# Appendix C

Sample QA Plan Outline
Sample QA Plan-Vermont DEC
Laboratory QA Plan

# Appendix C – QA Plan Outline

**PART 1:** This document has been modified from a QA plan prepared by Paul Gray at the New Mexico Department of Health's Scientific Laboratory Division (SLD).

Title Page (Who are you – When was this written)
Signature Page (the Manual MUST be signed by everyone processing samples every year).
If you have more than a few pages, include a Table of Contents.

This "*Outline*" follows the format of the EPA Checklist that is used during on-site evaluations. The **ONLY** purpose of this "*Outline*" is to ASSIST development of a Quality Assurance Manual. Use the "*Outline*" portion (black letters) to lead you through the process. Replace the RED LETTERS with your own information and descriptions.

## 1. PERSONNEL

### *1.1 Supervisor/Consultant*

Name, Position within Organization, Training, Training relevant to Microbiological Water Testing.

### *1.2 Analyst (or equivalent job title)*

Name, Position within Organization, Training, Training relevant to Microbiological Water Testing.

### *1.4 Personnel Records*

This is a good place for the Organization Chart

## 2. LABORATORY FACILITIES

Describe the facility and equipment.

## 3. DATA QUALITY OBJECTIVES

This is the section where you describe the QA plan the lab will follow to produce valid and defensible data. Describe what QA procedures are used to validate sample results such as method blanks, calibration standards, laboratory fortified blanks, duplicates, matrix spikes, precision and accuracy control charts, etc.

## 4. LABORATORY EQUIPMENT AND SUPPLIES

This section is the Standard Operating Procedures (SOP) for all of the equipment used in your Laboratory.
Leave out any Item that is not used in your Laboratory.
Describe the Item, where it comes from (is it bought ready to use or prepared in your laboratory) its use, what Quality Control procedures are performed, how often and the preventive maintenance done - with a schedule.

***4.1 pH meter***

***4.2. Balance (top loader or pan)***

***4.3 Temperature Monitoring Device***

***4.4 Incubator Unit***

***4.5 Autoclave***

***4.6 Hot Air Oven***

***4.7 Colony Counter***

***4.8 Conductivity Meter***

***4.9 Refrigerator***

***4.10 Inoculating Equipment***

***4.11 Membrane Filtration (MF) Equipment***

***4.12 Culture Dishes (loose or tight lids)***

***4.13 Pipets***

***4.14 Glassware and Plasticware***

***4.15 Sample Containers***

***4.16 Ultraviolet Lamp (if used)***

***4.17 Other lab equipment such as DO meters, Drying ovens, computers and software, microscopes, desiccators, muffle furnaces, etc.***

## 5. GENERAL LABORATORY PRACTICES

Describe the procedures in your laboratory. If an Item is not relevant to your lab, leave it out.

***5.1 Sterilization Procedures***

***5.2 Sample Containers***

***5.3 Reagent-Grade Water***

### *5.4 Dilution/Rinse Water*

### *5.5 Glassware Washing*

### *5.6 General Laboratory Safety Procedures*

## 6. ANALYTICAL METHODOLOGY

This is where you will have a description of how samples are processed in your Laboratory – the Method SOP. Include descriptions (SOP's) for all procedures performed in your laboratory. You must follow Standard Methods and the EPA Manual, but everyone slightly alters the specifics of what is done. You MAY be stricter in your interpretation of the Regulations (sample must arrive at the laboratory within 24 hours rather than 30), but you can NOT be less strict (Incubator temperature ranges from 30-40°C).

### *6.1 General*

### *Media, chemicals*

- What media, chemicals do you use?
- Where does it come from?
- Where is it stored? For how long?
- How do you challenge the media? When?
- How do you dispose of outdated chemicals?

### *6.2 Membrane Filter (MF) Technique* (for total coliforms in drinking water (dw))

### *6.3 Multiple Tube Fermentation Technique (MTF or MPN)* (for total coliforms in dw)

### *6.4 Total suspended solids*

### *6.5 pH*

### *6.6 BOD*

### *6.7 Titration for chloride*

### *6.8 DO*

### *6.9 Heterotrophic Plate Count*

### *6.10 Total Dissolved Solids*

### *6.11 Nitrates*

### *6.12 Ammonia*

## 7. SAMPLE COLLECTION, HANDLING, AND PRESERVATION

### *7.1 Sample Collector*

If you do not collect samples, this Item is not relevant.

### *7.2 Sampling*

Even if you do not collect samples, you should have information available in the Laboratory to hand out to the people who do submit samples to you.

### *7.3 Sample Icing*

### *7.4 Sample Holding/Travel Time*

### *7.5 Sample Information Form*

Include a copy of your submission form.

### *7.6 Chain-of-Custody*

Not required in New Mexico, but a good idea to have written up IF you ever need it.

## 8. RECORDS AND DATA REPORTING

### *8.1 Legal Defensibility*

Are you doing all of the above AND documenting it AND:

### *8.2 Maintenance of Records*

- Keeping your Documentation?
- Where?
- For how long?

### *8.3 Sampling Records*

Refer to the copy of the Sample Submission Form.

### *8.4 Analytical Records*

What information do you maintain on a "Bench Sheet"? A copy of your Bench Sheet would be good.

### *8.5 Preventive Maintenance*

- What do you do to insure that your equipment works as it should?
- How do you document what has been done?

## 9. ACTION RESPONSE TO LABORATORY RESULTS

### *9.1 Notification of normal results*

- When are they notified?
- How are they notified?

### *9.2 Notification of Abnormal Results*

- Who so you notify?
- When are they notified?
- Is there documentation (Contact Log) of that notification?
- What information is kept on the Contact Log?
- How long is the Contact Log kept?

### *9.3 Notification of Invalid results*

- How does the lab invalidate data?
- What happens if invalid data is distributed accidentally?

### *9.4 Lab Certification*

- How does the lab maintain certificiation?
- Who has to be certified?
- What happens if the laboratory obtains unsatisfactory results from analysis of PE samples?
- What happens if the lab is decertified?

## 10. QC Checks and Their Frequency

This section describes what QC information is to be performed and documented. It could be included under the specific equipment indicated in SECTION 4.
This section could discuss Performance Evaluation samples, precision and accuracy procedures, calculations, use of control charts.

# PART 2: SAMPLE QUALITY ASSURANCE PLAN

This QA plan is a modification of the *Vermont Department of Environmental Conservation Laboratory QA plan*. This example is not meant to be an example that can be simply copied and used as your QA plan. The various sections should be customized for the laboratory analysis performed in your lab and the procedures you use. Each laboratory should prepare its own QA plan and SOPs. Most small labs will have a QA plan much simpler than this but should consider each area.

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## 2.0 INTRODUCTION

The City of _____ Laboratory Quality Assurance Manual documents or references documents that describe the laboratory policies and procedures as required by the State of New Mexico Surface Water Quality Bureau and is reviewed prior to the laboratory's annual on-site inspection. This manual can be found in the Office of _____

### 2.1 Objectives and Commitments of Management

The City of _____ relies on its laboratory to provide data, which is the scientific basis for completion of its NPDES and decisions impacting other environmental concerns. The Department's management is committed to provide the necessary resources to insure the laboratory can produce high quality data and maintain its State of New Mexico certification.

**Management's Quality System Policy Statement**

The City of _____ Laboratory is committed to providing consistent, high quality data in a timely manner. Since each analytical result will be used to make important program decisions, each analytical result is of critical importance. It is imperative that the City of _____ Laboratory generate and report data of known quality. Through our
Quality Assurance Plan we have outlined all procedures and precautions taken to insure that reported data is consistently of high quality. City of _____'s commitment to maintain the laboratory's quality objective is demonstrated by the following:

1. An expertly staffed and fully equipped laboratory facility
2. Successful participation in State of New Mexico's approved proficiency testing programs
3. Annual management review of each analytical process
4. Timely reporting of analytical results.
5. Laboratory test results which are supported by quality control data and documented
testing procedures.
6. Systems to inform management if analytical data does not meet all quality control
requirements.

This policy is communicated to all new employees and is constantly reinforced to all employees. It is implemented and maintained by employees at all levels. This policy is documented by management through employee evaluations, by formal reviews of the QA Plan and all revisions, training procedures, internal audits and document control.

## 2.2 Employee Code of Ethics, Training, and Reporting of Unethical Behavior

### 2.2.1 Employee Code of Ethics and Laboratory Fraud

Laboratory fraud is defined as, the deliberate falsification of analytical data or quality control results, where failed methods and contractual requirements are made to appear acceptable.
The City of _____ Laboratory management recognize that employee ethics have
a profound effect on the integrity and quality of the work performed at the City of _____ Laboratory. Policy regarding the acceptable handling, reporting and review of data are outlined in this Quality Assurance Plan (QAP) and Laboratory Standard
Operating Procedures (SOPs). Improperly performed procedures including all steps in calibration, analysis and data reporting, are not tolerated. Examples of unacceptable procedures include: falsifying data, improper calibration, misrepresenting quality control data, manipulation of computer software, data file substitution and concealment of known problems or unethical behavior or action

from laboratory management. Laboratory personnel must remain free of commercial or financial pressure which might influence their technical judgment.

#### 2.2.2 Ethics Training

New full time employees receive information sheets describing City of _____ Policies and Procedure. The policies set standards for Agency employees that include policies on conflicts of interest. Each employee must sign a statement that states they have received the information and they will be responsible for reading the material. Records are maintained with the Personnel Administrator. New laboratory personnel must complete an orientation program at the City of _____ laboratory. Documentation is kept in laboratory training files that states that new employees have read, understood and will use the latest version of the Laboratory's QA Plan and Laboratory SOPs which relates to his/her job responsibilities. Documents that must be read include:

Laboratory Quality Assurance Plan (QAP)
Laboratory Sampling Plan
Relevant Laboratory Standard Operating Procedures (SOPs)
Laboratory Safety Plan

Annual ethics training is required for each employee. Documentation of the training is retained in each employee's continued demonstration of ability folder. The training focuses on issues arising from activities such as hiring, training and supervising staff; handling, analysis and reporting of quality assurance data and legal responsibilities including the potential punishments and penalties for improper, unethical and illegal actions. Annual training reinforces the policies and procedures outlined in the QA Plan. Training may be conducted internally or by an outside source.

#### 2.2.3 Reporting of Unethical Behavior

Employees are required to report any suspected unethical activities to Laboratory management. The reporting can be in writing or verbal. Unethical situations can be reported anonymously through the interoffice mail system. Reporting can be to the Laboratory Director, Supervisor, QA Officer or any other designated person. It then becomes the responsibility of that individual to initiate corrective actions which may include reporting the incident to upper management or the Department of Personnel. Each employee involved in the reporting and receiving of reported information must document the incident, actions taken, information reported and individual the incident was reported about and reported to. This ensures that the individual reporting any suspected unethical behavior has evidence that they have acted appropriately.

## 3.0 LABORATORY REVIEW OF REQUESTS FOR TESTING

### 3.1 Lab Contracting Policy

The City of _____ Lab and clients requesting environmental analysis require an agreement that is written and signed by the project manager and Laboratory Supervisory Issues addressed are:

1. Tests requested, required reporting limits.

2. Turn around times.
3. Enforcement administrative procedures required (Y/N).
4. Method of sample delivery.
5. Laboratory responsibilities for sample preparation.
6. Lead time needed to schedule sample delivery and to secure sampling bottles.
7. Analytical method used. Existing methods have demonstration of lab capability in place. New procedures will require method development and an initial demonstration of lab capability documentation be in place before samples are accepted.
8. Laboratory input on applicability of methods to sampling planned.
9. Report deliverables.

The contract will include a copy of the Laboratory's State certification status. The final agreement will notify the client if the Laboratory possesses the necessary physical, personnel and information resources that are necessary to meet the clients needs. The Laboratory must notify the client in writing if the Laboratory's certification status changes during the life of the contract or if the contract needs to be amended after work
has commenced.

### 3.2 Subcontracting of Analytical Work

Other than providing solicited advice the Laboratory is not involved with program contracts with private laboratories.

## 4.0 LABORATORY ORGANIZATION AND RESPONSIBILITY

The City of _____ Laboratory is an internal service organization, which is charged with providing analytical support to all city departments.

The City of _____ laboratory is organized into three analytical areas:

**The Metals Analysis Section** is responsible for analyzing metals in a wide variety of matrices. It supports a number of Departmental programs including industrial pretreatment, landfill assessment, hazardous waste investigations and lake sediment/fish studies. The center employs ICP/MS and a mercury cold vapor system as the methods of analysis.

**The Inorganic Chemistry and Microbiology Section** is responsible for all non metal/organic analyses performed at the Laboratory. The center supports a number of diagnostic water quality studies, landfill assessments, and departmental investigations. The center supports the monitoring of the wastewater treatment system and swimming water. Analyses are performed using auto analyzers, ion chromatography, a variety of manual chemistry and microbiology methods.

**The Organic Chemistry Section** provides identification for organic materials in a variety of matrices including water, fish, solids and air. The center provides the full complement of organic contaminant analysis required by department programs. These analyses include volatiles, carbonyls, PCBs, and pesticide compounds. Analyses are performed using gas chromatography, gas chromatography-mass spectroscopy and high performance liquid chromatography.

The laboratory analytical sections require considerable administrative support, including a laboratory supervisor, secretary, personnel to manage the safety and quality assurance plans, and laboratory technicians that assist in managing glassware, bottle orders and simple analyses.

The laboratory has 6 permanent positions, and when the budget allows, seasonal technician position(s). The administrative center is assigned 2 positions. The remaining personnel work in the analytical sections. The laboratory director is responsible for all aspects of analysis within their section: instrument control, technical method development, equipment purchases, quality control, supervision and training of seasonal technicians and workload management. Technicians assist in each work sections as work loads require. Position descriptions outlining education and experience requirements are available upon request. In order to ensure that data is of acceptable quality all data is subject to review. The laboratory data review process is described in Section 10.2. Individuals responsible for ensuring data is valid and for routinely assessing measurement systems for precision and accuracy are listed below.

## 4.1 Laboratory Users

### 4.1.1 Program Directors, Project Leaders

- Are responsible for providing the Laboratory and EPA (federally funded projects) with a QA Project Plan which identifies and defines data quality needs in terms of appropriate analytical levels; contaminants and levels of concern; required detection limits; critical samples; and completeness, comparability and representativeness requirements.
- Responsible for scheduling the collection of additional samples to assess precision for matrices the Laboratory has not routinely analyzed and collecting appropriate field quality control samples (Section 11.1).
- Review data for errors and outliers as it becomes available.
- Oversees the sampling process to ensure field personnel are following proper sample collection and preservation steps. Responsible for providing written standards on operating procedures for all aspects of field work.
- Periodically assess data and initiates corrective action when analytical results do not provide useable data i.e. quantitation level is unacceptable for a particular set of low level samples; unacceptable field duplicate, split

sample or instrument blank results or data does not conform to required accuracy, precision or completeness requirements.

- Notfies the Laboratory when Chain of Custody (COC) will be required on a sample set, preferably before the sampling event when possible. Responsible for maintaining proper sample handling, and delivery of COC samples.

## 4.2 Laboratory Positions and Job Duties

### 4.2.1 Laboratory Supervisor

- Responsible for the overall technical quality of the work performed in the Laboratory and for assuring the use of standard methods. Supervises all personnel employed by the Laboratory. Assures that the Laboratory has sufficient personnel having the necessary education, training and technical knowledge and experience for their assigned duties. Assures that the Laboratory has appropriate equipment and supplies.
- Assures that the laboratory has the capacity, facility and resources to perform new work.
- Acts as liaison between laboratory and regulatory agencies (NMSWQB, EPA) and laboratory users.
- Oversees the scheduling of projects and the completion of tasks within the required time schedule and sample hold times. Monitors progress of projects and communicates with laboratory staff and users as required.
- Oversees the transformation of analytical data which may be necessary to meet program needs.
- Provides technical assistance to laboratory users in regard to the selection of appropriate analytical and/or sampling methods. Review QA plans.
- Provides technical assistance to laboratory staff regarding QA problems and method and instrument selection. Reviews laboratory standard operating procedures. Generates and maintains current Standard Operating Procedures (SOPs) for laboratory operation within his/her work area. Assures that all referenced method requirements are part of the SOP. Assures that all SOPs are appropriately detailed for personnel performing a method or step of a method and SOP protocol is followed.
- Reviews all data before it is reported as final. Assures that results from different parameters of a sample correlate.
- Maintains a sample custody and tracking system within the laboratory.
- Performs internal performance evaluation standards.
- Maintains the supply of sample bottles used for sample collection.
- Oversees the Chain of Custody (COC) sample transfer into the laboratory and assures that data handling and COC records are organized and accessible.

### 4.2.2 Laboratory Technician

- Is responsible for the technical quality of work performed.
- Completes required Demonstration of Ability protocols for all procedures/methods prior to undertaking independent analysis.

- Remains current on equipment and methods used in the analysis of samples within their analytical section. Is capable of providing insight into equipment purchases and analytical methods.
- Is capable of resolving technical problems encountered in the analysis of samples.
- Responsible for ordering all consumables needed for methods performed and assuring they meet standards.
- Responsible for equipment maintenance and maintenance contract oversight
- Maintains quality assurance documentation on procedures, equipment, reagents and standards. Initiates corrective action when quality assurance data does not meet pre-established control and warning limits.
- Participates in Interlaboratory Performance Evaluation studies.
- Assures that all data generated is properly reviewed and that all reviewed data meets internal acceptance criteria or is properly flagged.
- Responsible for the generation of data packages that contain all relevant information needed to reproduce a result and for the maintenance of both paper and electronic copies (when applicable) of data for methods performed.
- Reviews Laboratory Quality Assurance Plan revisions and follows protocols and procedures outlined within the Plan.
- Follow SOPs and QA/QC requirements of methods and the Laboratory.
- Informs immediate supervisor when precision and accuracy values are beyond established warning and control limits. Maintains QA/QC records for tests performed.
- Assists in data review for his/her analytical area.
- Is responsible for providing clean glassware and sample containers.
- Monitors the temperatures of refrigeration units, calibrates analytical balances and monitors indicator lights on the Laboratory water system on a daily basis.
- Prepares containers and other sampling items needed by samplers.

## 5.0 QUALITY ASSURANCE OBJECTIVES

Analytical precision and accuracy are assessed through the analysis of reference standards and laboratory generated quality control samples such as analytical duplicates, matrix spikes, and matrix spike duplicate samples. The accuracy and precision of data are related to the procedures used to analyze samples and generate data, the sample matrix being analyzed and the sample concentration. When a method does not specify limits, upper and lower control limits are established using historical Laboratory data as a guideline. When insufficient data is available, default limits are used. Laboratory quality assurance objectives for analytical data in terms of reporting limits (practical quantitation limits) and precision and accuracy, are listed by compound, method in Tables 5.1 Section 14 describes how data quality indicators are calculated. Precision and Accuracy objectives listed are based on upper and lower control limits.

Data outside the upper and lower control limit is flagged and the laboratory supervisor notified. The lower reporting limit for a method is listed as the Practical Quantitation Limit (PQL). The PQL is typically 2 to 10 times the calculated MDL. If accuracy is a problem at the calculated PQL due to background levels of contamination, sample dilution or other issues the PQL will increase. The low calibration standard must be at the method reporting limit (PQL) or lower. Results reported outside the calibration range must be flagged. Occasionally data is reported when the accompanying quality control data are not within established quality control limits, sample hold times have been exceeded, Laboratory clients request data be reported below the Laboratory's established reporting limits or for some other reason the data is not to standard. Sample remark codes are used to alert the data end user to the fact that analytical data accompanied by a remark code may not be appropriate for the intended use.

**Sample Remark Codes ***

| **Remark Code** | **Description** |
|---|---|
| BH | Reported value may be biased high. |
| BL | Reported value may be biased low. |
| C | Colony count outside ideal range. |
| D | Dilution resulted in instrument concentration below PQL. |
| H | Hold time exceeded, value may be in error |
| I | Matrix Interference |
| N | Not processed or processed but results not reported |
| O | Outside calibration range, estimated value. |
| OL | Outside Limit |
| P | Preservation of sample inappropriate, value may be in error. |
| S | Surrogate recovery outside acceptance limits. |
| T | Time not provided or time/date discrepancy |
| W | Sample warm on arrival, no evidence cooling has begun |

* Codes may also be used to qualify quality control data.

" BH: The reported value may be biased high" is used if the analyst determines or suspects that a sample or method bias has elevated the reported values. All samples of the same matrix may be flagged if a representative sample shows a matrix effect.

"BL: The reported value may be biased low" is used if the analyst determines or suspects that a sample or method bias has suppressed the reported values. All samples of the same matrix may be flagged if a representative sample shows a matrix effect.

"C: Colony count outside ideal range" is used when microbiology membrane filtration counts are not within the method recommended ideal counting range.

“D: Dilution resulted in instrument concentration below PQL” is to be used when dilution is necessary to eliminate an interference for the parameter being reported. The interference can be a chemical or physical interference. For multi-parameter methods analysis and reporting from more than one dilution may be required if the interference is only compromising a portion of the chromatography.

“H: Hold Time is exceeded” is used to flag a result when sample or extraction method specified holding times are exceeded.

“I: Matrix Interference” is used if Laboratory data quality objectives can not be met due to a matrix interference and the analyst can not determine if the bias is high or low.

“N: Not processed or processed but results not reported”. The use of this code requires that the parameter related comment field be completed. The “comment field” may list one of the following comments or others: Lab Accident, Sample arrived in unacceptable container, Sample not properly preserved, Insufficient sample volume, Hold Time exceeded, Associated quality control data unacceptable.

“O: Outside calibration range, estimated value” is to be used for reported results that are above or below the calibration standards. It is not used if a sample dilution is made and the diluted sample result is within the calibration range. Laboratory policy requires that sample results be bracketed by standards. If there is insufficient sample volume or for some other reason the sample can not be diluted and reanalyzed to bring a result(s) within calibration range the result must be flagged. A high standard analyzed at or above the concentration of the sample with recoveries within Calibration Verification Criteria may negate the need to flag the data.

“OL: Outside Limit” is used to flag data that is outside the precision or accuracy criteria established by the Lab, or referenced method.

“P: Preservation of sample inappropriate, value may be in error” is to be used when there is a decision made by the Lab to analyze an inappropriately preserved sample rather than reject the sample.

“S: Surrogate Recovery outside acceptance limits” is used to flag surrogate recoveries that are outside laboratory acceptance criteria.

“T: Time not provided” Analysis with a hold time of <72 hours must have the sampling time entered at log-in and the analysis time entered by the analyst. Samples with inadequate time of arrival documentation will be flagged if a decision is made by the lab to analyze an inappropriately labeled sample rather than reject the sample.

“W: Sample warm on arrival, no evidence that cooling has begun” is to be used when samples requiring cooling arrive and are not on ice.

## 6.0 SAMPLE HANDLING

### 6.1 Sample Collection

The laboratory is responsible for collection departmental samples (WWTP). All other departments must properly collect and deliver samples to the laboratory. The laboratory does not accept samples collected by the general public. Table 1060:I in 20th edition Standard Methods for Examination of Water and Wastewater describes the required containers, preservation and holding times for the parameters analyzed at the laboratory. Sample collection and handling protocols are available for microbiological samples.

### 6.2 Sample Receiving

Most samples are delivered, logged into the Laboratory Logbook, labeled, preserved, subdivided, filtered if necessary and stored in refrigeration units by laboratory personnel responsible for the collection of the sample(s). Sample temperature upon delivery is monitored and the temperature of a representative sample is recorded in the logbook. Dedicated refrigeration units are used for samples that could become contaminated. Sample log in instruction and chain of custody sample handling instructions are described in Section 7.0. A limited number of samples arrive by courier and are logged in at the time of arrival. The Sample Log-In Sheet is used by field personnel to record required information. The Laboratory secretary, supervisor or analyst: insures that required information is provided, documents whether samples requiring thermal preservation have been iced, records the temperature of a representative sample, and labels the samples with the lab numbers. Chemical preservation of samples delivered by courier is generally performed at the laboratory by laboratory staff, however some samples require field preservation. When preservation or sample manipulation (filtration, subdividing) is performed by laboratory staff, a record is made of the processing steps.

### 6.3 Sample Preservation

#### 6.3.1 Temperature Preservation

Samples requiring thermal preservation will be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice for samples that are delivered to the laboratory the day of collection. If samples require thermal preservation and they are not delivered on the day of collection they will be considered acceptable if the arrival temperature is either within 2°C of the required temperature or the method specified range. For example if the specified temperature is 6°C, sample temperature ranging from above freezing to 6°C is acceptable.

#### 6.3.2 Chemical Preservation

In most instances chemical preservation can be initiated upon delivery of samples to the Laboratory, if samples are delivered the day of collection. If field preservation is required the sample container provided will contain the required preservative. If samples have not been field preserved lab staff must preserve

the samples at the Lab following protocols outlined in Table 1060:I in 20th edition Standard Methods for Examination of Water and Wastewater . All samples requiring acid preservation must be tested for proper pH prior to or after analysis. Preservation is verified by the analysts and documented on bench sheets or in laboratory notebooks. If a sample(s) was not properly preserved and if the sample has not been analyzed the requesting department may be notified before proceeding if practical (metals samples are an exception, policy is to preserve and wait 16 hours to analyze). If the sample was not properly preserved and the department requests that sample results be released, the analyst must flag data with a "P-preservation of sample inappropriate, value may be in error". The laboratory Supervisor must document in writing the clients decision to report results that do not meet acceptance criteria.

## 6.4 Sample Acceptance and Rejection Policy

### 6.4.1 Sample Acceptance

#### 6.4.1.1 Required Information:

Samples submitted to the laboratory must be accompanied with the following information either electronically or by completing a Sample Log-In Sheet if samples are to be logged in by laboratory staff:

- Customer Identification
- Sample Collector
- Date of Collection
- Time of Collection (time must be entered for all tests)
- Collector's Name
- Preservation
- Sample Matrix (solid, wastewater, drinking water, etc.)
- Customer Sample Identification(s)
- Requested Tests
- Sample Remarks
- Have samples arrived on ice? Yes/No
- Temperature of representative sample ($^{o}$C)

Chain of Custody samples (COC) must follow protocol outlined in the Laboratory QA Plan: Section 7.2 Chain of Custody Procedures. A lab COC Form must be submitted with the samples. If protocols are not followed the laboratory supervisor or his designated representative will refuse samples unless written instructions from the client instruct the laboratory to proceed with analysis. The final Lab Report will document irregularities and the client's decision on how the Lab was instructed to proceed i.e. discard samples or analyze and report results with a qualifier.

#### 6.4.1.2 Sample Labeling:

Samples must be clearly labeled with a unique identification. Labels should be water resistant and indelible ink used. At sample Login the laboratory will label samples with a unique bar code that allow analysts to enter Sample ID #s into laptop spreadsheets or analytical instrument data bases.

#### 6.4.1.3 Sample Containers:

Samples must be collected in appropriate sample containers provided by the laboratory as described in Table 1060:I in 20th edition Standard Methods for Examination of Water and Wastewater. If the Laboratory Supervisor accepts a sample in a container not provided by the lab a Sample Note or Order Comment must be added to the bench sheet.

#### 6.4.1.4 Sample Holding Times:

Samples must be delivered to the Laboratory to allow sample analysis to be completed within sample hold time. Samplers must schedule pH and chlorine (analyze immediately), Microbiology (6 hour hold time for wastewater samples), BOD5 (24 hour hold time). Sample holding times for other parameters are listed in the Table 1060:I in 20th edition Standard Methods for Examination of Water and Wastewater.

#### 6.4.1.5 Sample Volume:

Appropriate sample volume must be provided. Containers, which are provided for each test, will provide the appropriate volume if filled. Instructions on filling sterile microbiology bottles, total phosphorus tubes, and volatile vials (solid and water) must be followed. An additional sample volume may be required for some tests to provide the Laboratory with the sample volume required to perform required QC. Samplers are instructed to collect an extra volume of sample and designate the sample as a duplicate and/or matrix spike sample.

6.4.1.6 Sample Preservation:

Sample preservation protocols outlined in Table 1060:I in 20th edition Standard Methods for Examination of Water and Wastewater must be followed.

### 6.4.2 Sample Rejection:

If a sample is received that is not suitable for testing or insufficient details are provided for the laboratory to proceed the requesting department must be consulted for further instructions before proceeding with analysis.

The laboratory supervisor determines if submitted samples are to be rejected or results not reported for samples that have already been processed. Laboratory staff must immediately inform the supervisor of any non-conformities that may affect the validity of sample results. The Laboratory shall either:

- Retain correspondence and/or records of conversations concerning the final disposition of rejected samples; or
- Fully document any decision to proceed with the analysis of samples not meeting acceptance criteria.

When samples do not meet laboratory requirements, they are either rejected or reported with a comment for the appropriate tests. In either case the program manager is contacted in writing (e-mail or letter) requesting concurrence with the Laboratory's decision to reject the samples or to proceed with the analysis and include an appropriate comment on the laboratory report. If samples are

accepted, analysis data will be “qualified” on the final report. Samples may be rejected for the following reasons:

- Insufficient volume.
- Inappropriate container.
- Sample beyond hold time or laboratory is unable to perform analysis within required hold time.
- Inappropriate sample preservation or sample chilling has not begun for samples requiring thermal preservation.

### 6.5 Sample Containers, Preservation and Holding Times

Preservation, types of containers, and holding time information can be found in Table 1060:I in 20th edition Standard Methods for Examination of Water and Wastewater

## 7.0 SAMPLE MANAGEMENT

Detailed instructions are available for individuals that are required to login samples. The laboratory technician will assign a unique laboratory control number to track samples through analysis and final report.

### 7.1 Chain of Custody Procedures

#### 7.1.1 Introduction

Before deciding to use chain of custody procedures to insure that laboratory results can be used for litigation and enforcement, the collection and analysis of samples must be part of a well organized plan. The plan will delineate what, where and how the samples are taken and establish the level of quality assurance needed. A plan calling for chain of custody procedures will require documentation of sample integrity from collection to final disposition by the laboratory.

Chain of Custody Procedures are necessary to insure the legal integrity of sample materials collected and submitted to the City of _____laboratory for analysis. The validity of the test results is assured if the Department can show that after the samples were collected, they were kept safe from tampering or chemical contamination. This requires that complete written documentation of the security of the sample from collection to disposition be kept.

#### 7.1.2 Sample Custody

A sample is under custody if

- It is in your possession, or
- It is in your view, after being in your possession, or
- It was in your possession and then you locked it up to prevent tampering, or
- It is in a designated secure area.

### 7.1.3 Submittal of Samples

Any user of the City of _____ laboratory can request chain of custody handling of their samples; these requests presume that the results are likely to be used for enforcement. Whether samples are hand carried or delivered by a courier they must be properly preserved, individually sealed and include a Chain of Custody Record. After receipt of the sample, a copy of this record is returned to the sampler. To track their samples from collection in the field to receipt of a laboratory report, the sampler will use the laboratory's chain of custody transfer record form and will seal the individual sample containers.

Hand Carried – This is the most common approach and is used almost exclusively by department personnel. Unless special arrangements are made, these samples should be submitted Monday through Friday between 8:00 a.m. and 4:30 p.m. and relinquished to the Laboratory Supervisor or his designee. Thermal preservation must be maintained by packing samples with a sufficient volume of ice (blue ice does not cool samples sufficiently). Unless special arrangements are made, overnight delivery is required and samples need to arrive Monday through Thursday before noon. Note: Do not use US Postal Service since delivery is to a central location and not the laboratory building. Samples sent by a package carrier, UPS or Federal Express, are to be addressed to:

City of _____ Laboratory
Attn: Chemistry Laboratory Supervisor
103 South Main Street
North Pole

### 7.1.4 Sample Custody Procedures

- The Laboratory Supervisor must be given advance notice of samples requiring Chain of Custody handling procedures.
- Field personnel must document in a field notebook all details regarding sampling activities. Documentation must include exact information regarding date, time, location, names of people present, unusual events, field measurements, details of sample storage and security, and transfer of samples to others.
- Field personnel are supplied with the proper sampling containers, chemicals for sample preservation, coolers, sample labels, Chain of Custody sealing tape, a Chain of Custody Record, and a listing of acceptable hold times, sampling procedures, and preservation techniques.
- Field personnel must collect samples according to standard procedures and add preservative if required. Samples requiring field preservation must be collected in containers containing the preservative. Field personnel needing to break the seal(s) at the laboratory to add preservation chemicals are asked to transfer custody to the laboratory after the samples are resealed. Lab personnel must be notified if preservation is to be done at the Lab by laboratory staff.

- Field personnel must seal the top of the sampling container with a Chain of Custody Sample Seal, initial the seal, complete the identifying label and store and transport samples in a sealed cooler with ice, if thermal preservation is required. A secure container capable of being sealed is acceptable if thermal preservation is not required.
- At no time are samples to be left unattended unless they have been locked or secured with initialed seals in place.
- The samples must be delivered to the Laboratory Supervisor or designated staff chemist who will accept the samples and perform the following steps.
    1. Verify that correct containers were used and required preservation was performed.
    2. If thermal preservation is required:
        verify that samples arrived on ice and cooling has begun
        record the temperature of a representative sample.
    3. Verify that all samples listed on the Chain of Custody Record form are accounted for.
    4. Verify that all containers are properly sealed and that all seals are intact and the Chain of Custody form and seals are completed correctly.
    5. Accept the samples and sign the Chain of Custody Record form.
    6. Log samples into the laboratory logbook and designate the samples as enforcement. Label the samples with unique label or verify that sample login was completed correctly and samples are properly labeled with unique sample identification numbers.
    7. Store samples in a designated locked refrigerator(s).
    8. Provide the designated individual with the Chain of Custody Record form, to file.
    9. Notify analysts or technical directors responsible for the analysis of the sample(s).

#### 7.1.5 The Chain of Custody Record

- The sampler(s) and/or witness are required to sign the form when samples are collected.
- A witness is not required to be present during sampling to satisfy the Chain of Custody requirements of sampling.
- Enter the name of the laboratory performing the analysis.
- Enter the exact sample location.
- Record the date and time of sample collection and whether the sample was a composite or grab.
- The description and number of containers should include the tests to be analyzed by groups on the slanted lines and the number of containers for each group in the accompanying box; e.g. volatiles, metals, semivolatiles on the slanted line and the number of containers/sample in the box.
- The total number of sample containers per location and any remarks regarding the sample should be recorded.

- When custody is transferred from one person to another, both parties must sign and date this form. If someone other than the person whose signature appears at the top of the form transports the samples to the laboratory, that transfer must be documented on this form.
- If the sample is to leave the laboratory for any reason the sample must be resealed and a Chain of Custody Record form will be reinitiated.

#### 7.1.6 Responsibility of the Analyst

An analyst assigned to perform the required analyses on Chain of Custody samples is expected to follow the procedures listed to insure that the Chain of Custody is maintained throughout the analytical process.

That analyst, using a Sample Sign-Out Sheet, signs a Chain of Custody sample from the refrigerator, removes the container, breaks the seal and removes an aliquot of sample, which is adequate to perform the analyses requested. A majority of containers are designed to provide enough sample for one analysis or a series of similar analyses.

This assumes that the sample will not be analyzed by another laboratory. If enough sample for a valid retest remains in the container after the analyst removes an appropriate aliquot of sample, the container is returned to the Chain of Custody refrigerator for possible re-analyses. An analyst, who is responsible for a subsequent analysis to be performed on an aliquot of this sample, must also document the removal and return of the sample on the sample sign out sheet. If all analyses from the container are complete any remaining samples are placed into long-term storage (Section 7.1.7).

Empty containers and containers of samples in which insufficient volume remain to complete another analysis are discarded. If the client requests that samples be removed from the Laboratory facility, the samples will be resealed and a new Chain of Custody Record will be initiated.

The specific steps to be documented on the Sample Sign-Out Sheet are:

- Laboratory ID #.
- Date and time samples are removed from the refrigerator.
- Amount of sample removed.
- Initials of analyst removing the samples.
- Tests to be performed.
- Can a valid analysis be performed on the remaining sample Y/N? If N, then the remaining sample can be discarded. If Y, the sample is returned to the locked refrigeration unit.
- Date and time samples are returned to the refrigerator.
- Initials of analyst returning the container to the refrigerator or discarding vessel if insufficient sample volume remains.

#### 7.1.7 Long Term Storage of Chain of Custody Samples and Records

When all tests on a sample from a particular container have been completed and if any remaining sample in that container can be used to obtain a valid analysis, that container must be stored as a Chain of Custody sample. Unless the laboratory has been specifically instructed to retain the samples, once hold times for the individual analyses in that container have been exceeded by 30 days, the samples can be removed from the refrigerator and discarded. All paperwork with the exception of field notes, are retained by the laboratory or Public Records. All laboratory records must be kept secure and in confidence to the client. Laboratory policy on record retention described in
Section 10.0 Data Reduction, Validation, Reporting, Tracking and Storage must be followed.

#### 7.1.8 Sample Containers, Preservation and Hold Times

Required containers, preservation and hold times for regulated contaminants are listed in Table 1060:I in 20th edition Standard Methods for Examination of Water and Wastewater

#### 7.1.9 Chain of Custody Record Form

See appendix A.

## 8.0 CALIBRATION PROCEDURES

All instruments and equipment used within the Laboratory are routinely calibrated by laboratory personnel. Analytical balances and thermometers are annually calibrated by an external calibration service. A summary of calibration procedures for individual instruments and tests is provided in this section.

Primary Calibration Standards used for calibration are purchased from a reputable dealer or prepared at the laboratory using reagent grade material. All purchased primary standards are certified by the vendor for purity and identity and when available are NIST traceable. Vendor supplied Certificates of Analysis are retained within analytical sections for a minimum of 5 years.

Calibration Standards (working standards) are dilutions or mixtures of stock standards used to calibrate an instrument. These standards are prepared or restandardized frequently (Section 9.3). NIST traceable reference materials are used when available. Certificates of analysis are retained in analytical centers for a minimum of 5 years.

The calibration range defines how results are reported and samples are processed. Results below the low calibration standard are reported as less than (<) the Reporting Limit (PQL). Results above the high calibration standard must be diluted and reanalyzed so that the instrument reading is within the calibration range.

## 8.1 Organics

### 8.1.1 GC (Volatiles, Pesticides, PCBs, TPH)

An initial calibration curve is prepared for each analyte of interest. Five or more calibration standards are prepared with one of the concentrations at the lower reporting limit (PQL)and the other concentrations corresponding to the expected range of concentrations in real samples. Each standard is injected into the instrument and the area response is tabulated against the concentration. The coefficient of determination is calculated for each curve by the software and is used to judge the curve fit. A coefficient > 0.99 is acceptable. The initial calibration curve must be verified every 12 hours (continuing calibration) by the injection of a mid-range standard. If the response for any analyte varies from the predicted response beyond the acceptance criteria, a new calibration curve must be prepared for that analyte. Acceptance criteria is listed by method in Table 8.1.

### 8.1.2 GC/MS (Volatiles)

An initial calibration curve is prepared for each analyte of interest. Five or more calibration standards are injected. A response factor is calculated as follows:

RF = As X CIS
CS X AIS
As = peak area of analyte or surrogate
AIS = peak area of internal standard
Cs = concentration of the analyte or surrogate
CIS = concentration of the internal standard

The System Performance Check Compounds (SPCC) are checked for the minimum average response factor. The Calibration Check Compounds (CCC) are checked for percent relative standard deviation. The SPCC and CCC checks must fall within the method guidelines. An average response factor is generated for each parameter. One primary ion is used for quantitation. A continuing calibration verification standard (CCV) is run every 12 hours, and the SPCC compounds are checked for response factors, and the CCV compounds are checked for percent relative difference.

The mass assignments of the GC/MS system are determined by calibration with perfluorotributylamine (PFTBA). The system is then hardware tuned to meet method criteria for mass spectra of a 50ng injection of BFB (4-bromofluorobenzene).

### 8.1.3 HPLC (carbonyl compounds)

An initial calibration curve is prepared for each analyte of interest. Five or more calibration standards are injected with one of the concentrations at the practical quantitation limit and the other concentrations corresponding to the expected range of concentration in real samples. Each standard is injected into the instrument and the area response is tabulated against the concentration. The coefficient of determination is calculated for each curve by the software and used

to judge the curve fit. A coefficient above 0.999 for at least 5 of the 6 compounds and >0.995 for one is acceptable. The initial calibration must be verified every 10 injections by running a mid-range standard.

## 8.2 Metals

### 8.2.1 Mercury Cold Vapor Analyzers

Instrument calibration for metals analysis is performed daily. A multi point curve is generated. The calibration curves must have correlation coefficients greater than or equal to 0.995. Calibration verification is monitored by analyzing a second source standard immediately following calibration (Initial Calibration Verification - ICV). A midrange standard is analyzed after every tenth sample, and at the end of the sample run to assure that calibration is maintained throughout the run (Continuing Calibration Verification -CCV). The calibration blank is also reanalyzed immediately following calibration, after every ten samples and at the end of the analytical run. Calibration blank results must be less than one-half the reporting limit (PQL), and the ICV result should be within ±10% of the true value for analysis to continue. The CCV result(s) must be within ±10% of the initial value. Failure of a CCV sample requires recalibration or reanalysis of all samples analyzed after the last passing CCV.

### 8.2.2 ICP-MS

The instrument is tuned with multi-element tune solutions to meet method criteria. A performance report that verifies Mass calibration and resolution is performed daily. The detectors are cross calibrated when necessary. After instrument calibration criteria are met an initial calibration curve is prepared daily for each metal to be analyzed. Three or more standards are analyzed with one of the concentration at the reporting limit and the other concentrations corresponding to the expected range of concentrations in samples to be analyzed. The coefficient of linearity must be >.995. The calibration curve is verified by analysis of mid-range second source standard mixes containing all metals to be quantitated.

Calibration is verified after every 10 samples and at the end of the run by analysis of a mid-range standard. Results must be within ± 10% of expected values.

## 8.3 Inorganic Chemistry

There are several automated and non-automated analyses performed in the inorganic chemistry section. Calibration and calibration verification protocol will vary from test to test. For most tests calibration is verified by the analysis of a working standard (ICV) at the beginning of the analytical run. The Initial Calibration Verification (ICV) should be within + 10% of the true value for analysis to continue. A mid-range Continuing Calibration Verification standard (CCV) is analyzed after every 10 samples. A different concentration CCV is run at the end of the analysis. Results should be within 10% of the true value. Reagent Blanks/Method Blanks must be less than one-half the Reporting Limit (PQL).

For most colorimetric analysis a standard curve consisting of 4 to 6 points and having a correlation coefficient of at least .995 is generated. A typical ion chromatography run will have a standard curve consisting of 4 or 5 points for each ion of interest. A combined ion stock standard is used. The correlation coefficient of the standard curve for each ion should be >.998. The coefficient is calculated by plotting the peak area against the standard concentration using a linear fit.

## 8.4 Support Equipment

### 8.4.1 Thermometers

Thermometers used in the Laboratory are calibrated against a NIST-traceable thermometer. The NIST thermometer is re-certified every 5 years at thermometer readings 4°, 44.5°, 95°, and 122°. Correction factors are taken into consideration when the thermometer is used to determine correction factors of Laboratory thermometers. Correction factors are noted on thermometers if needed. Correction factors, date calibrated, temperatures of both thermometers and thermometer serial numbers are documented in a laboratory notebook.

### 8.4.2 Refrigeration Units

Temperatures within refrigeration units are checked almost daily. Temperatures are recorded in a logbook and should be 0-6°C for refrigeration units and -17° C + 2° for freezers. Thermometers are submersed in an appropriate solution within each unit. If temperatures exceed these limits the unit is monitored and corrective action taken if temperatures remain outside limits.

### 8.4.3 Incubators/Water Baths/Ovens

Microbiology incubator temperatures are checked twice daily when in use. Temperatures must remain within method specified limits. Oven temperatures for tests requiring a specified temperature are checked daily when in use. All temperatures are recorded. The water bath used for the digestion of mercury samples is checked and temperature recorded at the beginning of analysis, and at the end of the digestion and must be 95° + 2° C.

### 8.4.4 Balances

Calibration of analytical balances is performed annually by a calibration service that is ISO 17025 compliant. Calibration is verified monthly with NIST traceable Class 1 weights. Two weights bracketing the expected range of measurements are used, measurements should be within ± .5mg. All weights are recorded in a lab notebook. The NIST traceable weights are verified by the State of New Mexico Metrology Dept. every 3-5 years. Weights are cleaned with 95% ETOH 24 hours before they are checked. Weights must be within the balance tolerance of ± .0002g or the weight tolerance, whichever is greater.

#### 8.4.5 Automated Pipettes and Dispensing Devices

Dispensing devices used as Class A volumetric devices have routine maintenance performed and are calibrated by an external calibration service annually**.** The manufacturers precision and accuracy specifications must be met. Multi-volume dispensing devices have each dispensing head calibrated at a minimum of two volume settings each. NELAC requires that all class A dispensing devices be checked on a quarterly basis. This is performed in-house.

#### 8.4.6 pH Meters

A three point calibration is performed daily and after every 3 hours of continued use. Standards bracket the pH of the samples analyzed. The percent slope of the calibration curve must be >95%. If criteria is not met the meter must be recalibrated using appropriate standards.

#### 8.4.7 Computer Software

Computer software is purchased either to support new instrumentation, to upgrade the performance of existing equipment or to manage the tracking of Laboratory data. Software needs to meet bid specifications which is demonstrated during installation/or training. The IDA files contain relevant data that documents performance.

## 9.0 ANALYTICAL AND OPERATIONAL PROCEDURES

### 9.1 Analytical Methods

All methods commonly used City of _____ Laboratory are approved by 40CFR 141_____. Parameters with corresponding method numbers and references are summarized in Table 5.1 of this manual. Current Laboratory Standard Operating Procedures (SOPs) are available upon request. Technical SOPs describe in detail, routine analytical tasks performed at the laboratory and typically include:

- Method title and referenced documents
- Reagents and preparation
- Method summary
- Definitions
- Health and safety warnings
- Personnel qualifications
- Apparatus and materials
- Interferences
- Calibration and standardization
- Sample preparation and preservation
- Quality control procedures
- Equations, calculations and data reduction procedures
- Data bench sheets and reporting forms
- Deviations from referenced methods
- Troubleshooting
- Deionized Water System Maintenance SOP

#### 9.1.1 Method Review

SOPs for current methods must be reviewed biannually. The most current revision of the referenced method must be reviewed at the time of the biannual SOP review to assure that all method requirements and control limits are being met. The review/revision process must be documented.

#### 9.1.2 Method Revision

If a significant variation to a referenced method is made the Laboratory must first demonstrate the alternative protocol results are comparable. The Laboratory SOP must clearly describe the variance and comparability data must be on file at the Laboratory. To demonstrate comparability the laboratory must, at a minimum, analyze four consecutive representative split sample(s) using the standard method and the alternative protocol. The alternative protocol results must be within 10% of the approved test procedure. Each sample site may be subject to this demonstration of comparability.

The laboratory retains a copy of all archived SOPs. The analysts bench copy of an SOP must be updated whenever a protocol is changed and is the most current version of an SOP. Significant changes, including any change affecting the calibration or quality control acceptance limits must be authorized (initialed and dated) by the Lab Supervisor. The bench copy of an SOP must be officially revised every two years at a minimum.

### 9.2 Laboratory Water

Laboratory water meets or exceeds ASTM Type II Reagent Grade Water requirements. The laboratory's water system is described in the Laboratory Deionized Water System Manual. The SOP also provides a description of the daily, weekly, monthly and yearly water system maintenance and monitoring schedules.

### 9.3 Reagent Preparation, Documentation and Storage

All standards and reagents are prepared from reagent grade materials, primary standards or are purchased from reputable vendors. When standards are purchased the date of receipt is documented on the certificate of analysis and certificates are filed for a minimum of five years. Standards and reagents are prepared using Class A volumetric glassware and calibrated dispensing devices and ASTM Type II reagent water. An electronic log or log books are used to record the receipt of all vendor supplied standards and reagents. The vendor, date received, lot number, expiration date and other pertinent information must be documented in the Standards/Reagent Log. Log books or sheets are utilized to document all information needed to maintain proper traceability of all standards and reagents prepared or purchased by the laboratory. Logs document the date of preparation or opening of purchased standards, expiration date, a list of standards/reagents or solutions used, lot numbers and the

preparer's name (initials). Additional information may also need to be recorded such as pH.

Once a solution is prepared it is labeled with the solution name or description, concentration or normality, preparation and expiration dates and initials of preparer. Documented information must be sufficient to allow traceability to the preparation record which should provide traceability of all ingredients. Expiration dates for standards and reagents are usually specified in methods or by the manufacturer and are adhered to unless degradation prior to this date is observed. Purchased materials are labeled with the date received and opened and the expiration date if more stringent than manufacturer's expiration date. Reagents are stored according to Method or manufacturer's instructions and discarded upon expiration. When expiration dates are not specified the following guidelines are used:

**Stock Standards** used for calibration can be used for 1 year if properly preserved and stored.

**Titrating Solutions** need to be either re-standardized or a new bottle of vendor certified standard opened each month.

**Calibration or Spiking Standards** are dilutions of stock standards used to calibrate an instrument. These standards are to be prepared daily unless specified otherwise in the method SOP. All other solutions are used for no more than a year. They are valid for that length of time only if evaporation is minimized and proper preservation and storage techniques are used. If a bottle is opened often or is much less than half full more frequent preparation may be required. If degradation becomes apparent the solution is discarded immediately and holding times are reduced.

### 9.4 Miscellaneous Procedures

In addition to method specific procedures several operational activities are monitored at the laboratory. Documentation of the monitoring can be found in the following locations:

- Reagent and preparation notebooks
- Instrument maintenance logs
- Instrument service logs
- Laboratory water system maintenance logs
- Balance/refrigeration/incubator monitoring log books

### 9.5 Traceability of Measurements

All measurements are required to be traceable to a national or international standard of measurement when a traceable standard is available. Equipment and measurement devices including balances, thermometers, and dispensing devices, associated with the accuracy of a measurement are calibrated according to protocols outlined in this QA Plan. Reference standards and materials used at the lab or by equipment calibration services are traceability to a national standard. Traceability requires that lab employees document and retain all pertinent information related to a measurement. Records pertaining to

calibration, calibration verification, and analysis must be detailed and traceable to the standards used. All results, information and calculations needed to generate a result must be documented. Record retention will vary depending on the record but must meet lab policy outlined in the QA Plan.

### 9.6 Data Recording and Editing

All written records in notebooks and on bench sheets need to be legible and recorded in permanent ink. Sharpies or other markers should not be used. Corrections must be made by drawing a single line through the incorrect entry. Corrections must be initialed and dated (month-day-year). Writing over an incorrect entry or using white-out, correction tape or erasers is not allowed. Pages may not be removed from bound notebooks. All records must be signed or initialed (electronic or written signatures are acceptable) and the reason should be clearly indicated such as “prepared by”, “reviewed by” or “validated by”.

## 10.0 DATA REDUCTION, VALIDATION, APPROVAL, REPORTING, TRACKING AND STORAGE

All analytical data generated by the City of _____ laboratory is recorded, reported, reviewed and archived according to Laboratory protocols described in this Section of the QA Plan and in Laboratory SOPs. Analytical areas have slightly different data reduction, validation and reporting protocols depending on the means by which the data is generated and specific method requirements.

### 10.1 Data Reduction

Data reduction is the process of transforming raw data into final results that are reported in standard units to Laboratory users. The Laboratory's goal is to minimize the steps needed to transform raw data into reportable results Laboratory SOP's include equations used to calculate results or a reference to the instrument manuals or methods that include the equations, the method of calculation and bench sheets used to record pertinent data.

#### 10.1.1 Manual Integration

Situations arise where the automated quantitation procedures in the GC/MS, GC, HPLC and IC software provide inappropriate quantitations. This normally occurs when there is compound co-elution, baseline noise, or matrix interferences. In these situations, the analyst must perform a manual quantitation. Manual quantitations are performed by integrating the area of the quantitation ion of the compound. This integration shall only include the area attributable to the specific target compound, or internal standard compound. The area integrated shall not include baseline background noise. The area integrated shall also not extend past the point where the sides of the peak intersect with the baseline noise. Manual integration is not to be used solely to meet Quality Control (QC) criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Manual integration must be documented. Where manual integration has been performed, most software will mark the integrated

area with the letter “M” on the quantitation report. Removal of data computer operational codes, such as the “M” flag is not allowed. A hard copy print-out of the quantitation report will be filed with the modified report if an electronic copy cannot be archived.

## 10.2 Data Validation and Approval

The analyst generating the analytical data has the primary responsibility for its correctness and completeness. It is his or her responsibility to verify that the instrument was calibrated and performing correctly. Analysts are responsible for analyzing the appropriate type and quantity of quality control samples with their daily work. Results must meet pre-established control limits. If control limits are not met the analyst is responsible for reanalyzing samples or documenting, justifying and flagging final analytical results or reported quality control data. If data is deemed unacceptable due to quality issues the data should not be reported. The Laboratory Supervisor must be notified and written notification must be provided to the client. The protocol for reporting data in which pre-established control limits are not met is described in Section 5.0 of this manual. Results can not be approved or released until the validation step is performed. It is the data reviewer’s responsibility to know the frequency and type of quality control samples required and acceptance limits for each method he/she is reviewing; including curve acceptance, continuing calibration and precision and accuracy criteria. If criteria are not met and data are not flagged the data reviewer must return the data to the analyst responsible for flagging results. If the data reviewer feels the data should not be reported due to quality issues it is his/her responsibility to notify the technical director of the analytical center or the Laboratory Supervisor. The Laboratory Supervisor or his designee reviews and approves all data for a given sample before it is released. This final review insures that all QC reporting and data qualifying requirements were met and results from different parameters for a given sample correlate. The dates of data entry, validation, and approval and the name of the employee responsible for each step are tracked. Method specific checks are being incorporated into SOPs as they are revised.

### 10.2.1 Organics

In the organic section, each data set has a data review check off list that must be completed by a second analyst. The following information is verified when applicable. Method specific checks and acceptance criteria will eventually be detailed in each laboratory SOP.

- Checks all worksheet header information for completion. Checks dates (extraction, analysis and calibration). Insures extraction and analysis dates and times are documented and entered.
- Checks initial calibration data against established criteria.
- All criteria for instrument tuning, internal standard areas, retention times, surrogate recoveries and analytical quality control results are checked.
- Checks all method quality control data to assure the correct type and amount of checks are performed and results are within control limits

- Compounds identified on the quantitation report must agree with results reported. All manual integrations must be properly documented.
- All calculations such as total volatile hydrocarbons, soil concentrations, percent recoveries and dilutions are checked.
- Verifies that data is correctly calculated and correct standard concentrations and dilutions have been entered
- All irregularities are properly documented and if necessary data flagged when pre-established control limits are not met.
- Verifies that sample dilution factors are accounted for in manual, instrument calculations.
- Reports only the parameters that were analyzed for on multi-parameter tests.

### 10.2.2 Inorganics/Metals

In the inorganic analytical section, the second analyst checks the following items when applicable. Method specific checks and acceptance criteria will eventually be detailed in each laboratory SOP.

- Analysis date and time (required when hold times are <72 hours) are documented on bench sheets.
- Insures all calibration and continued calibration criteria are met.
- Checks all method quality control data and documentation to insure the correct type and amount of checks are performed and results are within control limits.
- Checks all bench sheets for completion (i.e. Lot # of chemicals, QC identification, Initials, dates and times when required).
- Checks all manual calculations or data entry into calculation programs designed to calculate final results.
- Verifies that dilution factors have been properly accounted for and that standard and spike concentrations are correct.
- Checks to be sure any irregularity is documented and if necessary data flagged when pre-established control limits are not met.

### 10.2.3 Microbiology

Method specific checks and acceptance criteria will eventually be detailed in each laboratory SOP.

- Checks all Data Management System entries against bench sheets for transcription and reporting errors for manually entered information. All dilution calculations are checked. MPN values are rechecked against MPN Tables if the MPN Program has not been used.
- Insures that all data is flagged if the sampling time has not been provided.
- Insures that the date and time of analysis and the chemist initials are entered on bench sheets.
- Checks for completion of required bench sheet information.
- Insures that membrane filtration results are flagged with a “C” Colony Counts

- Outside Ideal Range, if counts are not within method recommended range.
- Insures that documentation of the notification of appropriate contacts has been made when acceptance limits are exceeded for clients that require immediate notification.

## 10.3 Data Reporting

### 10.3.1 Policy

Laboratory staff shall not release results (electronic, paper or verbal) to individuals outside the laboratory. All inquiries for information must be directed to the Laboratory Supervisor who will either obtain written permission (e-mail is acceptable) or forward the request to a Program or Project Manager. All records are held secure and confidential.

### 10.3.2 Final Report Format

Data is transmitted to Laboratory users in one of three ways: paper reports for each lab number, condensed reports when multiple results are reported on a page and electronic reports. Final reports for test data are issued only after internal review has been completed. Electronic transfer of data is an option available to laboratory users that have access to the laboratory network. Condensed and Electronic Reports do not contain all the information presented on paper reports. Clients receiving electronic reports are aware that information such as quality control data and sample and order comments are not presented. If an order has both Organic and Inorganic tests requested two separate reports for the same order ID will be generated. The reports have a different format. The cover page of lab reports have general comments and may have Order specific comments that have been added by the laboratory supervisor. When opinions and interpretations are included in a Report the laboratory documents the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly identified as such. If a Report has been revised this will be indicated on the cover page.

## 10.4 Data Tracking and Record Storage

### 10.4.1 General Information

The Laboratory has policies and procedures for the retention and disposal of all quality and technical records (see Summary Table 10.1 Record Storage and Retention Times). The record keeping system allows for the reconstruction of all activities required to produce an analytical result. All records are stored under appropriate conditions for the type of media (electronic or hard copy), and are readily retrievable to individuals that are allowed access. Backup and access policies for electronic files are in place. Records must be legible and held secure and in confidence for a minimum of 5 years. Records may be destroyed after the minimum required hold times have been exceeded. In the event that the Laboratory is closed, all electronic and paper records will be transferred to the State Public Records center.

### 10.4.2 Sample Handling and Receiving

Records are maintained for all procedures and policies pertaining to sample handling and receiving for a minimum of 5 years. Records of any deviations from policies are also retained either on bench sheets.

### 10.4.3 Technical Records

#### 10.4.3.1 Paper Records

Original raw data for calibrations, samples and quality control measures, worksheets, and instrument response records are stored for a minimum of 5 years. Vendor supplied standard certification paperwork is archived at the Laboratory for a minimum of 5 years. If records must be retained indefinitely they are transferred to the State Public Records facility and microfilmed. Once microfilmed the original records are destroyed. Analyst observations and calculations are documented at the time of analysis and retained with the raw data. All written records are documented in permanent ink. Errors in records must be corrected by drawing a single line through the error. The correct value is entered alongside the incorrect entry with the initials and date of the individual making the correction. When results are changed due to reasons other then transcription errors the reason for the correction must be obvious, if it is not, the analyst must document why the documented result has been modified. Laboratory notebooks and logs (paper) are tracked and archived for a minimum of 5 years and can not be destroyed without the Laboratory Supervisor's consent. The notebooks are stored within each analytical section. Information contained in notebooks includes sample processing steps and details such as: extraction and digestion records, instrument maintenance and routine checks, data reduction and transformation steps and standard and reagent receipt and preparations. Earlier revisions of Standard Operating Procedures (SOPs) and Quality Assurance Plans are archived (paper and electronic). The document control system used in this QA Plan (upper right hand corner of page) is also used for lab SOPs.

#### 10.4.3.2 Electronic Records

Electronic logs and bench sheets are stored as both paper and electronic copies in most instances. Electronic Logs that are not printed out and retained with the raw data are periodically archived on CD. A detailed data archiving protocol is under development. Records that are stored or generated by computers must be retained as a hard copy or have a write protected electronic copy.

## 11.0 QUALITY CONTROL SAMPLES AND ROUTINES USED TO ASSESS ACCURACY AND PRECISION

The purpose of this section is to define quality control procedures that are necessary to develop information which can be used to evaluate the quality of analytical data. Quality control (QC) terms are defined and an explanation of how, when and why QC samples are taken or analyzed is provided. This section is intended to be used as a guideline for laboratory users.

## 11.1 Field Quality Control Samples

The results of quality control samples taken in the field reflect the precision and accuracy of the entire process, from sample collection through analyses. Below is a brief description of quality control samples laboratory users should collect when appropriate. Samples may be logged in as “blind” samples if desired. Synonymous terms are provided in parenthesis.

### 11.1.1 Blanks

#### 11.1.1.1 Equipment Blanks

Equipment Blanks are a type of field blank used to determine if contamination has been introduced through contact with sampling equipment or to verify effectiveness of equipment cleaning procedures. Laboratory water free of analyte is transported to the site and processed through the sample collection device, preserved if necessary and returned to the lab for analysis. Laboratory water should not be stored for future use, a hold time of one week is recommended. Do not contaminate the carboys with field equipment. Do not use water from other sources or return water to the carboy. Equipment blanks should be processed whenever contamination is suspected, with each analytical batch or every 20 samples. Corrective action for contamination detected in equipment blanks is addressed by laboratory users evaluating data.

#### 11.1.1.2 Field Blanks

Field Blanks are used to determine if method analytes or other interferences are present in the field environment. This would include contamination from sample bottles, storage, transport and sample preparation. A field blank is usually laboratory deionized water that is transported to the sampling site, opened to the contaminated environment, and processed as a sample (filtration, preservation, etc.). One field blank should be submitted with each analytical batch or every 20 samples or whenever contamination is suspected. Contamination detected in field blanks would need to be evaluated by both field and laboratory personnel.

#### 11.1.1.3 Filter Blanks

Filter Blanks (Cartridge Blanks) are used to determine if method analytes or other interferences are introduced during the filtration or sampling process. Laboratory water is used to rinse the filter and filtration apparatus. Air filter blanks may also be submitted to determine if sample breakthrough has occurred. At least one filter blank should be processed with each sample batch or whenever contamination is suspected.

#### 11.1.1.4 Trip Blanks

Trip Blanks are routinely used when sampling for volatile organic compounds. Volatile organic compounds are most susceptible to this type of contamination. The laboratory supplies samplers with a VOA vial containing acidified analyte free water. The vial is transported to the sampling site and returned to the lab without being opened. Sample contamination from penetration of the Teflon cap by halogenated solvents during transport or at the site can be detected with a trip

blank. Trip blanks are logged into the data management system and are assigned a sample ID number.

### 11.1.2 Precision and Accuracy Checks

#### 11.1.2.1 Field Duplicates

Field Duplicates (duplicate samples, replicate samples) are two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Results give a measure of the precision associated with sample collection, preservation and storage as well as with laboratory procedures. Field duplicate data provides the best measurement of precision from sample collection through analyses. Field duplicates should be taken on 5% of the sample volume. Duplicates are logged in as individual samples and can arrive at the laboratory as “blind” duplicates if the laboratory user desires. A field duplicate should not be confused with a split sample (Section 11.1.2.3).

#### 11.1.2.2 Matrix Spikes

Matrix Spikes are the same as analytical matrix spikes (Section 11.2.4.3) except that spiking is done in the field. Spiking samples in the field is less reliable and more difficult than spiking in the laboratory and is not recommended.
Results from analytical matrix spikes are used to detect matrix interference and measure method accuracy. If a sample is spiked a percent recovery is provided on the Final Laboratory Report. Spiked sample recovery results are useful to laboratory users.

#### 11.1.2.3 Split Samples

Split samples are aliquots of samples taken from the same sample container after thoroughly mixing or compositing the sample. They are analyzed independently and are used to document intra- or interlaboratory precision. Split samples may also be used to request matrix spike analysis for tests requiring two separate samples.

#### 11.1.2.4 Blind Samples

Blind samples are sample(s) submitted to the lab for analysis, the composition or origin of the sample is known to the submitter but unknown to the analyst. Blind samples can be a duplicate sample, blank, proficiency sample, or an interlab comparison sample.

## 11.2 Analytical Quality Control Samples, Solutions and Routines

Results of analytical quality control samples are used to estimate the precision and accuracy of data from sample preparation through analysis. Some of the data are reported with the associated sample result(s). In addition to the quality control samples in which results are released with the associated data there are several types of samples (solutions) that may be analyzed or procedures performed to verify the precision and accuracy of the entire system. Results may be used to verify calibration, identify reporting limitations or to help identify and if

possible correct for instrument, method or sample interferences. Not all of these sample types will apply to every analysis; some are instrument and method specific. Data acceptance criteria are method specified and if no specifications are provided they are based on historical data or internally established.

### 11.2.1 Negative Control (Blank) – Method Performance

The level of analyte of interest detected in the Method, Continuing Calibration, or Initial Calibration Blank is evaluated in relation to the sample result being reported within the batch. The general laboratory policy is to qualify any reported analytical results analyzed in the batch. If the blank is > ½ PQL but <PQL and the results are > 2X Blank no comment is needed. Analyst/Supervisor discretion must be used when reporting results. If there are method or regulatory requirements for qualifying data associated with Blank results those requirements must be followed. If the minimum acceptance limit is exceeded one of the following corrective actions must be taken. They are listed from the most to least desirable course of action.

The source of the irregularity must be found and corrected and the blank and samples re-prepared and reanalyzed.

All associated sample results and the blank(s) must be flagged with a "BH" Remark Code and either an Order Comment or Sample Comment describing the Irregularity must be added.

#### 11.2.1.1 Initial Calibration Blanks – ICB

Initial Calibration Blanks are aqueous solutions prepared and diluted with the same volume of chemical reagents and solvents used in the preparation of the primary calibration standards. They may be used to give a null reading for the instrument response when running a calibration curve and to establish instrument background. The initial calibration blank does not assess for possible contamination during the preparation and processing steps. The ICB is analyzed as a sample at the beginning of the analytical run.

#### 11.2.1.2 Continuing Calibration Blank – CCB

Continuing Calibration Blank is the ICB solution that is reanalyzed throughout an analytical run to assess baseline drift.

#### 11.2.1.3 Method Blank

Method Blank (Laboratory Reagent Blank, Preparation Blank), is a volume of deionized laboratory reagent water carried through the entire analytical procedure including all preparation and processing steps. The Method Blank contains the same reagents as the samples. Analysis of a method blank verifies that interferences from contaminants in solvent, reagents, glassware and other sample processing devices are quantified. A method blank is analyzed at a minimum of 1 per preparation batch (20 samples) for methods that have a preparation procedure.

### 11.2.2 Positive Controls – Method Performance

#### 11.2.2.1 Laboratory Control Samples – LCS

Laboratory Control Samples – LCS (Blank Spike, Laboratory Fortified Blanks) are prepared by adding known quantities of the method analyte(s) to a volume of reagent water. Laboratory Control Samples must be processed at a minimum of 1 per preparation batch (20 samples). The LCS solution is the same solution used for matrix spikes. The LCS must be processed exactly like samples within the analytical batch. The concentration is typically mid-range but must be within the calibration range. LCS results are used to evaluate the total analytical process including all preparation and analysis steps. LCS results are also used to validate or reject matrix spike recovery results since the solution used to spike the LCS is the same solution used for sample matrix spikes.

The results of LCS are reported as a percent recovery. LCS control limits are those established in the referenced method. If there are no established criteria, the lab determines internal criteria based on historical data. If an LCS recovery is outside the control limit the LCS solution is reanalyzed. If the reanalysis of the solution is acceptable a note is made on the bench sheet and results are accepted. Any samples associated with an unacceptable LCS must be reprocessed and re-analyzed or the associated samples and LCS results are to be reported with a data qualifier. For multi-parameter methods, the components to be spiked shall be as specified by the referenced test method or other regulatory requirement.

#### 11.2.2.2 Quality Control Sample – QCS (Certified Reference Material CRM: Standard Reference Material SRM)

Quality Control Sample – QCS can be either an uncontaminated sample matrix, (i.e. fish, soil, ash) spiked with known amounts of analytes or a contaminated sample matrix. The QCS is a NIST certified standard purchased to establish intra-laboratory or analyst specific precision and bias or to assess the performance of the measurement system. QCS results are tracked.

### 11.2.3 Standards – Method Calibration

#### 11.2.3.1 Primary Calibration Standards

Primary Calibration Standards (Primary Standard, Calibration Standard) are prepared from dilutions of a NIST traceable stock standard solution or are prepared in-house from reagent grade materials. The standards are used to calibrate the instrument response with respect to analyte concentration.

#### 11.2.3.2 Initial Calibration Verification Standard – ICV

Initial Calibration Verification Standard – ICV (Second Source Standard, Quality Control Check Sample-QC or C, Initial Performance Check-IPC) is a certified reference standard from a source different then the primary calibration standard. When available they are processed the same as the primary calibration standard and are an independent check on the primary standard used to calibrate the instrument.

ICVs are analyzed immediately following calibration and determine if sample analysis can proceed. The concentration of the ICV is approximately the midlevel of the calibration range. If acceptance limits are not method specified they are established in-house. If the first analysis does not produce an acceptable result the sample may be reanalyzed once. If the second attempt does not generate an acceptable result the analysis of samples may not proceed. The source of the error needs to be determined and corrective actions taken. The Laboratory Supervisor must be consulted and associated data will likely be qualified. The client may be contacted prior to releasing data. The second source standard result generated at the beginning of the run is calculated in percent recovery and tracked.

#### 11.2.3.3 Continuing Calibration Verification Standard – CCV

Continuing Calibration Verification Standard – CCV (Calibration Check Standards, Same Source Standard, Calibration Check Compounds, Calibration Verification Check – CVC, or Continuing Calibration Check Standards – CCC) is a primary calibration standard(s) that is reanalyzed with test samples to verify continued calibration of the analytical system. Continued Calibration Standards are analyzed at the beginning and end of the analytical run and after every 10 samples for large analytical runs. The concentration of the CCV at the beginning and the end must be varied within the calibration range. The CCV is expressed as a percent recovery. Reported results need to be bracketed by acceptable CCVs. If limits can't be met affected samples can be reanalyzed once prior to taking corrective actions.

Under unusual circumstances results may be reported without a passing CCV. The Laboratory Supervisor should be consulted and associated data will likely be flagged. The client may be contacted prior to releasing the data.

#### 11.2.3.4 Internal Standards

Internal Standards are used for some organic methods and ICP/MS technology. The standards are added to every standard, blank, matrix spike, matrix spike duplicate and sample extract at a known concentration prior to analysis. Internal standards are used as the basis for the quantitation of the target compounds for several organic methods. For ICP/MS the internal standard solution is used to monitor the analysis for matrix effects and correct for instrument drift throughout the analysis.

### 11.2.4 Precision and Accuracy Checks – Sample Specific Controls

#### 11.2.4.1 Analytical Sample Duplicate

Analytical Sample Duplicate (Duplicate, Lab Duplicates) are two aliquots taken from the same sample container that are processed and analyzed separately. Results are used to measure analytical precision from sample preparation through analysis for a given matrix. A minimum of 5% of all samples are analyzed in duplicate. When a sample is analyzed in duplicate the first result

recorded appears in the final laboratory report result column. The second result and the relative percent difference (RPD) of the duplicate values are reported in the QC results section of the report. The RPD calculation can be found in Section 14.1 of this manual.

Historical data from the analysis of laboratory duplicates are used by the laboratory to establish precision control limits. If there are method specified limits they must be met. Sample results in which the sample duplicate Relative Percent Difference (RPD) is outside the laboratory control limit must be flagged. If a result is outside the established control limit (OOC) the analyst must:

Flag the QC result that is OOC.

Flag associated sample results with an appropriate Sample Remark Code and provide a Sample or Order Comment if further qualification is needed or warranted.

Notify the Laboratory Supervisor if the analyst is unsure whether results should be reported.

When control limits are exceeded by 5% or more the analyst must take further action to assure that a correctable error was not the cause of the irregularity. The analyst must re-prepare and reanalyze the sample in duplicate (i.e. if the control limit is 10%, analysis resulting in RPDs >15% require reanalysis of the original sample). The analyst is allowed to repeat analysis once. If after re-analysis the RPD falls within the established limit the new result(s) can be reported as long as there is clear documentation and traceability of the reported result. If the RPD is still outside limits and if all other QC within the run are acceptable the analyst can report the initial result(s) if properly qualified. If there is evidence that the analytical system is not in control analysis must stop and results must not be reported. In some situations reanalysis is impossible (insufficient sample volume) or impractical (hold time has been exceeded or there is a known documented interference that can not be corrected for). If the analyst suspects that a processing error occurred that impacts all of the samples analyzed then the entire analytical batch must be reprocessed and reanalyzed. When results for a method are consistently below the reporting limit, precision data is obtained from the analysis of matrix spike duplicates (11.2.4.4) when spiking solutions are available.

#### 11.2.4.2 Instrument Duplicates

Instrument Duplicates are two aliquots taken from the same extract or digestate and analyzed in duplicate. Results are used to measure instrument precision only. The average value of instrument duplicates may be reported, however method precision may not be calculated using instrument duplicates for methods requiring predigestion, extraction or any other sample preparation steps.

#### 11.2.4.3 Matrix Spikes – MS

Matrix Spikes – MS (Laboratory Fortified Sample Matrix) are prepared by adding a predetermined quantity of stock solution of the analyte(s) being measured to a sample prior to sample extraction/digestion and analysis. The stock solution must

be the same solution used to prepare the LCS. The concentration of the spike should be at the regulatory standard level or spiked at a level that will result in a final concentration that is approximately 1.5 times the unspiked concentration. The volume of the spiking solution must be less than 5% of the sample volume being spiked. A portion of the unspiked and the spiked sample are analyzed and a percent recovery is calculated (Section 14.2). Recovery data provides a measure of accuracy for the method used in a given matrix. Recovery results verify the presence or absence of matrix effects and are particularly important when analyzing complex matrices (soil, sludge, sediment or samples with interferences). Five percent of all samples received at the lab are spiked when sufficient sample volume is provided or at a rate specified by the test method or project plan. If a sample is spiked the calculated percent recovery is reported on Final Laboratory Report forms. Samples of some methods cannot be spiked (i.e., chlorophyll, dissolved oxygen, turbidity). Samples to be spiked are selected by the analyst unless they are pre-selected by laboratory users. Acceptance limits for matrix spikes analyzed at the lab will vary depending on the analysis, matrix and sample concentration level. Acceptance limits are either method specified or established from historical laboratory results. The narrower limits must be used. If recovery data is unacceptable, and the laboratory control sample (LCS) is within acceptance limits a matrix interference may be the cause of the irregularity. Sample results associated with a Matrix Spike Recocvery (MS) outside the laboratory control limit(s) must be flagged. If a result is outside the established control limit (OOC) and the LCS is acceptable the analyst must:

Flag the QC result that is OOC.

Flag associated sample results with an appropriate Sample Remark Code and provide a Sample or Order Comment if further qualification is needed or warranted.

Notify the Laboratory Supervisor if the analyst is unsure whether results should be reported.

When control limits are exceeded beyond 10%, the analyst must take further action to assure that a correctable error was not the cause of the irregularity. The analyst must re-prepare and reanalyze the sample and matrix spike (i.e. if limits are 80-120%, reprep and analysis is required if outside 70-130%). The analyst is allowed to repeat analysis once. If after re-analysis the result(s) fall within the established limits the new result(s) can be reported as long as there is clear documentation and traceability of the reported result. If the MS result is still outside limits and all other QC within the run are acceptable the analyst can report the initial result(s) if properly qualified. If there is evidence that the analytical system is not in control, analysis must stop and results must not be reported. In some situations reanalysis is impossible (insufficient sample volume) or impractical (hold time has been exceeded or there is a known documented interference that can not be corrected for). If an interference is suspect, the analyst may spike a series of dilutions to verify and eliminate or reduce the effect of the interference. In some instances analysts are able to eliminate the interference and report uncompromised data. However, if the required dilution is large and the original sample is no longer represented in the diluted sample or

the analyte of interest is diluted below the PQL results may not be reported or reported as <PQL unless properly qualified. The client is responsible for determining the usability of flagged Matrix Spike sample results.

#### 11.2.4.4 Matrix Spike Duplicate – MSD

Method precision can also be calculated from matrix spike duplicates. Matrix spike duplicates are used to estimate method precision for analytes that are frequently found below the practical quantitation limit. A second aliquot of the sample is treated like the original matrix spike sample. The relative percent difference (RPD) of the matrix spike and the matrix spike duplicate is calculated and is used to assess analytical precision. Final laboratory reports indicate when RPD values are calculated from matrix spike duplicates.

#### 11.2.4.5 Surrogates

Surrogates are organic compounds, which are not found in environmental samples, but have similar chemical structures, and extraction and/or chromatography properties. These compounds are spiked into all blanks, calibration and check standards, samples (including duplicate and laboratory control samples) prior to analysis by GC or GC/MS. Percent recoveries are calculated for each surrogate. Surrogate compounds and their acceptable recovery ranges are specified in analytical methods and are listed in Standard Operating Procedures (SOPs) for organic methods. The Laboratory tracks surrogate recovery results and when sufficient historical data is available internal control limits are established. Recovery data is reported with every sample result. When a recovery value is not within acceptance limits calculations and surrogate solutions are rechecked. Samples or extracts may be reanalyzed. If results are still not within suggested limits a flag “S-surrogate recovery outside acceptance limits” must be added next to the surrogate result that exceeds a criterion.

### 11.2.5 Limits

#### 11.2.5.1 Method Detection Limits (MDL)

Method Detection Limit is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. It is determined from repeated analysis of a low level sample in a given matrix containing the analyte at a predetermined level. MDLs are determined annually for most analytes and matrices. The process and formula used to generate MDLs are described in Section 14.4.

#### 11.2.5.2 Practical Quantitation Limit (PQL)

Practical Quantitation Limit (PQL) (Reporting Limit) is the lowest level that can be reliably achieved during routine laboratory operating conditions. The PQL is approximately two to ten times the calculated MDL. PQLs are a preferred reporting limit because MDL values will change each time they are calculated even though the analytical procedures, instruments and sample matrices are the same. By reporting the low level value as less than (<) the established PQL, laboratory users can be certain that any reported value is reliable and that

reporting limits will remain relatively constant. In the organics analytical center a N.D. (not detected) appears on final laboratory report forms rather than <PQL value. However, if a compound is detected at a level that is less than the PQL and the value is no less than one half the PQL; a <"PQL" (of the compound in question) is reported rather than a N.D. For organic results the Laboratory Reporting Limit (PQL) must increase if sample dilution is required. The increase in the PQL will be equivalent to the dilution factor. For multi parameter methods the analyst must make an effort to report results from the least dilute analysis for each parameter. If sample results are reported from two analyses, then the PQL is increased for the parameters reported from the diluted sample. If for some reason it is impractical to increase the PQL at final report time, another form of qualification must be implemented.

#### 11.2.5.3 Instrument Detection Limit (IDL) (ICP-MS only)

Estimated by calculating the average of the standard deviation of three runs on three non-consecutive days from the analysis of a reagent blank solution (which is equivalent to a calibration blank for waters) with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample. IDLs must be determined at least every three months.

#### 11.2.5.4 Preparation Batch

Preparation Batch is composed of one to twenty environmental samples of the same matrix that are prepared and analyzed together with the same process and personnel, using the same lots of reagents. The maximum time between the start of processing of the first and last sample in a preparation batch is 24 hours.

#### 11.2.5.5 Analytical Batch

Analytical batch is defined as a group of samples (extracts, digestates or environmental samples) that are analyzed together with the same method and personnel, using the same lots of reagents and having a defined set of quality control samples analyzed with the samples. Several preparation batches can be analyzed together in an analytical batch but each preparation batch must have associated QC data.

### 11.2.6 Instrument Checks

#### 11.2.6.1 Tuning Solutions

Tuning Solutions are used to verify that the resolution and mass calibration of the instrument are within required specifications prior to calibration and sample analysis (GC/MS, ICP/MS).

#### 11.2.6.2 Interference Check Solutions (ICS)

Interference Check Solutions (ICS) contain known concentrations of interfering elements. They are analyzed prior to samples to demonstrate that correction equations are adequate (ICP/MS).

## 12.0 AUDITS AND DEMONSTRATIONS OF CAPABILITY

System and performance audits are used to assess the overall effectiveness of the City of _____ Laboratory's quality assurance program. Demonstration of capability must be made prior to using a test method or if there is a change of equipment type, personnel or test method.

### 12.1 System Audits

A system audit is a qualitative evaluation of all components of a measurement system. System audits can be conducted by external auditing authorities (external audit) or can be conducted inhouse (internal audit).
Internal System Audits are a tool to: verify analyst compliance with the laboratories quality policies; to address any on going quality issues; and to highlight technical, equipment or management support needed within the analytical center being audited. It is the responsibility of the Laboratory's QA Officer to plan and organize internal audits within each of the laboratory's analytical sections. Internal audits of each analytical section are performed annually. Audits are conducted by qualified personnel that are independent of the activity being audited. Under certain circumstances a qualified chemist from another organization assists in the audit. Internal audits generally review all aspects of sample analyses from sample preparation to data reporting and review. In some instances the analyst is required to analyze a sample(s) of unknown concentration(s) while the auditor(s) observe. All notebooks and records are checked for traceability. SOPs are reviewed prior to the audit to assure written protocols are being followed. Checklists from external auditing organizations are often used. Previous audit reports are reviewed prior to an internal audit to assure that previously recommended corrective actions have been implemented. An Audit Report summarizing the method(s) reviewed and findings and recommendations is distributed to management and analysts audited. The analysts being audited have an opportunity to add to the Audit Report any comments or recommendations to management that would assist in improving the overall function of the section and the quality of data being generated. If during the course of an audit or at any other time a significant departure from the QA Plan policies, method SOP, or requirements are revealed, the findings will be documented and corrective actions will be required. The need to contact customers will depend on the severity of the departure and the effect the departure had on released data. The Laboratory Supervisor must notify clients in writing if audit findings cast doubt on Laboratory results. Follow-up audit activities shall verify and record the implementation and effectiveness of the correction action taken.

### 12.2 Performance Audits

Performance audits determine quantitatively the accuracy of analytical data. This is primarily accomplished by means of interlaboratory performance evaluations. Laboratory staff analyze reference materials and are rated on their performance. Each proficiency provider has a unique rating system and acceptance criteria

and vary in difficulty. Proficiency samples must be handled in the same manner as real environmental samples. This includes using the same staff, methods, procedures, frequency of analysis, reporting protocol, equipment and facility used for routine analysis. The Laboratory maintains records of the analysis of all PEs for at least 5 years. Proficiency audit results are reviewed by the Laboratory Quality Assurance Officer and distributed to the Laboratory Supervisor, laboratory staff and to laboratory users when requested.

Unacceptable results or trends from proficiency evaluations may necessitate an internal audit of the method in question. The audit is initiated by the Laboratory Quality Assurance Officer who may submit blind check samples for analysis.

**12. 2.1 Water Pollution Study (WP Series)** - semi-annual evaluation. Results are submitted for both methods of analysis when the laboratory reports results by more than one method.

- Trace Metals (aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, iron, lead, mercury, manganese, molybdenum, nickel, selenium, vanadium, zinc, silver, strontium, thallium, uranium)
- Minerals (spec. cond., total dissolved solids, total hardness, calcium, magnesium, total alkalinity, chloride, sulfate, sodium, potassium)
- Nutrients (ammonia as nitrogen, nitrate as nitrogen, total Kjeldahlnitrogen, total phosphorus)
- Demands (COD, 5-day BOD)
- PCB's in water
- Pesticides (chlordane, aldrin, dieldrin, DDD, DDE, DDT, heptachlor, heptachlor-epoxide)
- Volatile Halocarbons
- Volatile Aromatics
- Miscellaneous Parameters (non-filterable residue, total suspendable solids, volatile solids, total residual chlorine, pH, turbidity, nitrite, silica)

## 12.3 Demonstration of Capability

### 12.3.1 Initial Demonstration of Capability (DOC)

#### 12.3.1.1 New Method or Technology DOC

The demonstration of capability must be made prior to using any test method or any time there is a change of instrument type or test method. If there are method specified criteria they must be followed. When the method does not specify a procedure, the following steps should be documented when applicable:

- The lab supervisor of each analytical section shall participate in vendor provided training courses when new technology is employed.
- Demonstration of linearity.
- Method Detection Limit study.
- Accuracy - typically demonstrated by analysis of an internal blind NIST traceable standard at one or more concentrations.
- Precision - repeated analysis of a known sample four times.

#### 12.3.1.2 Precision and Accuracy Assessment

- A quality control sample shall be obtained from an outside source. If not available, the QC sample may be prepared by the Laboratory using stock standards that are 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, or if unspecified, to a concentration approximately 10 times the method-stated or Laboratory calculated method detection limit.
- At least four aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.
- Using all of the results, calculate the mean recovery (X) in the appropriate reporting units (such as µg/l) and the standard deviations of the population sample (n-1) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the Laboratory must assess performance against established anddocumented criteria.
- Compare the information from above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in Laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.
- When one or more of the tested parameters fail the acceptance criteria, the analyst must locate and correct the source of the problem and repeat the test for all parameters of interest. Repeated failure, confirms a general problem with the measurement system that must be corrected. For some methods NIST traceable standards are not available.

#### 12.3.1.3 New Analyst Demonstration of Capability (DOC)

A demonstration of ability is required prior to a new analyst reporting results for an established method that has an initial method/instrument DOC on file but has not previously been performed by him/her. The analyst must review all referenced methods, pertinent instrument manuals, and current method SOP. The analyst must observe the current analyst through all aspects of the procedure (sample preparation through reporting). The new analyst must also be observed processing QC samples to assess accuracy and precision by the primary analyst. Requirements vary depending on the complexity of the equipment or procedure to be performed. At a minimum the analyst must be able to accurately analyze a blind Proficiency Sample and meet established internal quality control limits for accuracy and precision.

#### 12.3.1.4 Analyst files are to contain training documentation.

### 12.3.2 Continued Demonstration of Proficiency

Analyst must demonstrate continued proficiency at least once a year for tests they are reporting results for. Analyst training files must contain a copy of a proficiency sample result that is rated acceptable.

## 13.0 PREVENTATIVE MAINTENANCE

Preventative maintenance is scheduled for most analytical equipment within the City of _____ Laboratory to minimize poor performance, instrument down time and subsequent "interruption" of analysis. Major analytical equipment is maintained under service contract, other instruments are maintained by a qualified analytical instrument repair service. Preventative maintenance schedules are listed in Table 13.1. Routine maintenance is performed on all analytical equipment by qualified Laboratory personnel. When it is practical, an inventory of critical replacement parts and spare parts needed for routine maintenance is maintained for each instrument. Logbooks are kept for each major instrument to document instrument problems, repairs and routine maintenance.

**Table 13.1 Laboratory Instrument Maintenance Schedules**
**INORGANIC**

| Instrument | Manufacturer | Model | Contractor | Preventative Maintenance Schedule |
|---|---|---|---|---|
| pH/Millivolt Meter | Orion | 720A | QC Services | 1/year |
| Non-Ratio Turbidity meter | HF Scientific | Micro 100 | QC Services | 1/year |
| Spectrophotometer | Genysis | Thermo Spectronic 10 | QC Services | 1/year |
| Dissolved Oxygen Meter | YSI | 5100 | QC Services | 1/year |
| Fluorometer | Turner | TD-700 | QC Services | As Needed |
| COD Reactor | Hach | 45600 (2) | QC Services | 1/year |
| Centrifuge | International Equipment | EXD | QC Services | As Needed |
| TCLP Extraction Apparatus | Millipore | — | — | — |
| Conductance Meter | YSI | 3200 | QC Services | 1/year |
| Pensky Martin Flashpoint Apparatus | — | — | — | — |
| Oven | Precision | – (2) | QC Services | 1/year |
| Chlorine Pocket Colorimeter | Hach | | — | — |
| Micro Distillation System | Lachat | — | Lachat | — |
| Auto Analyzer Systems | Lachat | QC 8000 | Lachat | As Needed |
| Auto Analyzer System | Lachat | QuickChem FIA 8000 Series | Lachat | As Needed |
| Ion Chromatograph | Dionex | DX 320 | Dionex | 1/year |
| Sonification Bath | Ultra Clean Equipment | — | — | — |

## METALS

| Instrument | Manufacturer | Model | Contractor | Preventative Maintenance Schedule |
|---|---|---|---|---|
| ICP/MS | Thermo-Elemental | X Series | Thermo-Elemental | 1/year |
| Automated Mercury Analyzer | Perkin-Elmer | FIMS100 | Perkin-Elmer | As Needed |
| Microwave Digestion Furnace | CEM | MDS 2100 | CEM | As Needed |
| Hot Block Digestors | Environmental Express | (2) | — | — |
| Water Bath | Precision | — | — | — |

## MICROBIOLOGY

| Instrument | Manufacturer | Model | Contractor | Preventative Maintenance Schedule |
|---|---|---|---|---|
| Autoclave | Castle | 122LS | Castle | 4/year |
| Air Incubator | Boekel | 133000 | — | — |
| Waterbath | Precision | 260 | — | — |
| Air Incubators | Fisher Scientific | 650F(2) | — | — |
| Quanti Tray Sealer | IDEXX | 2X | — | — |

## ORGANICS

| Instrument | Manufacturer | Model | Contractor | Preventative Maintenance Schedule |
|---|---|---|---|---|
| GC/MS system | HP | 6890/5973 (2) | Hardware and Software Systems (HSS)[a] | 1/year |
| GC system | HP | 5890 (TPH only) (FID Detector) | HSS | As Needed |
| GC System (Volatiles) | HP | 5890 (PID/FID Detectors) | HSS | As Needed |
| TurboVap Evaporator | Zymark | 500 (1) | Caliper | |
| HPLC | Waters | 2487 | Waters | 1/year |
| Cryogenic Concentrator | Entech | 7100 | Entech | As Needed |
| Canister Cleaner | Entech | 3100 A | Entech | As Needed |
| Gas Mixing System | Entech | 4600A | Entech | As Needed |
| Pure Air Generator | Aadco | 737 | — | — |
| Autosampler | Entech | 7016CA | Entech | As Needed |
| GC System (Pesticides/PCBs) | HP6890 Dual ECD Detectors | — | Hardware and Software Services (HSS) | As Needed |
| Purge and Trap with Archon Autosampler | Tekmar | 3100 | Varian | As Needed |
| Purge and Trap with Autosampler (Aquatec 70) | Tekmar | 3000 | — | As Needed |

## ANALYTICAL BALANCES

| Instrument | Manufacturer | Model | Contractor | Preventative Maintenance Schedule |
|---|---|---|---|---|
| Balance | Mettler | AE200 | QC Services | 1/year |
| Balance | Mettler | AT400 | QC Services | 1/year |
| Balance | Mettler | PM400 | QC Services | 1/year |
| Balance | OHAUS B | 1500D | QC Services | 1/year |

**MISCELLANEOUS**

| Instrument | Manufacturer | Model | Contractor | Preventative Maintenance Schedule |
|---|---|---|---|---|
| Exhaust Hoods (9) | Peck | — | — | 1/year |
| Microzone Hoods (2) | ENV. Service | — | — | 1/year |
| Refrigeration Units (11) | — | — | — | — |
| Glassware Washer | Steris | — | — | 4/year |
| Water System | U.S. Filter | MilliRo 120 D. I. | Ionpure | 2/year |
| Electronic Pipettes (12) | Rainin | — | — | 1/year |
| Manual Pipettes (7) | Rainin/Eppendorf | — | — | 1/year |

[a] QC Services is ISO 9002 registered, ISO 17025 compliant.

## 14.0 PROCEDURES USED TO CALCULATE AND ASSESS DATA QUALITY

This section describes the data quality indicators that are tracked. Equations for precision, accuracy, completeness and method detection limits are provided. Method specific calculations can be found in Laboratory SOPs.

### 14.1 Precision

Precision is a measure of how well replicate measurements reproduce and can be calculated from laboratory duplicates, instrument duplicates, duplicate analysis of a Laboratory Control Sample (LCS) , method blank duplicates (MBD) or matrix spike duplicates (MSD). Relative percent difference (RPD) is the current measure of precision for most analytes and is calculated as follows:

$$RPD = \frac{(C1 - C2)}{m} \times 100\%$$

where: RPD = relative percent difference
C1 = larger of the two observed values
C2 = smaller of the two observed values
m = mean of two observed values

If calculated from three or more replicates, relative standard deviation (RSD) is calculated rather than RPD:

$$RSD = (s/m) \times 100\%$$

where: RSD = relative standard deviation
s = standard deviation
m = mean of replicate analyses

### 14.2 Accuracy

Accuracy is a measure of how near a result is to the true value and is expressed as a percent bias or percent recovery. Method accuracy is determined from the

analysis of a laboratory control sample, continuing calibration check, quality control check samples or matrix spikes. Method accuracy and matrix effects are assessed by evaluating matrix spike results. The amount of analyte recovered after a sample has been spiked and processed reflects matrix effects upon the accuracy of the method. Percent recovery is calculated from matrix spike results using the following equation:

$$\% R = \frac{100 \times S - U}{Csa}$$

where: %R = percent recovery
S = measured concentration in spiked aliquot
U = measured concentration in unspiked aliquot
Csa = actual concentration of spike added
The above calculation does not take spike volume into consideration. Lab protocol requires that a <5% volume change occurs when a spike is added negating the need to volume correct. Percent bias is another measure of accuracy and is calculated using the following equation:

$$\% B = \frac{100 \times (O-T)}{T}$$

where: %B = percent bias
O = measured concentration of reference material
T = actual concentration of reference material

### 14.3 Completeness

Completeness is defined as the number of measurements judged valid compared to the number of measurements needed to achieve a specified level of confidence in decision making. The number of measurements judged valid must be determined by laboratory users familiar with the collection site, laboratory detection limits, and anticipated sample concentrations. Measurements judged invalid or suspicious by laboratory staff will be
flagged on final laboratory report forms and should be considered in completeness calculation. Laboratory data flags of importance to laboratory users are < “less than” flags and those summarized in Section 5.0 of this manual. Completeness is calculated as follows:

$$\% C = \frac{100 \times V}{n}$$

where: %C = percent completeness
V = number of measurements judged valid
n = total number of measurements necessary to achieve a specified level of confidence in decision making

## 14.4 Detection Limits

### 14.4.1 Method Detection Limits

Method Detection Limits (MDLs) are defined in the Federal Register as “the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte”. The Method Detection Limit is a statistical determination of precision only and is not used as a reporting limit. Method Detection Limit studies are part of a methods initial demonstration of ability if there is a spiking solution available. If a Method requires that MDL studies be performed the study must be repeated at the method required frequency for each sample matrix. This would likely mean that studies would need to be repeated on an annual basis, each time there is a change in the method that affects how the test is performed, or if there is a change in instrumentation. If a frequency of greater than 1/year is suggested rather than required the Laboratory will perform the study 1/year. MDLs must meet method-required limits if specified or must be below the laboratory Practical Quantitation Limit (PQL). If a calculated MDL for a parameter exceeds the PQL then the PQL must be raised until results from a new study justify lowering the PQL. If an MDL study is not required the laboratory may not report to a level lower than the low standard. Method Detection Limits are determined according to the Federal Register Appendix B Part 136, Revison 1.11. A minimum of seven replicates of low level spiked reagent blanks or solid samples are processed and analyzed as described in the reference listed. The standard deviation of the responses is used to calculate the MDL as follows:

MDL = S(t•99) for n replicates

Where: n = number of replicates analyzed
S = standard deviation of the values
t•99 = student’s t value for a one-tailed test at the 99% confidence level for “n” replicates.

A replicate result may not be excluded from the MDL calculation unless it is statistically determined to be an outlier (Dixon’s Test for Outliers; α .05, two sided test). Only the results for the parameters tested to be outliers can be dropped in multi parameter tests. A calculated recovery of 70-130% should be achievable if the MDL is to be used to calculate the PQL. If this level of accuracy is not achieved the concentration of the spike for the study is likely not appropriate and the study should be repeated if practical. This level of accuracy may be difficult to achieve for all parameters in multi-parameter methods. All method detection limit study results must be provided to the QA Officer upon completion.
The following information must be included:

- Date of sample analysis and preparation
- Analyst(s) – sample preparation and analysis
- Parameter/matrix
- Method

- Instrument ID
- Spiking level and level of low standard
- Individual results for all of the replicates (including outliers)
- Recalculated results if outliers were excluded (justification must be failure of Dixon Test for outliers)
- Calculated mean, standard deviation and MDL (precision)
- Accuracy (mean of replicate results)

### 14.4.2 Practical Quantitation Limit

The Practical Quantitiation Limit (PQL) is the laboratory reporting level It is a concentration at which both the accuracy and the precision of a method have been taken into consideration. The PQL is generally 2-10 times the calculated MDL. The PQL may be established from variables other than the calculated MDL since the MDL is only an estimation of method precision. One variable that may be taken into consideration is the concentration of the analyte found in the laboratory reagent blank. If background interference cannot be removed the PQL will be greater than 2-10 times the calculated MDL in order to reflect method inaccuracy at low levels. The laboratory reagent blank processed with the analytical batch should be no greater than 1/2 the concentration of the PQL. Another variable that may effect the Reporting Limit is sample dilution. If a sample dilution is required to remove an interference from an inorganic sample and the diluted result is below the PQL a "<PQL" result may not be reported. In this situation an "N" –sample not processed or processed but result not reported" Sample Remark Code is used and an order or sample comment is added to explain the unusual situation. For organic method the laboratory PQL increases by the sample dilution factor. There are alternative approaches to establishing and validating the PQL that may be more practical and appropriate for a given method. If an alternative approach is utilized the protocol must be approved by the Lab Supervisor and Quality Assurance Officer and the protocol and acceptance criteria clearly described in the Method SOP. The PQL is to be verified annually for each matrix, method and analyte according to a defined, documented procedure. Verification is not required for methods or analytes in which a spiking solution is not available. The validity of the PQL must be confirmed by the successful analysis of a QC sample containing the analyte of concern in each quality system matrix 1-2 times the claimed PQL. A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria or client data quality objective for accuracy. Unless there are method or client specified acceptance criteria for accuracy the laboratory's criteria for the low level PQL verification is + 20% for Inorganic methods and + 30% for Organic methods. The concentration of the low level standard used to calibrate an instrument must be at or below the PQL.

### 14.4.3 Instrument Detection Limits

Instrument Detection Limits (IDLs) are an estimate of instrument precision. A reagent blank solution is analyzed on three non-consecutive days with seven consecutive measurements per day. Each measurement must be performed as

though it were a separate analytical sample. IDLs are estimated by calculating the average of the standard deviations of three runs. The IDL only defines the instrumental limitations of a method and does not take the precision of processing and analyzing real samples into consideration. IDL studies are required only when they are method specified. A method requirement for performing an IDL study does not eliminate the requirement of annually verifying the Limit of Quantitation (LOQ).

#### 14.4.4 Limit of Quantitiation

The Limit of Quantitiation (LOQ) is a term synonymous to the laboratory's Practical Quantitation Limit (PQL) (see Section 14.4.2). The most commonly used procedure at the Lab for determining and verifying the LOQ is the performance of an MDL study followed by the calculation of a PQL (see Sections14.4.1 and 14.4.2).

### 14.5 Tracking of Quality Control Data

The five QC types: spikes, duplicates, standards, blanks and surrogate data. Each QC Type may have sub-categories. The following terms are defined in Section 11.2 of this Plan.

- Spike: Matrix spikes (MS)
- Laboratory Control Samples (LCS)
- Duplicates: Sample Duplicates (Duplicate)
- Laboratory Control Sample Duplicates (LCSD)
- Method Blank Duplicates (MBD)
- Matrix Spike Duplicates (MSD)
- Standard: Initial Calibration Verification Low Level (ICV Low)
- Initial Calibration Verification Mid Level (ICV Mid)
- Initial Calibration Verification High Level (ICV High)
- Continuing Calibration Verification Low Level (CCV Low)
- Continuing Calibration Verification Mid Level (CCV Mid)
- Continuing Calibration Verification High Level (CCV High)
- Quality Control Standard (QCS)
- Surrogates:
    - Blanks: Method Blank (MB)
    - Continuing Calibration Blank (CCB)
    - Initial Calibration Blank (ICB)

### 14.6 Quality Control Acceptance Criteria

Quality control acceptance criteria were established by reviewing historical data for each method/matrix. The limits meet method specified criteria but are generally narrower. The established limits are annually reviewed and adjusted if necessary. The validity of established limits can be verified by reviewing data. The mean ± 3 standard deviations is used. When acceptance limits are validated the data set may be tested for outliers. Statistical outliers are removed prior to calculating acceptance criteria. If a data set is from a matrix specific measure of precision or accuracy e.g. matrix spike recovery data, and the data set

predominately represents an unusually “clean” or “dirty” matrix from a specific project the Laboratory will look at historical performance and either widen or narrow a calculated acceptance limit to avoid creating an unrealistic window of acceptability. If a method specifies a required acceptance limit the limit must be met unless data is qualified. Laboratory acceptance criteria currently being used are summarized by parameter and matrix in Section 5.0 Quality Assurance Objectives.

### 14.7 Reporting of Quality Control Data

Laboratory analysts assign the appropriate quality control types at the required frequency to a QC Batch. Results are reported by QC Batch at data entry. Relative Percent Difference and Percent Recovery are automatically calculated from the information entered. Laboratory Reports have a “QC Information” summary at the end of each report. The QC information section of the report summarizes all QC data for Matrix Spikes-MS (percent recovery), Analytical Duplicates – Dup (RPD) and matrix spike duplicates –MSD (RPD) for the entire Order by parameter and Sample Number. When Control Limits are exceeded a flag is added to the QC data by the analyst. A sample result qualifier or comment must be added if the failed QC result indicates that a sample result(s) may be compromised.

## 15.0 Corrective and Preventative Actions and Customer Complaints

### 15.1 Corrective Actions

Corrective actions may be initiated as a result of a problem identified through a system or, performance audit, data review or data end user’s request. The process is generally initiated by the Quality Assurance Officer or Laboratory Supervisor and documented on a Quality Assurance Irregularity Report Form (Figure 15.1) by the analyst or Technical Director responsible for the data. The lead analyst has the ultimate responsibility of evaluating the effectiveness of the corrective actions. If a corrective action is ineffective it is the analysts’ responsibility to notify the Laboratory Supervisor. Laboratory management must verify that corrective actions have been effective – by performing a follow-up data review, submitting a proficiency sample or performing other internal audit activities. The steps taken in the corrective action process are:

- identify and define the problem
- assign responsibility for investigating the problem
- determine the cause of the problem
- determine the actions needed to eliminate the problem
- implement corrective action
- establish effectiveness of the corrective action
- management verifies effectiveness of corrective action

Corrective action may also be initiated by an analyst during or after analysis of samples. Laboratory personnel are aware that corrective actions may be necessary if:

- Unacceptable instrument conditions or calibration or continuing calibration data is generated.
- QC data are outside the warning or control limits for precision and accuracy.
- Peak shapes and or baselines are unacceptable.
- Blank(s) contain target analytes above acceptable levels.
- A surrogate recovery falls outside the expected range.

Investigation of problems revealed by the routine analysis of laboratory QC samples are the responsibility of the analyst generating the data or the Technical Director of the analytical center reviewing the data. Quality control sample results and instrument conditions are checked against established limits and deviations are immediately addressed. Predetermined limits for data acceptability beyond which corrective action may be required can be found in Sections 5 and 8. Additional method specific limits or conditions are described in the Standard Operating Procedures for each of the analytical methods used in the laboratory. If an analyst determines that corrective actions have not resolved an irregularity and a data set is compromised it is the analysts responsibility to notify the Technical Director or Laboratory Supervisor immediately. The Technical Director or Laboratory Supervisor must assess the data and determine if the data is to be released and how it will be qualified. This process is likely to require contact with the client(s) and written instructions on how to proceed. The clients instructions must be retained on file.

### 15.2 Non-conforming Work

If at any time it is determined that any aspect of the analytical process has compromised the Laboratory's ability to generate defensible data the analyst must notify the laboratory supervisor immediately. The laboratory supervisor must notify clients in writing (e-mail is acceptable) of the irregularity. This policy applies to situations in which the laboratory supervisor has determined that the significance of the irregularity justifies recalling work that has already been released or when the laboratory has decided that it will not report results that are considered invalid. This policy does not apply to those situations in which a data flag or sample note can be used to qualify the data. Corrective actions described in Section 15.1 must be taken immediately to remedy the situation.

### 15.3 Preventative Actions

All laboratory staff are encouraged to identify opportunities for improvement and notify management if resources are needed. A Preventative Action Form (Figure 15.2) is available to all laboratory staff and is used to document needed improvements and potential sources of non-conformance either technical or pertaining to the quality system in general. Forms are submitted to the laboratory supervisor. Preventative actions and follow-up are documented to assure that the preventative action was implemented and successful.

### 15.4 Customer Complaints

Customer complaint regarding the quality of data or service provided by the Laboratory shall be placed in writing and addressed to the laboratory supervisor (e-mails or letters are acceptable). The laboratory supervisor is responsible for evaluating the nature of the complaint. Once individuals or systems are identified as being deficient a corrective action will be put into place. The laboratory supervisor is responsible for verifying that a corrective action has been implemented and is effective in resolving the customer's complaint. The laboratory supervisor must respond to the written complaint in writing in a timely fashion. Complaints and Laboratory responses are kept on file. If a customer is not satisfied with the Laboratory's response the customer has the option of bringing the complaint to the Laboratory Director's attention.

**Figure 15.1 - Quality Assurance Irregularity Report**

A quality assurance irregularity report should contain the following information.

Date:

Due Date:

Date Returned:

Sample ID Number(s) Involved:

Reason for Initiation:

Description of QA Irregularity:

Name of Employee who Performed Work:

Steps taken to investigate irregularity:

Explanation of probable cause of irregularity:

Steps taken to prevent future occurrence:

Reviewers Comments:

Date(s):

Analyst:

Reviewer:

Laboratory Supervisor:

### 15.5 Preventive Action Plan

A preventive action plan contains the following information:

Date Submitted:

Response Date (2 weeks after submittal date):

Name of Employee Requesting a Preventative Action:

Needed Improvement and Potential Sources of Non-conformance:

Recommended Preventative Action:

Supervisor's Response:

Laboratory Supervisor:

Date:

Employee's Review Response:

Date:

RETURN FORM TO LABORATORY SUPERVISOR

## 16.0 QUALITY SYSTEM REVIEW

### 16.1 Quality Assurance Reports to Management

The Laboratory Supervisor will provide the following QA information:

- Precision/Accuracy Report for each analyte/matrix (annual).
- Laboratory QA Plan Updates (annual or as needed).
- Performance Audit results will be distributed to Laboratory Director as they become available. Irregularity reports issued as a result of performance audit ratings will be provided to the Laboratory Supervisor and maintained in a central location.
- QA office goals and objectives for the upcoming year (annual performance evaluation/work plan).
- Internal system audit reports from each analytical center (annual).
- Standard Operating Procedure (SOP) updates.
- Completed irregularity reports and correspondence related to corrective actions or investigations.
- Will inform Laboratory director when QA information required/requested of analyst is not provided. This may include MDL data, irregularity reports or SOP revisions. The Laboratory Director will also be notified if it is determined analysts are not complying with the QA policies described in this manual, or method SOPs.

### 16.2 Laboratory Director's Review of Quality System

An annual review of the laboratory's quality system must be documented. This review must consider:

- Suitability of policies and procedures.
- Technical director's issues and concerns that have been identified in Internal Audit
- Reports, Preventative Action Plans or Irregularity Reports.
- The outcome of recent internal audits.
- Corrective and preventative actions.
- Assessments by external bodies.
- Results of proficiency tests.
- Changes in the volume or type of work.
- Client feedback/customer complaints.
- Other relevant factors, such as quality control activities, resources and staff training.