or submitted to the South Carolina Department of Health and Environmental Control. Certification is presently offered under the Safe Drinking Water Act (SDWA), Clean Water Act (CWA) and Resource Conservation and Recovery Act (RCRA). Certification will also be offered as requested by the specific Program areas of SCDHEC for other analyses. Laboratories performing analyses for the organic contaminants must use only those methodologies approved by these regulations or specified by SCDHEC. Strict adherence to the approved methodology is a minimum requirement for certification under the South Carolina Laboratory Certification Program.

The EPA approved methodology for the Safe Drinking Water Act is referenced in 40 CFR Parts 141 and 142. The EPA approved methodology for the Clean Water Act is referenced in 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act". The methodology approved for RCRA analyses is referenced in 40 CFR Part 261 along with specified methodology in "SW-846, Test Methods for Evaluating Solid Wastes, Physical and Chemical Methods" required by SCDHEC.

To obtain certification with the South Carolina Laboratory Certification Program, visit our “How to Apply” website at: http://www.dhec.sc.gov/environment/EnvironmentalLabCertification/ELCHowToApply/

Initial and Continuing Demonstrations of Capability

An initial demonstration of capability (IDOC) is required for each method and analyte that the laboratory is seeking certification to perform. The IDOC is required for all methodology as an initial validation of the method and as a means to verify precision and accuracy of the analysts performing the methodology. The analyst is permitted to modify GC columns, GC detectors, GC operating conditions, continuous extraction techniques, concentration techniques, internal standards or surrogate compounds. Each time such method modifications are made the laboratory must repeat the IDOC described in the method. If the DOC is not repeated when these steps are modified or added all data produced by the modified method are considered invalid. This procedure is described in detail in the applicable methodology. Periodically, analysts should perform a continuing demonstration of capability (CDOC) to continually verify precision and accuracy of the analyst(s).

The DOC consists of an analysis of four replicate analyses with the pollutants of interest spiked into reagent water or clean soil matrix (e.g. Ottawa sand) at a concentration specified in the method. If the method does not specify a concentration, the laboratory is to use a concentration at 5 to 10 times the detection limit for the method. The laboratory is required to perform this procedure prior to practicing the method. Records of the DOC must be available for each chemist participating in an analysis to demonstrate the required precision and accuracy.

The mean recovery and the standard deviation between the four replicate determinations are compared with the method specified performance criteria. If the precision or accuracy test fails, the test must be repeated until the laboratory is able to meet the precision and accuracy requirements. For PCBs, the DOC must be performed using an early and late eluting PCB instead of performing it on all the PCBs.

The IDOC is required for all new analysts whether they are extraction analysts or the analysts performing the instrumental analyses to ensure that each analyst can meet the method specified accuracy and precision. The DOC must also be repeated when the laboratory modifies a method. The laboratory must demonstrate that the accuracy and precision specifications can be met with the modification; otherwise the modification is not permitted. This documentation must be maintained on file in the laboratory. If cleanup steps are employed, the accuracy and precision of this technique must be demonstrated in the DOC.

Quality Control

The laboratory must operate a formal quality control program and maintain records to document the quality of the data that is generated. The quality control practices documented in each method must be followed. Minimum quality control criteria are:

1) A laboratory reagent blank (LRB).
2) A laboratory control sample (LCS) or laboratory fortified blank (LFB). The LCS or LFB is spiked reagent water blank.
3) Internal standards (GC/MS).
4) Surrogate(s) if applicable to the method
5) Matrix spikes and/or matrix spike duplicates (MS/MSD)
6) Sample duplicates
7) Trip blanks (if applicable to the method).
8) Calibration initial and continuing verification (check standards)
9) Method Detection Limit (MDL) Study performed annually
10) Lower Limit of Quantitation (LLOQ) verification for each instrument (SW-846 methods)

Some methods contain additional quality control requirements that must also be included in the quality control program. These include but are not limited to verifying the GC/MS tune at least every 12 hours with BFB or DFTPP, analyzing a laboratory performance check solution or documenting Endrin and p,p'DDT breakdown. If the method requires additional quality control practices these must be documented.

As a general rule, the initial calibration for a method must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed using a calibration verification standard (QC check sample with some 600 series methods) prepared at the appropriate level of concern. Some methods may specify more frequent verifications and recommended standard concentrations. The 12-hour analytical shift begins with the injection of the calibration verification standard (QC check sample) and/or the MS tuning standard (BFB and/or DFTTP) 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. The calibration verification must fall within the method specified recovery limits, otherwise recalibration is required. Weekly at least one standard at the concentration of the reporting limit for each analyte must be analyzed to verify adequate instrument response.

Several of the 600 series wastewater methods specify the use of a Q.C. check sample to verify instrument calibration. When using these methods a Q.C. check sample must be used in place of the calibration standards. The Q.C. check sample must be from a source independent from the stock solution used to prepare the calibration standards. In the methods specifying the Q.C. check sample, the Q.C check sample must be analyzed at the beginning of each 12-hour shift and the results must meet the acceptance criteria "Q" as given in the applicable method.

LCS recovery limits specified in the method must be met. Drinking water and wastewater LCS recovery limits are specified in the analytical methods. SW-846 LCS recovery limits of 70-30% are specified in EPA 8000. Accuracy and precision data must be compiled by the laboratory with control limits established and updated periodically for matrix spikes, duplicates, and surrogates. Control limits must be documented with the analytical results. All quality control/assurance data must be readily available for review by the Laboratory Certification Program.

Laboratories must successfully analyze proficiency testing (PT) samples annually for those contaminants for which PT samples are available. The Water Pollution (WP) Study is used for certification of Clean Water Act and Solid and Hazardous Waste parameters and the Water Supply (WS) Study is used for the Safe Drinking Water Act parameters. Refer to the Proficiency Testing Requirements document for detailed information regarding PT studies.

Organic Record Keeping Requirements

Appropriate documentation must be maintained for all quality control practices and analyses performed by the laboratory. Without this required documentation the data are not legally defensible.

1. Instrument Calibration and Sample Analysis Records

   The laboratory must maintain the following information for all analyses performed under State Regulation 61-81. All records must be in ink and initialed or signed.

   1) Date and time of analysis
   2) Analyst performing the analysis
   3) Analytical method employed
   4) Unique laboratory sample I.D. number
   5) Instrument ID
   6) Detector ID
   7) Column ID
   8) Compilation of area responses versus concentration for each analyte
   9) Mean calibration factor, standard deviation, and the percent relative standard deviation for each analyte if using a calibration factor
   10) When using a linear calibration curve the slope, y-intercept and the correlation coefficient must be recorded for each analyte. Non-linear calibration models are unacceptable
   11) Percent error/relative standard error (RSE) checks for each calibration standard must be performed regardless of calibration model per section 11.5 of EPA 8000D (SW-846 methods).
   12) Daily GC and GC/MS Performance test results
   13) Daily instrument calibration check results
   14) Volume of sample purged or injected
   15) Extract volume of sample injected
   16) I.D. number of internal standard material
   17) Dilutions if performed
   18) Confirmations required
   19) Peaks must be labeled with retention time and contaminant I.D.
   20) Analytical results with the appropriate units
   21) Raw data generated by the analysis and the results
   22) All quality control data including standards, blanks, spikes, duplicates, surrogates, trip blanks, etc.
   23) Calculations
   24) Reference to the calibration curve used to generate results or data table for linear regression analyses
   25) All instrument operating conditions

26) Notations for unusual samples or other comments warranted are to be recorded. This includes clear documentation for manual integrations or any other changes made by the analyst to the original data.

When subcontracting analyses to another laboratory, a copy of the Certificate of Analysis from the originating laboratory must be retained on file. It is the responsibility of the laboratory to ensure that the contracted laboratory is certified for all analyses requested.

2. Sample Preparation or Extraction Records

Sample preparation or extraction records must be maintained for any analysis conducted by the laboratory performed under Regulation 61-81. This record must contain the following items as warranted by the method employed.

1) Method Number
2) Date and time of sample preparation or extraction
3) Unique sample I.D. number
4) Extraction or sample preparation technique
5) Extraction or sample preparation solvent
6) Volume or weight of sample used (include dry weight and wet weight of sample)
7) Sample preparation analyst
8) I.D. number of surrogate spiking mixture with concentration and volume added
9) I.D. number of spiking mixture with concentration and volume added
10) Sample clean-up and/or derivatization method employed
11) Date and time sample clean-up or derivatization employed
12) Clean-up/derivatization analyst
13) Final volume of extract and solvent
14) Comments

Some laboratories use a logbook to record sample preparation as a batch or have a separate sample preparation record for each sample. The instrument operator must be provided with a copy of the extraction or sample preparation records to be able to associate blanks, spikes and duplicates with the appropriate batch of samples. Laboratories must have a system in place by which blanks, matrix spikes and duplicates are identified and traceable to the applicable batch of samples. The instrument operator must also know the sample volume or weight prepared or extracted along with the final volume of sample submitted for analysis for determination of the analytical results.

3. Chemical Inventory Record

All laboratories are to maintain a chemical inventory record by recording the following information for the reagents/chemicals, standards, and solvents received by the laboratory.

1) Reference or identification number
2) Reagents/chemicals, standards, or solvents name
3) Vendor or manufacturer
4) Order or catalog number
5) Volume/weight
6) Lot number
7) Purity/concentration
8) Date received with initials
9) Storage location
10) Date opened and initials
11) Date of disposal and initials

This inventory record helps laboratories in estimating the use of certain standards, solvents and chemicals. All reagents/chemicals, standards and solvents are to be labeled with the date received and date opened and must be traceable to their use.

4. Standard Preparation Record

All standard preparations are to be documented. Records can be differentiated by type of contaminant such as volatile, base/neutral or acid extractable, pesticide/PCB, herbicide, etc. Laboratories divide each of these groups into concentrated stock standards by weight, standard mixes by dilution, and standards by dilution. An index can be maintained on all standards prepared by the laboratory with the section and page number for easy reference by the analyst.

Each standard received or prepared by the laboratory is to be assigned a unique standard I.D. number and this number must be printed on the vial label for the standard material. This number is to be referenced in analysis records and on chromatograms in order to verify the correct preparation for the standard material and to ensure that it has not exceeded its holding time. When multiple vials or containers of the same lot are received, each vial/container must be uniquely identified.

5. Instrument Maintenance Record

An instrument maintenance record is to be kept on each instrument used for regulatory analyses. This would include all preventive and corrective maintenance. By keeping an instrument maintenance record, analysts will be able to consult these records for changes made to the instrumentation that could be related to a change in resolution or sensitivity. The analyst can also use this record for troubleshooting if a certain problem had been encountered before.

The types of items that need to be included in this record include but are not limited to replacing carrier gases, traps, columns, detectors, septa, injector inserts, and filaments. Maintenance and cleaning of the detectors or ion source is also required to be documented. On a daily basis check column, detector, and injector temperature along with carrier gas pressure and carrier gas flow rate. If a change in retention time, resolution, or sensitivity is noted the analyst is to evaluate all instrument operating conditions to isolate and correct the problem. Always record the problem in the maintenance record and tell how the problem was corrected.

Major maintenance includes cleaning an ion source, cleaning quadrupole rods etc. which results in recalibration of the GC/MS. This will also include replacing the MS electron multiplier or any other faulty component.

A written log must be maintained on each instrument recording the following information.

1) Date of installation and serial number of each detector installed
2) Date of column installation and performance
3) Results of the column evaluation mixture (if applicable)
4) Documentation of column, septum or gas changes. This must include the repacking of the chromatographic column or replacing the glass wool. For capillary columns if the column is shortened or rinsed it must appear in the records.
5) For purge and trap apparatus, trap changes must be documented.
6) Any other maintenance performed on the gas chromatograph or gas chromatograph mass spectrometer by the laboratory analysts or by the authorized service representative.

Any major instrument modifications will result in the need to perform the required initial demonstration of the laboratory's capability. The MDL study must also be repeated.