

# **QUALITY ASSURANCE PROGRAM**

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## **INTRODUCTION**

To ensure the validity of the analytical data, an established, routine, and rigid quality assurance program is continually necessary to monitor the reliability (precision and accuracy) of the results reported and to control the quality so that it meets the program requirements for reliability. This manual describes the standard operating procedures and quality assurance/quality control program utilized by Analytical Consulting Services, Inc., to provide assurance that the quality control duties are being performed effectively.

## TABLE OF REVISIONS

<u>Revision No.</u>	<u>Page No.</u>	<u>Revised by</u>	<u>Date</u>	<u>Approved By</u>
Original	All			E. P. Williams
1	All	S. Rawn	6/14/94	E. P. Williams
2	All	E. Williams/ D. Parkin	6/10/95	E. P. Williams
3	15, 16	E. Williams	4/4/96	E. P. Williams
4	All	E. Williams	4/28/98	E. P. Williams

## I. OBJECTIVES

As an independent laboratory in today's society, Analytical Consulting Services' major function is to generate technical information. This information is put to many uses by our clients – to demonstrate compliance with a government regulation, to evaluate a raw material, to demonstrate quality of a finished product, or to decide a legal dispute. This information has a high and intrinsic value over and above the cost of providing it. Analytical Consulting Services is firmly committed to an extensive and strict program of Quality Assurance/Quality Control (QA/QC) which will assure that it provides the necessary quality to its clientele.

The main objectives of our Quality Assurance Program as directed by Corporate management are:

1. To establish quality control procedures for the determination of acceptable limits of precision and accuracy.
2. To ensure that quality control measures are being carried out.
3. To ensure accountability and traceability of the data.
4. To provide rigid guidelines for consistency and strict adherence to standard laboratory procedures adopted by Analytical Consulting Services.

The basic elements of our QA/QC Program are ***control, evaluation and correction.***

ACS Laboratory's QA/QC Goals:

1. Be the best in quality
2. Achieve the best quality through continuous improvement
3. Deliver, on time, a consistent product that meets customers' expectations and that instills their confidence in the laboratory's reliability and dedication to quality.

## II. STANDARD OPERATING PROCEDURES

The formal structure of our Quality Assurance Program is contained in a set of Standard Operating Procedures (SOP's), through which each specific topic is detailed individually. Copies of these SOP's have been provided to Laboratory Managers and are available to all laboratory personnel. Strict adherence to the correct procedure is required and documented in each of the SOP's, which are continually monitored and updated as changes or new developments occur. The following describes our current SOP's.

### A. Sample Receiving

#### **SOP Sample**

Sample Receiving and Shipping - A unique sample identification is assigned and Chain of Custody documentation is assessed as soon as it is received at the laboratory. The process of logging-in and distribution is described in this SOP.

#### **LIMS (Visual Lab Pro) Year 2000 Certified**

Century Compliant

Sample and data documentation and traceability

System capabilities (Client and sample archive database)

Generation of Certificates of Analysis

Generation of Chain of Custody (Receiving and Return Samples)

Generation of QA/QC data

### B. Approved EPA Guidelines

#### 1. **National Pollutant Discharge Elimination System**

The method analysis of National Pollutant Discharge Elimination System (NPDES) monitoring required by the Clean Water Act of 1977 are listed in 40 CFR 136.

#### 2. **Safe Drinking Water Act**

The EPA has established maximum contamination levels in national primary drinking water and required water suppliers to conform to these regulations and demonstrate compliance by testing for these contaminants.

#### 3. **The Resource Conservation Recovery Act (RCRA)**

This act was established to manage hazardous wastes. This is designed to protect ground water contamination from hazardous waste and also provide the framework for determining if a waste is

hazardous.

4. **ISO 9000**

ISO 9000 is a European series of guidelines for a global quality system, It consists of five standards for quality management and quality assurance: ISO 9000 through 9004. These standards were first issued by the International Standards Organization (ISO) in 1987.

C. Operational Requirements

**SOP Water**

Laboratory Pure Water Supply - Type I and Type IV deionized water are available in the laboratory. This manual describes the difference in the usage of the two and also the frequency of quality checks.

**SOP Reagents and Standards**

Chemical Reagents – All reagents and standards prepared in the laboratory are logged for the purpose of inventory and traceability.

**SOP Weight**

Weighing Instruments - Analytical balances are used throughout the laboratory. This manual describes the proper procedure of daily calibration and record keeping.

**SOP Glass**

Glassware – The accurate measurement of trace levels of pollutants requires clean and calibrated volumetric glassware. This manual describes the procedure of cleaning techniques for different types of glassware used for special parameters.

**SOP Temp**

Temperature Measurement – The calibration of thermometers for the accurate measurement of temperature required in the proper analysis of parameters is clearly described in this procedure. All temperature-controlled devices, including refrigerators and ovens, are monitored to insure proper storage temperature.

D. Instrumental Requirements

The following is a list of Standard Operating Procedures for the operation of instruments that must be measured and controlled to assure adequate performance.

**SOP pH**                      pH Meters

<b>SOP UV/VIS</b>	UV/VIS Spectrophotometers
<b>SOP COND</b>	Conductivity Meters
<b>SOP DO</b>	DO Meters
<b>SOP TOC</b>	TOC Analyzer
<b>SOP DCP</b>	Direct Current Argon Plasma Emission
<b>SOP ICP</b>	Inductively Coupled Plasma Emission Spectrophotometer
<b>SOP ICP/MS</b>	Inductively Coupled Plasma Emission Spectrophotometer (ICP/MS)
<b>SOP CVAA</b>	Cold Vapor Atomic Absorption
<b>SOP TPH-IR</b>	Total Petroleum Hydrocarbon Infrared Spectrometer
<b>SOP N-S</b>	Antek Nitrogen Sulfur Analyzer
<b>SOP USN</b>	Ultra-Sonic Nebulizer

### **III. INTERNAL EXTERNAL QUALITY CONTROL CHECKS**

Assurance of quality analytical results will be achieved by applying the following techniques to all analytical methods. In addition to the necessary calibration standards, blanks, duplicates, spikes, reference materials and/or control samples are to be included at the rate of 10% or one of each set of runs of less than ten (10) samples.

#### **A. Matrix Blank Analysis**

Blanks are to be included in analytical methods which lend themselves to this consideration. A blank is characterized as a sample included in the analytical process which has all the properties of the actual sample except that it does not contain the substance of interest. It could be necessary to create a bank which replicates the samples in terms of salt content or any

other variable. Blanks will be subjected to reagent additions and other treatments that the samples may receive during analysis.

B. Duplicate Analysis

Duplicate sample analysis is the analysis of the same sample twice in order to determine the precision of the analysis. The relative percent difference (RPD) between the two determinations will be calculated and compared to values determined by statistical analysis of historical data.

C. Matrix Spike Analysis

Spiked samples are samples fortified with a known amount of target analyte and subjected to the entire analytical procedure. The recovery of the method will be calculated for accuracy. The acceptance criteria is based on past information generated. A matrix spike duplicate can also be analyzed to yield precision information about the analysis as well.

D. Standard Analysis and Calibration

Inclusion of standards in analytical methods is necessary to quantify the amount of analytes present in the sample. It establishes a reproducibly reference point to which all sample measurements can be correlated. Calibration is the process for determining the correctness relative to physical or chemical standards.

A set of standards and a secondary source standard will be analyzed along with sample testing to verify calibration. In addition, a check standard shall be analyzed every 10 samples and at the end of analysis. This standard is necessary to verify the standard curve and may serve in some cases as sufficient for calibration.

1. Traceability of Standards

All inorganic and organic standards utilized for instrumentation/methodological calibration and preparation of quality control check samples shall be NIST traceable. Primary standards must be obtained from a reliable, certifiable source and be of the highest possible purity. Standards will be purchased from approved commercial vendors such as Chem Services, Inc., Fisher Scientific, Supelco, etc., for use in all laboratory analyses. Commercial standards prepared will be verified against certified Standard Reference Materials (SRM).

All standards must be stored under conditions and in containers that provide the greatest protection against deterioration and/or

contamination.

Stock and working standards solutions must be made fresh as often as required by their stability and as stipulated in procedure. Standards will be checked regularly for signs of deterioration (i.e., discoloration, formation of precipitates, and changes in concentration). Standards solutions will be properly labeled as to element or compound name, concentration, solvent, date, and preparer as dictated in SOP REAGENT.

## 2. Definitions of Standards

**Primary compound** is a liquid or solid compound in a pure form obtained from an approved commercial distributor.

**Stock standard** is a standard prepared or obtained from the primary compound (i.e., liquid or solid) at a high concentration.

**Working standard** is a standard used in the calibration and quantitation of the compounds of interest.

## 3. Expiration of Standards

All standards obtained or purchased from approved commercial sources, as well as those from EPA or NIST, are dated upon receipt. Date of expiration will also be noted and, if not available, will be obtained from the supplier or manufacturer. If no information is available from the supplier, the laboratory holding time or shelf life shall be one (1) year for most elements or compounds.

Standards prepared as stock or working stands will be properly labeled to include name of element or compound mixture, concentration, solvent, medium, date, and preparer.

Where applicable, laboratory control sample (LCS) or an independent check standard available from EPA, NIST, or SRM will be used to monitor standard and will be compared against the given target range values.

## 4. Sample Analysis

- a. All analyses are recorded in logbooks, worksheets or computer printouts. All runs performed on an instrument are

recorded in the logbooks, worksheets, or computer printouts and each run is numbered sequentially.

b. Calibration Curves and Standards

**Definition:** Calibration curves are prepared from the results of the analysis of at least two standards and a blank. Calibration curves are plots of the instrument response versus concentration. Typically, the plot will be linear. A plot is defined as linear if the correlation coefficient (R) calculated from linear regression analysis is 0.996 or greater.

**Application:** Each component being analyzed is standardized by analysis of at least two standards and a blank. The standard concentrations should be evenly distributed throughout the range of the method. The calibration data is plotted and applied to a least squares regression analysis.

$$m = \frac{n(xy) - (x)(y)}{n(x^2) - (x)^2}$$

$$b = \frac{y - m(x)}{n}$$

$$R = \frac{n(xy) - (x)(y)}{(n(x^2) - (x)^2)(n(y^2) - (y)^2)}$$

where: m=slope of line  
n=number of (x,y) pairs  
x=instrument response  
y=concentration of standards  
b=intercept of line  
R=correlation coefficient

The slope of the line is the response factor and the correlation coefficient is a measure of the fit of the line to the data (linearity). If the response is not linear, additional data points are required to define the response curve. The calibration curve is checked by analyzing standards. The response of the standards should be such that the response factor (slope) of the calibration curve calculated with the new data is within +/- 20% of the original response factor.

E. Quality Control Check Analysis

1. Reference Materials Analysis

Reference materials are samples which contain a known amount of target analyte. These may be prepared in the laboratory or acquired from any source other than the source of standards used for calibration.

This accuracy information is valuation because variables specific to sample matrix are eliminated, and it also ensures the correctness of the analysis.

2. Analysis of Controls

Control samples are similar to reference materials except that the true value of the target analyte is not known to the analyst. These types of samples, which may be purchased or prepared by laboratory managers, will be analyzed at least quarterly.

F. External Performance Evaluations

In order to verify that a laboratory possesses the capability to provide accurate and reliable test data in its day-to-day operations and to maintain high standards of performance, a competent, disinterested third party is necessary to evaluate laboratories based on personnel, physical facility, instrumentation, and quality assurance/quality control programs, and the laboratory's performance. To this end, we have actively sought certification and accreditation by organizations offering it in those areas relevant to our technical expertise.

G. Audits

Continuing evaluations of processes are reviewed through the use of control samples, replicate measurements, and use of reference materials in conjunction with control charts.

Audits conducted by the Director of Quality Assurance shall be inclusive of, but not limited to, the following parameters:

1. Verification of Standard Operating Procedures and analyst(s) understanding.
2. Verification and documentation of procedures and documents.

3. Review of analytical data and calculations
4. Review of analyst QA/QC data for accuracy, precision, completeness, representativeness, and comparability.
5. Review of instrument logs, performance test results and analyst performance
6. Review and performance indicators such as blanks, spike recoveries and duplicate/matrix spikes.

#### IV. DATA ASSESSMENT AND VALIDATION

Data assessment and validation incorporates a variety of techniques to evaluate the quality of the measurement process and the data generated. Parameters of major importance are precision, accuracy, completeness, representativeness, and comparability.

##### A. Precision

The laboratory uses duplicate analysis of samples, matrix spikes, and matrix spike duplicates to assess precision and accuracy. A sample duplicate and/or matrix spike duplicate will be analyzed for each 10 sample lot for in-house QC and are dependent upon the sample matrix and method of analysis. This analytical precision, Relative Percent Difference (RPD), is expressed as a percentage of the difference between the results of duplicate sample analyses or two matrix spikes for a given analyte. It is calculated as follows:

$$RPD = \frac{(MS - MSD) * 100}{(MS + MSD)^2}$$

where: RPD = Relative Percent Difference  
MS = Matrix Spike Result  
MSD = Matrix Spike Duplicate Result

B. Accuracy

The accuracy of measured data is evaluated by the comparison of the percent recovery of the spiked sample analysis and/or a QC reference material of known or established concentrations, independent of routine calibration. Statistically-based control limits have been established for each method of analysis and sample matrix. A spike analysis will be performed for each 10 sample lot, dependent upon the sample matrix, method of analysis, and concentration level. Recoveries will be assessed to determine method of efficiency and matrix interference effects.

Analytical accuracy will be expressed as the percent recovery of an analyte/parameter which has been added to the samples at a known concentration before preparation and analysis. The equation used to calculate percent recovery (%R) is as follows:

$$\%R = \frac{(SSR - SR) * 100}{SA}$$

Where:      %R = Percent Recovery  
              SSR = Spiked Sample Result  
              SR = Sample Result  
              SA = Spike Amount Added

C. Completeness

For the data to be valid, it must meet all the acceptance criteria, including accuracy, precision, and any other criteria specified by the analytical method used. Data validation procedures will be employed to eliminate incorrect data getting through data collection.

The specific accuracy and precision level will be dependent upon the type of sample matrix and the analytical method applied. The laboratory's historical statistical control limits will be used as guidelines to validate the data generated.

D. Representativeness

Data generated by the laboratory shall be representative of the overall population of samples collected and analyzed. It shall be representative

of the laboratory data base of accuracy and precision measurements for a given parameter, matrix and analytical method.

E. Comparability

Data generated shall be used to evaluate completeness of extensive monitoring programs and testing purposes based on historical data measurements of parameters, matrices, and analytical methods. Data shall be reproducible under similar conditions whether generated by Analytical Consulting Services or another laboratory.

F. Control Charts

1. Theory of Control Charts

The performance of a measurement system can best be demonstrated by the measurement of a stable and homogeneous control sample in a planned repetitive process. The data generated is plotted as a control chart to indicate whether the measurement is statistically in control. The control chart warns the laboratory of possible deviation from 95% confidence level by identifying errors, drifts, or other types of assignable variations.

2. Uses of Control Charts

- a. Provide a graphical representation of accuracy and precision for the analysis of each analyte and instant detection of erroneous data.
- b. Allow efficient observation of recovery trends for a particular analysis and provide long-term mechanism for self-evaluation of analytical data.
- c. Provide assessment of analytical capability of the staff chemist with regard to the output of valid analytical data.
- d. Allow observation of deviations from control trends.

3. Types of Control Charts

- a. Percent Recovery
- b. Relative Percent Difference

#### 4. Control Chart Preparation

The mean and the standard deviation of (%R) will be calculated. From this data, the upper and lower control limits and warning limits will be calculated.

$$\text{Upper Control Limit (UCL)} = \text{Mean of \%R} + 3s$$

$$\text{Lower Control Limit (LCL)} = \text{Mean of (\%R)} - 3s$$

where s denotes standard deviation

The (%R) of each Quality Control check sample, spike, or matrix spike duplicate will be plotted on a Shewart control chart and compared with statistically generated control limits.

$$\text{Warning Limit (WL)} = \text{Mean of (\%R)} \pm 2s$$

Data precision will be evaluated on the results of the samples analyzed in duplicate. The range will be calculated and then divided by the average of the two analysis, then multiplied by 100. This value equals the relative percent difference (RPD). The results of RPD will be plotted and the resultant point locations evaluated in terms of their deviation from historical values.

#### 5. Interpretation of Control Charts

The control chart distinguishes between random and assignable causes of variation. Each point on a control chart represents a test of "quality data" hypothesis. Critical to data validation and assessment is a thorough understanding of what constitutes an out-of-control situation. The following criteria will be used to evaluate out-of-control analysis:

- a. One or more points outside the control limit (3s).
- b. A run of two or more consecutive points outside warning limits (2s).
- c. A run of seven or more points above or below average, indicating trends or shifts.
- d. Non-random patterns in the data
- e. A run of five successive data points in the same direction

## G. Corrective Action

Whenever the analytical process is out-of-control, investigation/corrective action will be initiated by one or more of the following individuals:

The **analyst** must be able to recognize out-of-control conditions and immediately notify the Laboratory Manager for action plan.

The **Laboratory Director** must review all analytical and Quality Control data for reasonableness, accuracy, calculation errors, and completeness of records. In the event of an out-of-control analysis, the Lab Director will work with the analyst to solve the problem and prevent the reporting of suspect data by stopping the work in question and ensuring that all results that are suspect are repeated, if possible, after the source of the error is remedied.

The **Quality Control Officer (QCO)** must, in the event that an out-of-control situation goes unnoticed by the above personnel, notify the Laboratory Director, help identify and solve the problem where applicable, and ensure that the work is stopped on the analysis and no suspect data is reported.

### 1. Determining and Reporting Out-of-Control Data

- a. Factors that affect data quality (failure to meet calibration criteria, inadequate record keeping, miscalculations, improper storage, or preservation of samples) require investigation and corrective actions.
- b. Factors easily assessed through the use of control chart interpretation are those of shifts, trends, biases, and data outside of control limits.

The detection of any of the above conditions should be documented on the control chart and on a corrective action form, the out-of-control condition will then be investigated to determine whether the condition indicates a truly out-of-control situation or a possible random error. The Quality Control Officer shall document corrective action taken (i.e., whether the sample was re-analyzed/recalculated or whether the data was released for reporting to the client) on the corrective action form.

## 2. Corrective Action Implementation

Corrective action implemented will depend entirely upon the type of analysis, the extent of the errors, and whether the error is determinant or not.

Examples of determinant errors are:

- a. Instrumental failures, spectral interferences and sample matrix.
- b. Sample numbers mixed up
- c. Contaminated digesting solvent
- d. Analytical results out of specification

Examples of corrective actions:

- a. Re-digesting and analysis of a complete batch of samples due to contamination
- b. Re-digesting and analysis of out-of-spec samples in duplicate
- c. Re-calculation of data

Non-determinant errors will result in a complete re-analysis of sample batches, provided holding times are not exceeded. The resulting analyses will be investigated in a step-by-step method to isolate and correct faulty operations.

In all instances, the corrective action must be determined on a case-by-case basis. All corrective actions will be documented, and a compilation of known out-of-control situations and resolutions of problems will be established.

## 3. Documentation of Corrective Action

All out-of-compliance instances will be documented using a corrective action form. This form should be used by the Analyst, Lab Director and/or Quality Control Officer whenever an out-of-control situation is recognized. The report should include the following information:

- a. Description of out-of-control problem
- b. Dates (data recognized, date occurred, date corrected) and

- sample control numbers affected.
- c. Cause of problem - known or suspected
- d. Corrective action taken
- e. Resolution of problem
- f. Signature of the Quality Control Officer or Lab Director

#### H. Data Review and Validation

The review of data quality involves several levels of evaluation. In general, the analysts and the Laboratory Director will be responsible for reviewing and validating the data relative to instrument calibration, standard preparation, method blanks, raw data, calculations and transcriptions.

Additional verification by the Quality Control Office will be accomplished through routine audits of the data collection and flow procedures and by monitoring the results of Quality Control check samples.

The minimum review and validation requirements for each audit conducted by the Quality Control Officer.

1. Two point calibration plus blank point and one calibration check standard or as method appropriates.
2. Continuing calibration check using an EPA, NIST, or ASTM reference, if available.
3. Quality control check sample included in each analysis whenever possible.
4. One reagent blank per matrix and per concentration level for every sample batch analyzed (i.e., one for ten samples).
5. Matrix spike, and sample duplicates per matrix for every sample batch analyzed (i.e., one per 10 samples).
6. Review of sample documents for completeness by the analyst(s) at each step of the analysis.
7. Review of instrument logs, performance test results, and analyst performance.
8. Review of performance indicators such as blanks, spike recoveries, duplicate/matrix spikes.

9. Random calculation checks.
10. Review of data with emphasis on reasonability, spectral interference, significant figures, and analysis detection limits.

The Quality Control Office will do a complete audit of approximately 15% of the data generated. The emphasis will be on the data acceptability reliability to the data quality indicators and the accuracy of the final data summaries.

All analytical problems encountered during the sample analysis will be properly addressed, documented and resolved.

## **V. TRAINING**

In keeping with Analytical consulting Services' major objective to provide sound analytical data, we are committed to provide continuous training to our experienced personnel, as well as the familiarization and orientation of new employees in the principles of Quality Assurance/Quality Control.

- B. QA/QC Policy  
Every analyst is thoroughly trained in Analytical Consulting Services Quality Assurance, Quality Control Program as described in this manual.

- C. Training Guidelines

There are fundamental analytical techniques that apply to all individuals that work as laboratory personnel. Training SOP's ensure that the analyst understands the underlying principles of the analytical/measurement system and perform assigned tasks satisfactorily. Training SOP's that are based in sound statistical methodology for each method are used to evaluate the employee's ability to produce "quality" data.

- D. Safety

Analytical Consulting Services is committed to providing a safe and injury-free workplace for our employees. The Safety Manual describes the safety program at ACS, and incorporates by reference the ACS Hazard Communication Program and the ACS Chemical Hygiene Plan. In on-going training, employees are individually and collectively alerted to safety requirements.

Approved by:

\_\_\_\_\_  
Lab Director

\_\_\_\_\_  
Date

\_\_\_\_\_  
QA/QC Officer

\_\_\_\_\_  
Date