

GPL Laboratories, LLLP
Quality Assurance Program Plan

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GPL Laboratories, LLLP

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1.0 Introduction

GPL Laboratories, LLLP is committed to providing the highest quality laboratory data available. All laboratory analyses are performed in full compliance within applicable State, Federal, or CLP Quality Control guidelines. The Quality Assurance (QA) and Quality Control (QC) program is defined in the Laboratory Quality Assurance Program Plan (QAPP) and the Laboratory Standard Operating Procedure (SOP) Manual. The QA program plan meets or exceeds EPA recommended guidelines with quality control samples accounting for at least 20% of the total number of samples analyzed. The Quality Assurance Manager ensures that facilities, equipment, personnel methods, records and Quality Control procedures are in conformance with GPL Standard Operating Procedures (SOPs) as well as with applicable EPA QC guidelines.

Each laboratory project is monitored through application of a QA/QC program, which includes the following elements:

- Centralized Project files
- Written Standard Operating procedures
- Rigorous Chain-of-Custody procedures
- Documentation of nonconformance events and corrective actions taken
- QC of data by analysis of reference samples, spiked samples, duplicates and surrogate spikes
- Periodic inspections of projects in progress
- Frequent equipment calibration and maintenance inspections
- Archiving of project records under controlled access

GPL has implemented a quality assurance program that is an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that our services meet our standards of quality with stated level of confidence.

2.0 Quality Assurance Policy Statement

Statement of Authority and Responsibility

This document is the QAPP for GPL Laboratories, LLLP. This Plan describes the activities necessary to meet or exceed the data quality objectives of GPL clients. The policies and operational procedures are established in order to meet the NELAC standards.

The Management of GPL is dedicated to the quality assurance program described in this Plan, and procedures as defined in the SOP manuals. Each manager, and supervisor as well as their staff members, as assigned accordance with the Plan, are obligated to comply with its stated requirements, responsibilities, and objectives throughout all data generating and processing operations.

All laboratory personnel are familiar with the quality policies and procedures found in this document and/or in laboratory standard operation procedures. All laboratory personnel are responsible for the implementation of quality practices in all aspects of their work associated with preparing, processing and reporting analytical information.

The QAPP has been prepared by the Quality Assurance Manager (QAM), who shall be responsible for revisions as necessary to ensure all reportable data are of uncompromising quality. The QAM has the additional responsibility and authority to terminate nonconforming work.

Approvals:	_____	_____
	Paul Ioannides, Laboratory Director	Date
	_____	_____
	Richard Turner, Assistant Lab Director	Date
	_____	_____
	George Parris, Technical Director	Date
	_____	_____
	Yemane Yohannes, QA Manager	Date

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3.0 Quality Assurance Management

3.1 Introduction

An organizational chart, which depicts the management structure at GPL, is provided on the following page. As shown, the QAM is independent of the data generating. Project Management and analytical groups.

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ORGANIZATION CHART

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3.2 Assignment of Responsibilities

The QAM operates independently of all data generating areas. The QAM reports directly to the President.

Roles and Responsibilities

The goal of the QA Program is to assure that data generated by GPL Laboratories, LLLP is of the highest quality available. To reach this goal the program seeks to develop policies and procedures to monitor, maintain and improve data quality, and maintain the necessary documentation of laboratory performance. A listing of QA responsibilities is detailed below.

Quality Assurance Manager

The QAM has overall responsibility for the development and administration of the QA Program. This effort is supported by the President, Laboratory Director, Laboratory Staff, and Administration Staff. QAM oversees and is responsible for the review of the entire technical operation of the laboratory. An analytical quality control program is conducted to ensure the production of valid data. The QAM supervises implementation of the analytical QC Program and interacts with the project staff in determining corrective action procedures.

Additionally, the QA Manager duties include:

- Preparation of written documents defining QA/QC Procedures.
- Review and approval of SOPs.
- Maintaining copies of all current procedures.
- Scheduling and performance of quality audits.

- Employee training in QA/QC techniques.
- Maintaining current knowledge of approved methods and other regulatory requirements.
- Oversight of inter-laboratory and Performance Evaluation testing programs.
- Serving as a liaison to regulatory agencies in QA matters.
- Reviewing Nonconformance Reports and corrective actions to assure that operations have been appropriately corrected.
- Informing management of the status of the QA Program.
- Continually assessing the QA program.
- Checking the outcome of QC Samples on a routine basis to assure that control limits are being met and internal SOPs for control chart analyses are followed.
- Performance of inspections of lab operations and records to assess compliance with SOPs and contract requirements.
- Reviewing and approving performance evaluation sample results prior to submission to regulatory agencies.

The QAM evaluates data and performs assessment objectively. The QAM has the final authority to stop or change any incorrect or improper sampling or analytical procedure to assure data quality.

President

The President is responsible for administrative oversight and overall operation of the laboratory. The President supervises the quality assurance officer to ensure the production and quality of all results reported by the laboratory.

Laboratory Management

The laboratory management has the responsibility for the direction of the laboratory sections to follow the QA/QC program. This obligation is met through the following steps:

- Recruiting, hiring, and training of suitably qualified personnel.
- Allocation of sufficient resources including staff, time, materials and equipment, to complete required tasks.
- Integration of Quality Control measures into the Job Descriptions of laboratory personnel so that each employee is responsible for the quality of the work they produce.
- Effective response to corrective action requirements identified by QA.
- Assignment of SOP development as required by QA.
- Review and approval of SOPs.
- Review and approval of final reports.

Laboratory Supervisors

Laboratory Section Supervisors are an integral part of the implementation of the QA/Quality Control program. Each Supervisor is responsible for the quality of the data generated by their group. All activities performed in the lab section must comply with the internal SOPs and individual contract requirements. It is the responsibility of the Supervisor to train analytical personnel, prepare and update SOPs for each operation, and instruct analysts to perform QC checks at the appropriate intervals. The Supervisor reviews data and assures that all QC criteria for each data set have been met before releasing results for reporting. Additionally, it is the responsibility of the Supervisor to document nonconformance events and corrective action taken.

Chemists and Lab Technicians

It is the responsibility of the individual analysts to follow the appropriate methods, documenting the activities and results concisely, and implementing the QC checks as required by the contract and/or SOP manual. The analyses are expected to produce data of measurable quality and, therefore, must evaluate the outcome of QC samples as part of the regular analytical procedure. Individual analysts, as the first line of quality control, must identify quality problems and initiate a Nonconformance Report.

Any employee, who notices any deviation of the GPL Standard Operating Procedures practice in the laboratory resulting in a potential hazardous, and/or unsafe situation, has the authority of stopping the work process and must inform the laboratory management immediately.

RSO Duties and Responsibilities

- Review and approved designation of use areas and all protocols for use of radioactive material.
- Distribute and receive personnel monitoring devices (dosimeters), timely review of results, maintenance of dosimetry records, and issuing reports.
- Provide for the training of all radioactive material users and initial and annual refresher training to ancillary personnel and maintain records of this training to include topics covered, the amount of time spent, the date, instructor, and student names.
- Supervise and coordinate radioactive waste disposal, including the maintenance of waste in storage and disposal records.
- Maintain records of radioactive materials inventory, receipt and transfer of licensed material, radiation surveys and audits, waste disposal, instrument calibration reports, and personnel dosimetry reports.
- Provide supervision and assistance for the management of emergency, accident, spill, or exposure situations.

- Conduct health physics surveys of all laboratories or areas where radioactive materials are used or stored.
- Ensure that the terms and conditions of the radioactive materials license are met and that the license is amended for changes in the use of radioactive material, responsible individuals, or commitments provided to the State of Maryland in the licensing process.
- Ensure that licensed materials are properly secured against unauthorized removal at all times when not in use.
- Ensure that the radiation safety program is reviewed at intervals not exceeding 12 months.

Safety Officer (SO) Duties and Responsibilities

- Provide safety orientation training and basic safety equipment to meet staff needs.
- Develop and implement a program of periodic safety and health inspections.
- Ensure that safety equipment is properly maintained.
- Assist in the selection and/or development of safety and hygiene training materials and coordinate and document the presentation of training programs.
- Coordinate the correction of unsafe conditions, including modifications of operating procedures and changes in physical facilities.
- Maintain records of accidents and incidents in accordance with OSHA requirements (e.g., OSHA log).
- Document and respond to safety concerns and/or complaints from GPL staff.
- Provide and maintain an inventory of health and safety equipment and a library of health and safety information.
- Provide technical assistance for GPL and client

Temporary Absence of Key Personnel

In the absence of key personnel, the Laboratory Director/General Manager assigns the backup who will take over the responsibilities of the temporarily absent employee.

If the Laboratory Director/General Manager is temporarily absent, the Assistant Laboratory Director or Technical Director takes over the responsibilities.

3.3 Communications

The QAM communicates with other laboratory sections in two predominant methods, by scheduled meetings and by memorandum or report.

Production meetings are held daily; the attendees of these meetings are the Project Managers, Laboratory Section Managers, and Supervisors. The QAM attends the meetings when QA concerns or issues need to be addressed.

Production planning, marketing efforts, and laboratory management issues are discussed. This forum provides immediate access to responsible individuals for the resolution of QA concerns.

In addition, on a regular basis, a meeting is held with the President, QA Manager, Laboratory Management and Senior Project Managers to evaluate all QA related issues.

Reports are issued to document findings of audits, inspections, and data reviews performed by the QAM. Reports are issued to supervisors responsible for the work reviewed, and to lab management. The Supervisor responds to each of the findings and documents corrective actions. The report is then reviewed by the lab managers. QA verifies that corrective actions have been implemented and then files the report in QA files.

Communicating project specific requirements will be accomplished by issuing "project outlines" to each department manager, detailing the differences from standard methods. Changes in work requirements will be handled in the same manner.

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3.4 Document Control

QA reports are maintained in locked file cabinets, which are separate from other study records. QA records are often direct and forthright in addressing problems and to allow these records to become public knowledge would hinder the performance of the QA Program. Thus, these records are considered most confidential and are not available for inspection by persons outside the company, without the consent of the client.

Original copies of SOP documents are maintained in the QA files. Additionally, a historical file of obsolete SOPs is also maintained. When a SOP document is revised and replaced by a new version, the original is marked "Obsolete". The document is then placed in the historical file while the new version is placed in the current SOP file. New versions of SOPs are distributed to the laboratory, while old versions are removed. Distribution lists of SOP documents are maintained by the QA.

Document control of QAP and SQAP are basically the same as that described for the SOP documentation described above. A current and historical file system, distribution list and limited copies of the document are used in the production of the QAP and SQAP to maintain its integrity.

The minimum review frequency for all controlled documents is annually. If any revision is necessary, the distributing of updated controlled documents is also enforced.

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3.5 **QA Program Assessment**

QAM Assessments

The QAM conducts assessments of the total QA Program. The review shall take account of reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of interlaboratory comparisons or proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions and other relevant factors. Based upon these assessments, and an annual review of the QA Program Plan, an annual written status report of QA activities and progress is forwarded to the President. This report is used to define areas of focus for the coming year and will determine changes required in the QA Program Plan. This report shall include such information as:

- Status of or changes to QA Program Plans.
- Status of QA project plans, if any.
- Measures of data quality.
- Significant QA problems, accomplishments, and recommendations.
- Results of performance audits.
- Results of systems audits.
- Summary of QA training, if applicable.

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4.0 Personnel Qualifications

4.1 Introduction

GPL has over 60 employees within the Laboratory having the scientific and technical expertise needed to serve the analytical needs of our clients. These employees have been chosen based upon their education, training and experience to successfully perform their assigned tasks.

GPL provides its employees with opportunities for continuing education and training to enhance employee growth within the company. The benefits of supplying continuing education and training, and on the job experience are not only for the individual employee. The company benefits also, since it profits by the stability of the work force and the internal promotion of its employees. Finally, the benefits to the clients are that GPL provides confidence in the precise and accurate performance of contracted analyses.

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4.2 Qualifications

GPL has minimum education and experience qualifications for all job grades within the laboratory. In-house training programs and policies augment these basic education and experience requirements by supplying additional information about technical subjects, safety, corporate policy, quality assurance, and supervisory and managerial techniques.

Documentation of personnel qualifications and training is accomplished through the use of a standardized qualification system. For each position critical training and skills requirements have been identified including: organizational orientation, safety training, quality control procedures training, technical training and analytical skill requirements. Completion of each of these requirements is documented in the employees training, experience, and qualifications file by the signature of the trainer. The employee must have acceptable training and, where necessary have shown proficiency in each area before the trainer or supervisor documents qualification. The training and qualifications files are maintained by the QA and permanently archived in our on-site storage location.

Resumes of laboratory personnel are available upon request.

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4.3 Training

New employees are trained on a one-on-one basis by their supervisor or assigned individual. Training is initiated by discussion of the applicable method document for a particular analysis. The procedures as described in the methods are then demonstrated by the trainer, to be repeated by the new employee, on a set of trial samples. Results of the trainee's analysis, and an appraisal of techniques used are reviewed by the trainer. Successful results and suitable techniques are the basis for determining the qualification of an analyst in performing a particular procedure. Failure in either of these areas must result in additional one-on-one training. Until the trainer is satisfied with the overall performance of the new employee, the new employee may not perform analysis on client-supplied samples.

After initial training, an employee's performance is monitored by the supervisor for compliance with quality, production and safety goals.

Documentation of employee training procedures is accomplished through the employees training, experience, and qualifications files as described in Section 4.2. Additionally, training is routinely performed upon the introduction of new instruments into the laboratory. Generally, the instrument manufacturer who may issue training certificates upon successful completion of the course provides these courses. Copies of such certificates are to be placed in the employees' qualification files.

Training is sometimes provided in the form of seminars presented to explain new methods, techniques and procedures. These seminars, in most cases, are presented by senior level personnel to benefit employees.

Each employee is trained under the Maryland Right-to-Know statute. We believe that employees well trained in safety issues, while working in a safe environment produce a better quality product.

Each employee is also trained in ethics, confidential information and conflict of interest, with special emphasis in data fraud and inappropriate practices. The information is documented in the "Ethics and Data Integrity Agreement", which is accepted and signed by all employees, and kept as part of their training records.

SOP E.8 "Laboratory Personnel Training and Qualifications" is the procedure for establishing that personnel are adequately experienced in the duties they are expected to carry out or receive any needed training.

SOP E.8 "Laboratory Personnel Training and Qualifications" also documents the required training such as safety, general laboratory procedure, laboratory quality assurance program and demonstration of capability. The overall performance of each employee is re-evaluated at a minimum of at least once a year.

5.0 Facilities, Equipment and Services

5.1 Introduction

GPL is located in Frederick, Maryland (north of Washington, DC) along the I-270 technology corridor. The facility encompasses nearly 18,732 square feet and includes laboratories, private offices, a data processing area, a copy and graphics area, and an administrative area. Electrical power is supplied by Allegheny Power, with a service capacity of 1600 amperes at 480/277 3-phase volts. All entrances to the facility are locked and alarmed after hours. Access is controlled by the use of cipher locks on doors leading to critical areas and by magnetic key locks for exterior doors. Visitors are escorted while in the facility by members of the staff after the visitor has signed-in. The entire facility is provided with a sprinkler system for fire protection. Additionally, there are fire extinguishers throughout the building and emergency showers, fire blankets, and eyewash stations located in the laboratories.

The laboratory has a full complement of support equipment and instrumentation, such as hoods, refrigerators, freezers, ovens, autoanalyzers, a Type II water system, etc. All instruments are maintained by trained employees, and by manufacturer service personnel, in some cases, working under service contract for critical equipment. The support equipment maintenance is described in the appropriate SOP for each piece of equipment. Acceptance criteria are also listed within each SOP. Instrument logbooks are maintained for each individual instrument in each of the laboratories.

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5.2 Laboratory Facilities

The analytical laboratories adjoin the administrative offices In order to provide close interaction between management and the analytical staff. Figure 1 presents a floor plan of the facility. Laboratory environmental aspects, which could affect the quality of data generated, are discussed below.

Environmental Control

The facility is divided into sixteen (16) zones, each with separate air handler and electronic control systems. The office and support areas are served by five of the units while the lab areas are served by the remaining eleven. These units are maintained by a local HVAC contractor who has a service agreement with the landlord. Filters on the units are replaced on a quarterly basis to reduce dust and pollen infiltration into the facility. Temperature is maintained between 68°F and 72°F to prevent temperature-induced artifacts in the data obtained from the instrumentation. Laboratory hoods are required to have a face velocity of at least 60 linear feet per minute flow at all points across the hood face. The individual section shall be responsible for the maintenance of those compliance check records. Metro Enterprises, a general cleaning contractor has the responsibility of maintaining general housekeeping. Wet mopping of all floors is required at least twice weekly to provide for additional dust control. All technical employees have an unencumbered work area to ensure that adequate working conditions are available for the tests. All labs and office areas are adequately lighted with fluorescent-type lighting. Emergency battery powered lighting is installed in all areas in the event of total power failure.

Electrical Power

Power is supplied to the facility via underground cable by Allegheny Power. Service capacity is 1200 amperes at 460 volts. Transformers are used to provide the proper voltages needed for the instrumentation and mechanical systems; i.e., 115 volts, 230 volts, and 208 volts 3 phase. Dedicated circuits supply power to the instrumentation to limit inter-instrument interferences often seen with computer-controlled instruments, which use switching-type power supplies. Three-stage surge and spike suppression equipment is employed on instrumentation sensitive to this type of power problem.

Laboratory Utilities

The laboratory benches are supplied with electrical power, compressed air, vacuum, hot and cold potable water, and Type II reagent water utilities. Compressed air and vacuum systems are maintained by the section supervisor. Hot water is supplied by an electric water heater.

The laboratory complex is equipped with a water system capable of supplying the laboratory with Type II reagent water. The system is located in the cylinder/DI water area and distributes water throughout the laboratory to the following areas: glassware preparation, wet chemistry, organic sample preparation, metals sample preparation, and organic and inorganic instrumentation. The system has incoming municipal water which is filtered, softened, and processed through a reverse osmosis membrane for storage in the permeate water tank. Water drawn from the tank for distribution to the users is passed through carbon beds, mixed resin deionizing beds, an ultra filter and finally sterilized by UV radiation before being circulated to the laboratories. Water returning from the recirculation system is reintroduced to the system at the carbon bed filtration point. The system is maintained by service contract personnel.

Laboratory Facility Safety Engineering

Laboratory safety is regarded as a serious responsibility. The laboratory maintains special solvent storage and waste storage areas.

- Solvents are stored in the solvent storage cabinet. Bulk solvents are stored here while small quantities of solvents for immediate use are stored in flammable solvent lockers beneath the laboratory hoods. Corrosive liquids are stored separately in corrosive liquid storage lockers.
- Waste solvents are placed in waste solvent containers for transfer to 55-gallon drums in the waste storage facility. This facility provides an area, which is designated, for the accumulation and storage of laboratory wastes prior to shipment.
- The laboratory is equipped with dry chemical, carbon dioxide, and halon fire extinguishers strategically placed throughout the lab. Locations for eye wash stations and emergency showers are VOA, Metals, Metals Digestion, Wet Chem/Organic Extraction areas. Safety glasses are issued to each employee for use in the laboratory.

Figure 1
Facility Floor Plan

- 1 –Office
- 2 –Office
- 3 – Reception Area
- 4 – Front Conference Room
- 5 – Office
- 6 – Office
- 7 – Office
- 8 – Office
- 9 – Office
- 10 – Office
- 11 – Office
- 12 – Office
- 13 – Wet Chemistry Lab
- 14 – Office
- 15 – Archive Storage
- 16 – Reporting Dept.
- 17 – Walk-in Cooler
- 18 – Waste Disposal
- 19 – Lab
- 20 – Bottle Storage Area
- 21 – Sample Management
- 22 – Extraction Lab
- 23 – Glassware Prep. Area
- 24 – Wet Chemistry Lab
- 25 – Radiochemistry Lab
- 26 – Radiochemistry Lab
- 27 – Server Room
- 28 – Women's Restroom
- 29 – Custodial Storage
- 30 – Men's Restroom
- 31 – Metals Digestion Area
- 32 – Cylinder/ DI Water Area
- 33 – Tel/Gas Area
- 34 – Sprinkler Area
- 35 – Electrical Room
- 36 – VOA Lab
- 37 – SVOA/GC Lab
- 38 – HPLV/IC Lab
- 39 – Metals Lab
- 40 – Back Conference Room
- 41 – Break Room
- 42 – Kitchen

5.3 Instrument Maintenance

In an effort to reduce unexpected instrument failure, ensure reliable and accurate data generation, and control the costs associated with non-routine maintenance and down time, the laboratory has implemented a preventative maintenance system. Routine preventative maintenance is performed as suggested by the manufacturer. When discovering that maintenance is required more frequently or that additional maintenance is required, the information must be documented.

A written SOP entitled, "Instrument Maintenance", documents laboratory equipment information for all instruments. The SOP describes the methods for routine inspection, cleaning, maintenance, testing, calibration and/or standardization. Materials and standards required to perform these operations are specified and are kept in stock.

The temp. monitoring SOP addresses the monitoring of ovens, freezers, refrigerators and incubators. Temperature logs (including acceptance criteria) are assigned and are monitored and documented daily. SOPs for balances and pipettes also exist and are monitored and documented daily which constitutes a significant part of the overall QA Plan. In addition, corrective action forms are routinely completed, documenting the performance of each support piece of equipment, within the lab.

Written records are maintained to document all inspection, preventative and non-routine maintenance, test, calibration and/or standardization procedures. The documentation must include:

- Name of item;
- Manufacturer name;
- Model and serial number;
- Manufacturer's instructions;

- Date received;
- Date placed in service;
- Current physical location.

The records include date, description of activity, actual findings, the name of the person performing the operation and a statement as to whether the maintenance operations were routine or unscheduled. Non-scheduled repairs performed as a result of equipment malfunction are documented in the instrument logbook to show the nature of the problem, when the problem was discovered and remedial actions taken. Repairs made by the manufacturers instrument repair technicians must also be documented and the service reports filed in the instrument logbook. Following major maintenance activities, instrumental return to analytical control must be demonstrated in the maintenance records prior to analysis of samples.

On-site instrumentation service is available on and as needed basis usually within 24 hours. The on-site service includes hardware support for all GC, GC/MS, ICP, AA, and other analytical instruments.

All Tekmar LSC2000 and Varian Archon closed system autosamplers are under service contract. Professional Technical Services (PTS), a reputable instrument service company, maintains the routine maintenance and any instrument repair needs that arise from time to time.

5.4 Laboratory Materials Procurement

Each chemical purchased for laboratory use is ordered by specifying the grade required for the intended use. Persons who place the orders are not permitted to make any substitutions without authorization from the Section Manager. This restriction is intended to avoid inadvertent purchase of materials of substandard quality. The grades typically used include the following:

- Technical – used for cleaning or non-quantitative purposes.
- Purified – used for some qualitative analytical work where purity is not critical and specific contamination is noted to be absent.
- ACS Reagent – used for analytical work.
- Spectrograde – used in IR, AA, and UV applications.
- Pesticide Grade – used for pesticide determinations and other GC applications.
- Primary Standard – used for preparation of standards, calibration, quality control, and standardization.

Standards for organic compounds are typically obtained as concentrated solutions from a commercial source. Metals standards are obtained from commercial sources as 1,000 or 10,000ppm certified solutions. Standard materials for inorganic parameters are typically primary standard grade, when available, or analytical grade. Independent quality control standards are from a commercial source also. QC standards must be certified when obtained from a commercial source and must not originate from the same lot as materials used for calibration.

All reagents, acids, solvents, standards, and other chemicals are dated upon receipt and when opened by the technician. If an expiration date is supplied by the manufacturer, the material is discarded after that date. If manufacturer's expiration dates are not provided, the laboratory must assign an appropriate expiration date, based on professional judgement and in consideration of the shelf life for similar materials at similar concentrations. The technical basis for each such determination must be documented by the section supervisor or a senior chemist. If a specific method of analysis requires a shorter lifetime, then the specific method is followed accordingly. As part of the regular laboratory inspections performed by the QA, reagents, acids, solvents, standards, and other chemicals in the laboratory will be randomly checked for expiration date. If materials are found which are past the expiration date, the section supervisor will be immediately notified to institute corrective actions.

Solvents are stored in a locked solvent cabinet. Individual bottles of solvents may also be stored in the "flammable" cabinets located under the laboratory hoods. Acids are stored in a safety cabinet for corrosives and in "corrosives" cabinets located under fume hoods. Dry chemicals are held on designated shelves at ambient lab temperature. Organic compound standards are stored in several small freezers, which are dedicated to standards only. Standards for inorganic compound analysis are stored under refrigeration, while standards for metals analysis are maintained in room temperature cabinets.

To control quality of purchased chemicals, the oldest supply is used before a new bottle is opened ("first in, first out"). Analysts are responsible for checking the appearance of the chemical prior to use to assure that the physical state of the material is correct. Purity and stability of reagents are monitored by performing blank determinations and QC samples along with analytical batches.

Additionally, each manufacturer's lot of solvent is checked for potential contaminants by analyzing the solvent through the appropriate method. If a lot has not been accepted based on this prescreening check, it is not released from the solvent storage room.

The procedure for laboratory glassware cleaning is defined in SOP, "Glassware Washing Procedures".

The procedure for radiochemistry materials is defined in SOP, "Ordering and Receiving Radiochemistry Materials".

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6.0 Data Generation

6.1 Quality Assurance Project Plans

Large contracts for selected projects require the development of and the adherence to a Quality Assurance Project Plan. The USEPA document, "EPA Requirements for Quality Assurance Project Plans" EPA QA/R-5, Nov. 1999, is used as general instruction for writing the Quality Assurance Project Plan. Specific requirements of the client are incorporated into the document. This Quality Assurance Project Plan contains the elements as follows:

- Title and Approval Sheet
- Table of Contents
- Project/Task Description
- Project/Task Organization
- Documentation and Records
- Quality Objectives and Criteria for Measurement Data
- Sample Handling and Custody
- Instrument/Equipment Calibration and Frequency
- Analytical Methods
- Data Review/Verification and Validation
- Quality Control
- Assessments and Response Actions
- Instrument/Equipment Testing, Inspection, and Maintenance
- Reports to Management

Quality Assurance Project Plans provide for the review of all activities, which could directly or indirectly influence data quality, and the determination of those operations, which must be covered by SOPs. Activities to be reviewed may include:

- General Project Management Design
- Specific Sampling Site Selection
- Sampling and Analytical Methodology
- Probes, Collective Devices, Storage Containers, and Sample Additives or Preservatives
- Special Precautions, such as heat, light, reactivity, combustibility, and holding times
- Federal Reference, Equivalent or Alternate Test Procedures
- Instrument Selection and Use
- Calibration and Standardization
- Preventive and Remedial Maintenance
- Replicate Sampling
- Blind and Spiked Samples
- Collated Samplers
- QC Procedures, such as intra-laboratory and intra-field activities and inter-laboratory and inter-field activities
- Documentation
- Sample Custody
- Transportation
- Safety
- Data Handling Procedures
- Service Contracts
- Measurement of Precision, Accuracy, Completeness, Representativeness, and Comparability
- Document Control

Quality Assurance Project Plans are prepared in document control format, with provision for revision, as needed, and with a record of the official distribution.

The quality requirements of proposal requests from prospective customers shall be identified upon the initial review and evaluation of the requests. When the quality requirements have been identified, the designated QA staff member shall ensure that they are adequately addressed in the Project Plan.

The following are QA Program Objectives to be met as a project becomes operational:

- Development of a Quality Assurance Project Plan for the project, if required by the customer, or upon management request.
- Assignment of responsibilities for achieving the required quality of materials, services, and quality assurance.
- Organizing and staffing appropriately to implement quality assurance activities.
- Development of working plans and procedures to implement the QA Project Plan.
- Implementation of the Quality Assurance Project Plan.
- Coordination of QA activities with the customer, subcontractors, suppliers, etc.

The contractual requirement for a Quality Assurance Project Plan will be identified by the project management group at the initial review stage of the Request for Proposal (RFP). The Quality Assurance Project Plan will be prepared by a team consisting of the project management group and the section managers. Necessary personnel from each of these groups will review the final document to assure that it is accurate and complete. After approval, copies of the Quality Assurance Project Plan are distributed to all laboratory personnel with supervisory responsibilities involved with the project. The Project Management group coordinates contract with the client regarding development and implementation of the Quality Assurance Project Plan. Any variance of standard methods will be reported to the client prior to the analysis. The approval or acceptance of methods will be determined by the client.

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6.2 Standard Operating Procedures

Standard Operating Procedures (SOPs) are utilized by GPL to define exact routines to be followed in each section. There are SOP documents covering all aspects of the laboratory operation, from sample receipt and analytical methodology through data review and archiving. The entire SOP Manual is available for review during client visits. A copy of the SOP Manual Index is provided as Appendix F.

Each SOP document is individually reviewed and approved. A Document Control System has been designed for SOP documentation and a historical file is maintained. SOPs are identified by a SOP numbering, revision identification system and an effective date administered by QAM. Obsolete documents are maintained in a historical file where they are marked obsolete. Standard Operating Procedure documents are reviewed at least annually to determine if updating is required.

SOP documents may be initiated by the lab director or section manager/supervisor. The proposed document is submitted to QA, which, after review, circulates the draft document to the department management and the lab director for comments. The draft document and management comments are returned to the originator for resolution. The revised document is then circulated by the QA for approval signatures. Each SOP must be signed by the originator, the section supervisor and manager, and the lab director.

Each laboratory is furnished with a SOP Manual. Additionally, the SOPs that are specific to a particular area may be prepared as a quick reference; i.e., glassware washing procedure.

The QAM has a critical role in the establishment and maintenance of the SOP documentation program. The QAM prepares or assists others in the preparation of many SOP documents, is responsible for the circulation and review of draft SOPs, for maintenance of the SOP document control system, including the historical file, and the distribution of the SOP manuals to the lab. All laboratory employees are responsible for reading, understanding and following SOPs particular to their designated job function.

6.3 Sample Chain-of-Custody

All incoming samples are delivered to the Sample Control office for inspection, log-in, and storage. Immediately upon receipt, the sample set is unpacked and checked versus any accompanying client paperwork. All the documents, like the field COC, the courier airbill, etc. become part of the client file for the said sample batch. If a field chain-of-custody sheet is received with the samples, it is the responsibility of the Sample Coordinator to sign for laboratory custody.

The Sample Control inspection of the samples include the following checks:

- Custody seal status
- Sample container integrity
- Cooler temperature at time of receipt
- Type of container (plastic or glass)
- pH of sample if chemical preservation is required (not applicable for VOA analysis)
- Volume of sample
- Sample identity

The procedures for inspection of samples and EPA requirements concerning sample preservation and holding times are detailed in SOP "Sample Receipt, Inspection, Preservation, and Storage Condition Requirements". Procedures utilized in the logging of samples are detailed in SOP "Sample Logging and Record Keeping" and SOP "Secure Sample Storage".

GPL normally provides all sample bottleware and containers from its laboratory facility. All sample containers are Class I (I-Chem 300 or equivalent), precleaned, tested and are accompanied by the batch certificate of analysis. The GPL SOP titled "Sample Container Quality Assurance Program" clearly describes a program whereby the laboratory provides fully traceable, properly documented sampling bottles of known quality to field sampling operations.

The results of the incoming sample inspection are documented on the Sample Receipt Form. The Sample Receipt Form is the basis of the sample management system, which is described in detail in Section 6.4 of this Plan.

The GPL laboratory management reserves the right to refuse acceptance of any sample that could present a potential danger to the health and safety of the laboratory employee. GPL policy is to accept environmental samples that present no known immediate hazards and will only accept samples that contain radiological constituents, which are within the limits of GPL radioactive license.

Samples are assigned a unique, sequential number during the logging process. GPL utilizes an internally developed LIMS software package over client/server network. The system generates individual sample labels, which list the GPL sample number, the client's sample ID, test to be performed, sample location and sampled date. These labels are placed upon each sample bottle.

The samples are stored in locked sample storage areas by Sample Control. Distribution of samples to the laboratory and corresponding return of samples is documented via signatures on a system-generated chain-of-custody form. The Sample Control Staff is responsible for the documentation.

Commercial samples are kept for at least 90 days from the date that the samples are received. All samples for DOE projects are kept for six months. After the archival date the samples are disposed of unless otherwise specified by the client. Disposal of all samples must be recorded in the sample disposal logbook.

The extracts and digestates are under internal COC procedures. When extracts and digestates are transferred, the digestate/extract transfer form is completed, both by the person relinquishing custody, and the person assuming custody. Extracts and digestates submitted after the analyses of the digestate/extract is completed, the extract/digestate will be retained for the period of time specified by the method, project or program. If requirements are not specified, the default

retaining time is 90 days following data submission. At which time, the extract is transferred to sample management for disposal, the digestate is disposed by the lab technician. The internal COCs of digestates/extracts are kept on file after disposal.

SOP "Laboratory Waste Handling and Storage Program" details the procedures used to handle, label, store and dispose of both hazardous and non-hazardous laboratory wastes including sample, sample byproducts, waste chemicals and spent solvents.

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6.4 Sample Management

GPL uses two techniques as part of its complete sample management program, LIMS generated printouts of assignments, work backlog and a centralized project filing system. Each function will be described in detail below.

As discussed in Section 6.3 of the QAP, the Sample Receipt Checklist Preservation Form for documenting incoming sample inspections is completed by the sample control personnel. After this step, the Sample Receipt Form and a copy of the field paperwork or client paperwork, which arrived with samples, is used for initial login into LIMS system. Project Management compares the submitted information to the client requirements to assure that the sample set agrees with the work arranged via previous communication. Project Management then checks the test codes required for each sample, if not previously established. Special instructions communicated to the lab regarding report due date, sample preparation, QC requirements or special handling procedures are also recorded by project management. The initial login paperwork, after being examined, is returned to Sample Control for distribution of the sample set.

Each set of samples, which is received from a client, during the same time period, is assigned to a Work Order.

Work Order numbers consist of a set of numbers as follows:

702004

Where: 1 signifies the year – 2007
 02 signifies the month – February
 004 signifies the fourth Work Order assigned in that month

In cases where more than 999 samples sets are entered into the system within a given month the system automatically changes the first digit of the three digit Work Order number to the letter A and increments through the alphabet.

Individual samples are labeled with the work order number and a suffix code of three digits. In the suffix, the three digit (001) number indicates a sample identification and the next figure (01, 02, 03) denotes a sample fraction.

Example:

7 02 004 – 001-01

001-02

001-03

7 02 004 – 002 (different sample)

After log-in, hard copy printouts are generated from the database. Sample Control maintains a printout of the Work Order and Chain-of-Custody form.

Project management initiates the project file by placing the sample receipt form, original client paperwork, and corresponding LIMS printouts of work orders into a file folder labeled by Client and Work Order number. The project manager places the project file in the controlled access active central file location. This allows supervisors access to this file for additional information during normal work hours. File security is maintained through restricted access.

Worksheets generated by the LIMS system is electronically transmitted to the section supervisors. The LIMS system has been programmed to create a separate Work Sheet for each department. The Work Sheet contains essential information such as sample identification, test required, due date to comply with both methods required holding times and the date which results are due to the client.

Each supervisor is responsible for assigning analytical batches for processing. The supervisor distributes a list of samples to be analyzed, the name of the tests to be performed, and the analytical protocol to be followed including quality control samples and any special instructions. The actual documentation used to prepare the batch assignments may vary according to the type of test performed.

All study data are filed in the central project file. As each test is completed, the LIMS database is updated to close out the test. Each day, a printout is obtained from LIMS, which lists, by test, all samples received but not yet analyzed. These reports are used by department supervisors to coordinate work assignments.

Reports of analytical results are tabulated and placed in the central project file. All correspondence, verbal or written, internal or external, is documented in the central file. The Project Manager monitors the progress of each project and reviews the final report. All reports are reviewed and signed by the Lab Director. A copy is placed in the central project file. As work is completed, its status is changed to complete and thus removed from the work schedule. When the work is actually reported its status will once again be changed to reported to indicate that no further work is required.

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6.5 Additional Procedural and Calibration Requirements to Achieve QA Objectives

6.5.1 Organics

6.5.1.1 Sample Preparation

Three (3) surrogate standard compounds are added to each organic sample requiring GC/MS volatiles analysis as per methods SW846 8260 and 40 CFR624. When the methods require a different number of surrogates (such as 524.2) the analyses are performed as per the method. Six (6) surrogate compounds are used for semivolatile analyses (SW846 8270 and 40 CFR625). CLP and its revisions, require eight (8) surrogates, which are utilized when the method is performed. For pesticide and herbicide analysis at least one (1) surrogate is utilized as per the method. The laboratory may also use two (2) surrogates when specified in the methodology. For explosive residue analysis one (1) surrogate is utilized. These surrogate compounds are quantitatively analyzed in the GC/MS, GC or HPLC phases. Control limits for surrogate compounds are maintained. This data forms the statistical basis upon which preparation techniques are monitored. Surrogate recoveries must meet acceptance criteria before the analytical data will be released. In some instances, the sample matrix may produce interferences, which adversely affect recoveries. These interferences must be confirmed by a re-analysis and/or re-preparation of the samples. Affected data are qualified in the report.

One method blank is prepared and analyzed for each analytical/prep batch. A batch consists of 20 samples undergoing simultaneous processing. The purpose of the method blank is to ensure that contaminants are not introduced by the glassware, reagents, personnel, sample preparation or sample analysis environment.

6.5.1.2 Standards

Calibration standards are traceable to the National Institute of Standards and Technologies (NIST) or EPA whenever such standards are available. Commercial sources of standards and reagents are checked for purity, and approved prior to use. All standards prepared for use throughout the organics laboratory are logged into solutions manager, which gives a unique identification. This unique identification, along with receipt date is written on the Certificate of Analysis, and on the bottle. The Solutions Manager prints out a receipt report with the manufacturer, vendor, catalogue number, receipt date, expiration date and lot number.

6.5.1.3 Instrumentation

- Gas Chromatography/Mass Spectrometer (GC/MS)

The Gas Chromatograph/Mass Spectrometer analyses are an integral part of the analytical services provided by GPL. The analyses involve very sophisticated instrumentation, which is operated by a highly trained staff. To assure that the results from this phase area of the highest quality, a rigorous program of calibration and quality assurance has been established.

Prior to the utilization of the instrumentation, the instrument performance is adjusted to assure that all manufacturer's and accrediting body's performance criteria are met. The instrument's performance is monitored and control charts exhibiting instrumental response have been established. The instrument is continually monitored and is adjusted on an as needed basis (specified in the Standard Operating Procedures).

When needed, the mass spectrometer is adjusted to meet the method defined tune criteria, using FC-43. Every 12 hours Bromofluorobenzene (BFB) or Decafluorotriphenylphosphone (DFTPP) is then used to confirm that the instrument meets this criteria. The BFB ion abundance criteria is outlined within the particular methods and must be satisfied for all volatile organic analyzes. The DFTPP ion abundance criteria is also outlined within the applicable methods and must be satisfied for all semivolatile organic analyzes. After confirming that the tuning criteria have been satisfied, the instrument is calibrated for the analytes of interest.

The analytical procedures followed for analyses for both volatile and semivolatile organic compounds involve an initial and continuing calibration of the instrument. This calibration is performed using multiple concentrations of standards as specified in the appropriate method. The validity of the calibration standard is confirmed using an EPA traceable standard mix containing known concentrations of each analyte. On a daily basis, the instrument calibration is confirmed to be unchanged by

analysis of a single standard. The standard must meet the criteria as outlined in the method.

After calibration, a method blank is analyzed to demonstrate that the system is virtually free of any of the analytes of interest. The method blank consists of organic free water for aqueous analyses and purified soil matrix (e.g., Ottawa sand) for soil samples. After demonstration that the system is free of contamination, sample analyses are begun. Maximum allowable levels of contamination are less than or equal to the contract required quantitation limit (CRQL) for most organic compounds and up to 5X the CRQL for common laboratory contaminants as defined in the EPA Statement of Work for CLP analysis. For non-CLP methods, the acceptance criteria should be at least one half of the method reporting limit.

- Gas Chromatography (GC)

Pesticide, Herbicide, Polychlorinated Biphenyl (PCB), TPH and selected CSM degradation compound analyses are performed using a gas chromatograph equipped with the appropriate detectors. These analyses often are performed on complex matrices, which require an experienced staff for the interpretation of the results. The analysts also must determine the clean-up requirements for each individual sample, when necessary.

Prior to all analyses, the elution time and elution order for each analyte of interest is determined. They are determined by analyses of several standards. The retention windows allowable for the identification of the

target analytes are then calculated and defined as stated in the different methodology.

The instrument is calibrated by analysis of a standard mixture, which contains the analytes of interest. The number of standards and their concentration are method specific, but all assure an accurate determination of the concentration of an analyte in the sample. The instrument's sensitivity is adjusted so that all standards are integratable and are also within the instruments linear response range.

After calibration, a method blank is analyzed to demonstrate that the system is optimized. The method blank consists of an extraction blank and must not contain any analytes of interest at or above half of the reporting limit. After demonstration that the system is free of contamination, sample analyses are begun.

- High Performance Liquid Chromatography (HPLC)

Explosive residues, nitroglycerine analyses are performed using a high performance liquid chromatograph equipped with UV and fluorescence detectors. These analyses require analysts experienced in the use of HPLC instrumentation and skilled in the interpretation of HPLC chromatograms.

Prior to all analyses, the elution time and elution order for each analyte of interest is determined. They are determined by analyses of several standards. The retention windows allowable for the identification of the target analytes are then calculated and defined as stated in the different methodology.

The instrument is calibrated by analysis of a standard mixture, which contains the analytes of interest. The number of standards and their concentration are method specific, but all assure an accurate determination of the concentration of an analyte in the sample. The instrument's sensitivity is adjusted so that all standards are integratable and are also within the instruments linear response range.

After calibration, a method blank is analyzed to demonstrate that the system is optimized. The method blank consists of an extraction blank and must not contain any analytes of interest at or above half of the reporting limit. After demonstration that the system is free of contamination, sample analyses are begun.

6.5.2 Metals

6.5.2.1 Standards

Calibration standards must be prepared fresh each time an analysis is to be made and discarded after use for cold vapor. The expiration date of standard solutions for ICP and ICPMS is three months from the date of preparation or whenever one of the certified standards expires, whichever is first.

For trace ICP and ICPMS, minimum of two standard and blank are required. A daily low level calibration verification at the method reporting limit would also be required. Source identification, analysis date and preparation procedure must be documented.

6.5.2.2 Instrumentation

ICP, ICPMS, CV

The analyses performed on the ICP, ICPMS, and CV instrumentation are an extremely important part of the analytical services provided by GPL. The analyses involve very sophisticated instrumentation, which is operated by a highly trained staff. To assure that the results from this phase of the operation are of the highest quality, a rigorous program of calibration and quality assurance has been established.

Prior to the utilization of the instrumentation, the instrument performance is adjusted to assure that all manufacturer's and accrediting body's performance criteria are met. The instrument is continually monitored and is adjusted on an as-needed basis (specified in the Standard Operating Procedures).

Instruments must be calibrated daily, once every 24 hours or each time the instrument is set up. The instrument standardization date and time must be included in the raw data.

- Initial Calibration Verification

Immediately after each of the ICP, ICPMS and CV systems have been calibrated, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of Initial Calibration Verification Solution(s) at each wavelength/mas used for analysis. When measurements exceed the control limits, Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses, the analysis will be terminated, the problem corrected, the instrument re-calibrated, and the calibration re-verified.

The initial calibration verification solution(s) must originate from a different source other than those being utilized in the standards for the instrument calibration.

For ICP, the Initial Calibration Verification Solution(s) must be run at each wavelength used for analysis. For ICPMS, the initial calibration verification solution must be run at each mas.

- Continuing Calibration Verification (CCV)

To ensure calibration accuracy during each analysis, one of the following standards is used for continuing calibration verification and must be analyzed and reported for every wavelength/mass used for the analysis of each analyte, at a frequency of 10% or every 2 hours during an analytical sequence, whichever is more frequent. The standard must

also be analyzed and reported for every wavelength/mass used for analysis at the beginning of the sequence and after the last analytical sample. The analyte concentrations in the continuing calibration standard must be one of the following solutions at or near the mid-range levels of the calibration curve:

1. EPA Solutions
2. NIST SRM 1643a
3. A Contractor-prepared standard solution

The same continuing calibration standard must be used throughout the sequence for that particular case of samples received. If the deviation of the continuing calibration verification is greater than the control limits, the analysis must be stopped, the problem corrected, the instrument must be re-calibrated, the calibration verified and the reanalysis of preceding 10 analytical samples or all analytical samples analyzed since the last acceptable calibration verification must be performed for the analytes affected.

- Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Analyses

A calibration blank must be analyzed at each wavelength used for analysis immediately after every initial and continuing calibration verification, at a frequency of 10% or every 2 hours during the run, whichever is more frequent. The blank must be analyzed at the beginning of the run and after the last analytical sample. Note: A CCB must be

run after the last CCV that was run after the last analytical sample of the run. If the absolute value blank result exceeds more than the reporting limits, terminate analysis, correct the problem, recalibrate, verify the calibration and reanalyze the preceding 10 analytical samples or all analytical samples analyzed since the last good calibration blank.

- Preparation Blank (PB) Analysis

At least one preparation blank (or reagent blank), consisting of deionized distilled water processed through each sample preparation and analysis procedure must be prepared and analyzed with every sample batch. This blank is to be reported for each sample batch, if required, and is used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner.

- If the absolute value of the concentration of the blank is less than or equal to one half of the reporting limit no correction of sample results is performed.
- If any analyte concentration in the blank is above one half of the reporting limit, the lowest concentration of that analyte in the associated samples must be 10X the blank concentration. Otherwise, all samples associated with the blank with the analyte's concentration less than 10X the blank concentration and above the DL, must be re-

digested and re-analyzed for that analyte (except for an identified aqueous soil field blank). The sample concentration is not to be corrected for the blank value.

When performing SW846 procedures, the matrix of the preparation blank is acceptable if the concentration of any analyte of concern is no higher than the highest of either: one half of the reporting limit, or ten percent of the measured concentration of the sample.

- If upon investigation, the stated criteria is unacceptable, all samples associated with the blank are re-digested and reanalyzed for that analyte.

- Spike Sample Analysis

The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. The spike is added before the digestion (i.e., prior to the addition of other reagents) and prior to any distillation steps. At least one spike sample analysis must be performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) or for each sample batch.

If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations must be performed using the results of the sample designated as the “original sample”. The average of the duplicate results cannot be used for the purpose of determining percent recovery. Samples identified as field blanks cannot be used for spiked sample analysis. The analyte spike must be added in the method-required amount for each element analyzed or as requested by the client. If two analytical methods are used to obtain the reported values for the same element within a sample batch (i.e., ICP, ICPMS), spike samples must be run by each method used.

If the spike recovery is not at or within the control limits the data of all samples received associated with that spike sample and determined by the same analytical method shall be noted in the report. An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the recovery criteria.

- Duplicate Sample Analysis

One duplicate sample must be analyzed from each group of samples of a similar matrix type (i.e., water, soil) or for each sample batch.

Duplicate sample analyses are required for percent solids. Samples identified as field blanks cannot be used for duplicate sample analysis. If two analytical methods are used to obtain the reported value for the same element for a sample batch (i.e., ICP, ICPMS), duplicate samples must be run by each method used. The relative percent differences (RPD) for each component are calculated as follows:

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Where:

RPD = Relative Percent Difference

S = First Sample Value (original)

D = Second Sample Value (duplicate)

A control limit of RPD = 20% shall be used for original and duplicates sample values greater than 5X DL (Table 6). If the duplicate sample results are outside of the control limit, the data shall be flagged on the final report.

- Instrument Detection Limit Determination

The instrument detection limits shall be determined for each instrument and performed at a frequency of once every three calendar months. The established limits must be equal to or below the levels specified the method.

The Instrument Detection Limits shall be determined by multiplying by 3 the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) or for ICPMS reagent water only at a concentration 3x – 5x the instrument manufacturer’s suggested IDL, with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDL’s must be determined and reported for each wavelength/mass used in the analysis of the samples.

Instrument Detection Limits are measured primarily for metals analyzed by Cold Vapor Atomic Absorption spectrophotometry (CV), and Inductively Coupled Plasma (ICP) and mass spec. The IDL should be determined when new equipment is acquired, after major instrument repairs, and when required by specific contracts. The IDL is obtained by the following procedure:

1. A standard is prepared at 3-5 times the level of the estimated detection limit.
2. On 3 non-consecutive days, 7 consecutive measurements on the standard are obtained. The standard is treated as a sample, with rinses or blanks run between each replicate.
3. The average of the daily standard deviation is multiplied by three to obtain the IDL.

The quarterly determined IDL for an instrument must always be used as the IDL for that instrument during that quarter. If the instrument is adjusted in any way that may affect the IDL, the IDL that instrument must be re-determined and the results submitted for use as the established IDL, for that instrument, for the remainder of the quarter. Instrument detection limits are retained and are available for inspection.

- Linear Range Analysis

For all ICP and ICPMS analyses, a linear range verification check standard must be analyzed daily. The analytically determined concentration of this standard must be within $\pm 10\%$ of the true value. This concentration is the upper limit of the ICP linear range beyond which results cannot be reported without dilution of the analytical sample.

- Laboratory Control Sample(LCS) /Blank Spike Sample Analysis (BKS)

Aqueous and solid LCS/BKS must be analyzed for each analyte using the same sample preparations and analytical methods as the samples being analyzed. One LCS/BKS must be prepared and analyzed for every batch of samples digested. If the percent recovery exceeds the internal limits, or contractor supplied control intervals, the analysis will be terminated, the problem corrected and the samples associated with that LCS/BKS re-digested and reanalyzed. The stated control limits are utilized until laboratory derived control limits are established.

On an annual basis, background correction factors are determined for ICP and ICPMS analysis using single element standards. This measure determines the potential false analyte signals caused by the presence of high levels of certain commonly occurring elements found in environmental samples.

6.5.3 Radiochemistry

6.5.3.1 Personnel

- Training

All personnel working with or around radioactive materials shall be properly trained in all areas of GPL's Radiation Protection Plan. Mandatory annual refresher training will be conducted by the facility RSO. Records of training will be maintained by the QA manager and shall be stored in each employee's respective file.

All personnel preparing samples for radioactive analysis will be trained in accordance with the laboratory SOP pertinent to the analysis at hand. Individuals must show a demonstration of capability by performing the preparation method using 4 laboratory control samples. The relative percent difference to the nominal activity will be assessed to determine the confidence of the batch and the competence of the chemist. The acceptance criterion is +/- 25%. Failure to demonstrate the capability to perform the preparation method will result in more training and may not perform the analysis without supervision of a qualified mentor.

All personnel analyzing a prepared sample on an instrument will be trained on the proper use of the instrument by a qualified mentor. The trainee may become qualified to use the instrument without supervision at the discretion and liability of the qualified mentor. Signature clearance shall go on file with the QA manager in the trainee's employee folder.

- Safety

All personnel working with or around radioactive materials will comply with the safety practices defined in GPL's General Safety Plan. This includes but is not limited to:

- Personal Protective Equipment (gloves, labcoat, safety glasses)
- Utilize operational fume hoods
- Time, distance, and shielding
- Buddy system
- RSO notification in the event of a spill or accident involving RAM

All employees have the right to stop work if he or she feels the conditions are unsafe to proceed. Notification of the RSO, Safety Officer, and/or Laboratory director is required if any such occasion arises.

6.5.3.2 Radioactive Standards

All standards shall be verified before initial use by 3 verification measurements. The mean and standard deviation of the 3

measurements will be determined. The mean value is within 5% of the certified value and the 2 sigma deviation is <10% of the mean value.

- Primary Sources are those sources that come direct from the manufacturer. These sources come typically in 5mL bottles and are diluted into the working standards. Custom standards may vary. All primary standards are valid for 5 years contingent upon preservation (HCl, HNO₃) and an annual verification. Any primary source ordered from a vendor must be NIST traceable and come with a calibration certificate to be kept on file with the Radiochemistry manager. Upon receipt, the RSO or RSO designate will assign the primary source a unique identification number under GPL's numbering system (i.e. GPL-01).
- Stock Solutions are the initial dilutions of primary sources coming in volumes <100mL. Stock solutions end volumes are usually around 100mL depending on the activity concentration post dilution. Stock solutions are preserved to 1N of either HCl or HNO₃ to help minimize plating; the preservation type shall be the same as the initial preservation. Upon creation of the stock solution, the chemist will place a label on the bottle identifying the isotope, total activity, assay date, expiration date, chemist's initials, preparation date, activity concentration, and unique identifier. For stock solutions, the unique identifier will be a subpart of its respective primary source (i.e. GPL-

01A). Stock solutions are valid for 5 years contingent upon preservation and annual validation.

- Working standards are further dilutions of stock solutions that are used for daily batch QC including matrix spikes and blank spikes. These standards are typically 500mLs and have concentrations >5 times the MDA. Working standards must not be from the same lot or batch as those standards used for calibration. Working standards shall be preserved in either HCl or HNO₃ and are good for 1 calendar year. After 1 year the source may be re-verified for For every working standard, the unique identifier will be a subpart of its respective stock solution (i.e. GPL-01A01).

- Logbooks

Tracking logbooks shall be implemented for each primary standard, stock solution, and working standard to track the amount of radioactivity used from that respective standard. These logbooks will help with inventory reporting for radioactive material and also ensures that anyone who has not been trained to work with radioactive material is not using the sources.

Standard preparation logbooks are used to link stock solutions and working standards to their respective parent standard. The logbook acts as a tool to show the dilution process of the secondary

standards. A logbook record shall be recorded for every dilution made to any standard.

6.5.3.3 Preparation and Batch Quality Control

A batch blank and laboratory control samples shall be analyzed with each batch, and a matrix spike and duplicate shall be analyzed when there is sufficient sample to do so. All batch QC types shall be prepared and analyzed in the same manner as unknown samples.

- Batch QC types

Batch blanks are used to determine the extent of cross contamination during the procedure and to identify potential contamination of the detectors, sample holders, and any reagents used during the preparation process. Blank frequency shall be 1/20 samples or 1 per preparation batch. The acceptance limit for batch blanks is < the MDA or 1.65x blank TPU when questions of detectability exist.

Blank spikes are used to determine the validity of the batch sequence and the calibration of the instrument used. A known amount of activity is spiked in a control sample (typically DI water) and prepared and analyzed along with the batch. Percent recoveries are calculated to validate the batch. Some procedures may utilize 2 laboratory control samples, where 1 is used for a batch efficiency determination and the other used for batch validation. The frequency for an LCS shall be 1/20 or 1 per preparation

batch. The acceptance limits for percent recovery are 75-125%.

Matrix spikes are unknown samples that are spiked with a known amount of radioactivity and validity is determined by percent recovery. The samples are taken through the exact process of the rest of the batch. MS are similar to bks but can also provide useful information about matrix interference. The frequency for a matrix spike for level 3 or higher packages only is 1/20 or 1 per preparation batch when sufficient sample is available and/or per client request. The acceptance limits for percent recovery is 60-140%.

Duplicate samples are used to determine precision of the preparation batch and the analysis sequence. A sample is split into 2 samples and is prepared and analyzed along with the batch. The frequency for a sample duplicate for level 3 or higher packages only is 1/20 or 1 per preparation batch when sample is sufficient sample is available and/or per client request. The acceptance limits are an RER value of 0-3.

- Reagents

All reagents shall be of reagent grade or better, and validation certificates shall be kept on file with the QA manager.

All reagents shall be given a unique identifier upon receipt in the laboratory.

Preparation of working solutions made from the primary reagents shall be recorded in a preparation logbook and labeled with the chemical, concentration, chemist, preparation date, and expiration date. All reagent working standards are valid for 1 year.

- Standards

All radioactive standards used for calibration and blank spikes shall be NIST traceable and a calibration certificate shall be present for every standard used.

Standards used for laboratory control samples and matrix spikes shall be an independent lot or batch from that derived from the standard used for initial calibration.

Standards ordered shall be measured against the Radioactive Materials License to ensure activity limits are not exceeded.

- Tracers

For certain procedures, all samples (including QC samples) are spiked with a tracer that chemically mimics and does not interfere with the target analyte through radiochemical separations.

Tracer and carriers are added at the beginning of sample preparation to accurately determine preparation efficiency.

The acceptance criterion for isotopic tracers is 30-110% and for stable carriers is 40-110%.

- Logbooks

Logbooks will be maintained for all instruments, preparations, and standards to be used as a tracking tool for daily operations within the laboratory. All logbooks will be reviewed at least weekly. Logbooks include, but are not limited to, analysis logs, preparation logs, utilization logs, and instrument maintenance logs.

6.5.3.4 Instrumentation

- Identification

Every instrument in the radiochemistry laboratory will be identified at a minimum by the make and model. If as the laboratory grows, and multiple instruments of the same make and model come online, identification of the instruments will use their respective model and/or serial numbers.

- Berthold LB 770 GFPC
- Canberra LB 4100 GFPC
- Beckman 6500 LSC
- Canberra Gamma Spectroscopy System
- Canberra Alpha Analyst Alpha Spectroscopy System

- Logbooks

Maintenance logbooks will be required for each instrument to track any and all services performed on the instruments including the date, service provided, and vendor.

Maintenance logbooks will be reviewed at the time of service.

Analysis logbooks will be required for each instrument type to track samples analyzed. The logbooks will contain such information as the GPL sample id, daily QC acceptance, date and time, detector # (if applicable), calibration date, and the analyst.

- Universal Calibration Requirements

Calibrations of each instrument will be performed annually or when major service that may have affected the instrument electronics is performed.

Calibrations will be conducive for expected radionuclides and counting geometries.

Count time for calibrations of all instruments will be set to achieve a minimum of 10,000 counts and shall achieve counting uncertainties < 1%.

Sources used for calibration will be NIST traceable and its respective certificate shall be kept on file in the Radiochemistry office.

Calibration verifications will be performed using standards of either a different lot number or different vendor than that of the source used to establish the calibration.

- Universal Daily QC Requirements

For each instrument, daily backgrounds will be run and control charted to ensure the electronics of the instrument is working properly and no contamination is present in any detector. No sample shall be analyzed on a detector that falls outside the daily background limits. For multi-detector systems, failing detectors will be re-run on the next subsequent count and control charted. Acceptance during this run will allow for those initial failures to be used for the remainder of the day.

For each instrument, efficiency checks will be run and control charted to ensure the electronics and general calibration is sufficient. No sample shall be analyzed on a detector that falls outside the daily efficiency limits. For multi-detector systems, failing detectors will be re-run on the next subsequent count and control charted. Acceptance during this run will allow for those initial failures to be used for the remainder of the day.

Consecutive failures of any detectors will result in instrument shutdown to assess the problems and recalibrate as necessary. Note: A recalibration will not be required for background failures.

- Universal Background Requirements

For all instruments and applications, long background counts (1000 minutes) will be established at least monthly and control charted and will be used for background

correction. In the case of gamma spectroscopy, an official background shall be established for only the empty shield and will be used for background correction.

All background count times will be longer than the sample count times.

- Gas-Flow Proportional Counter (GFPC)

The plateau voltage range will be 300-1500V with 50V intervals when applicable.

The isotopes used for calibration and daily checks will be either Sr-90 or Cs-137 for Beta and Am-241 or Th-230 for alpha. These sources will be of pure nuclide.

Attenuation curves and crosstalk factors will be assessed for each detector, and a minimum of 7 points will be used to determine the curves. Calibration curve validation will be performed prior to sample analysis. Crosstalk curves will be established for alpha:beta with a minimum of 7 points; a single point beta:alpha ratio will be established.

Efficiency calibrations will be re-determined after service including hardware repair, changes to the system, or changes in the quality or manufacturer of the counting gas.

Efficiency calibration will be verified daily on all detectors except when long count times are used. In the case of long count times, efficiency verifications are performed between analyses.

Check sources will be counted to a minimum of 5, 000 counts.

Efficiencies and backgrounds are verified after a bottle exchange.

Daily background checks will be counted for a period of time similar to that of samples.

Residual weights of prepared sample will fall within in the calibration range. Re-digestions may be required to achieve these masses.

The gas flow will remain on 24 hours a day. Following a gas exchange, a 15-minute purge time is required.

When the detector is first initiated, allow the high voltage to stabilize for 15 minutes.

- Gamma Spectroscopy (GS)

Will perform GS calibrations for the specific geometry and matrix considerations used in the sample analysis.

Standards reference half-life, abundance, and peak energies.

Universal background measurements used for correction will be performed monthly for all geometries.

Backgrounds are established after any system modifications.

Samples will be counted in the same source-to-detector geometry as used to establish the efficiency.

Energy calibrations are counted for a duration to give sufficient peak counts to permit measurement of peak positions with a precision of <0.5 keV.

Energy peaks cover a wide energy range. Isotope analysis that require peak analysis outside of the energy range will require isotopic specific calibration.

Efficiency calibrations shall be analyzed to 10,000 counts in the majority of the peaks from a multi-line source. This requirement will be waived for short-lived isotopes such as Hg-203.

Samples will be analyzed under the same analysis sequence procedure as that of the calibration.

Elapsed time clock will be checked after any maintenance is performed to the MCA.

DC offset and pole zero will be checked annually.

Energy calibrations will be checked semi-weekly.

Daily efficiency checks will be verified using a low-energy and high-energy gamma ray. Verifications will be counted to accumulate 20,000 counts for the low-energy and high-energy isotope.

FWHM resolution will be monitored daily and control charted.

Gamma-ray emission rate is decay corrected to time at which the count rate is measured at the mid-point of the counting period.

- Liquid Scintillation (LS)

Water samples are checked for preservation and results are recorded in the analysis logbook.

Tritium analysis requires distillation of sample and QC samples.

Counting vials shall be made of low potassium borosilicate glass and/or high-density polyethylene vials.

Batches will be dark adapted for a minimum of 30 minutes prior to analysis.

Vials will be grounded prior to analysis to eliminate static charge.

Type of water used for background measurements will be noted on the preparation document.

Water to cocktail ratio is consistent for all samples.

Instrument background used for background correction will be established weekly.

Quench curves will be generated annually and/or after any maintenance to the instrument that affects quench correction. Curve validation will be assessed prior to running any samples.

Instrument performance analysis will be assessed following any maintenance.

Quench calibrations will be counted to accumulate 100,000 counts.

Each sample and QC spectrum will be assess for correct ROIs, acceptability of peak shape, and interferences due to non-target analytes or luminescence.

H-3, C-14, and background control charts will be assessed daily.

The check sources are counted to accumulate 20, 000 counts or for at least 30 seconds.

Each LS instrument will have its own assigned set of check sources.

Flame-sealed IPA check sources shall have a clear label with date of seal, expiration date, activity, and source; and shall not be stored in direct sunlight or fluorescent light.

H-3 check source will have between 100,000-300,000 dpm.

C-14 check source will have between 30,000-150,000 dpm.

- Alpha Spectroscopy

Calibration of the alpha spectroscopy

- Channel vs. energy – monthly
- Energy calibration verification check- weekly
- Efficiency determination when the check source is outside of the acceptable control limits.
- Efficiency verification performance check- monthly
- Background determinations for each ROI - monthly

Energy calibrations

- A curve is fit for energy vs. channel.
- Slope is <15 keV
- Performed using at least 3 isotopes in the energy range of 3-6 MeV
- Final peak energy positions of all observed isotopes are within 40keV of the expected peak energy.

Background will be established and documented for each target analyte and tracer isotope ROI.

Each sample and QC sample spectrum will be assessed for correctly chosen ROIs, acceptable spectral resolution, acceptable energy calibration and interferences with the analyte and tracer ROIs. All spectrums will be saved for review upon request.

Efficiencies

- Are corrected for background in each ROI unless the background ROI is <0.5% of the total counts in the ROI.
- Is determined on at least 10,000 counts in each ROI.
- Daily checks accumulate 2,000 counts.
- Error is documented.
- Checks and limits of acceptability are documented via control charts.

FWHM <100keV for each detector.

Internal tracers will be used for isotopic specific analysis.

Internal tracers will be prepared in the same manner and treatment as target analytes.

Calibrations for energy, background, and efficiency will be performed when a new detector is put in service.

Tracers will be tested for contribution in the ROIs of the analytes of interest.

6.5.3.5 Total Propagated Uncertainty

- General – total propagated uncertainties will be reported for each radiochemical result. Calculated uncertainties reported will combine random and systemic uncertainties as a function of that particular analysis and/or batch. To match units, percentages of uncertainties will be used.

- Systemic - systemic error is the uncertainties associated within a system. This includes but is not limited to glassware, pipettes, efficiencies, and standards. Systemic errors will consider any apparatus that has an error associated with it.
- Random – is the counting error associated with the detector used for analysis, which includes the background and counting error.
- Calculations

$$\overline{TPU}_{1\sigma} = \sqrt{\sigma_S^2 + \sigma_R^2}$$

TPU will be assessed in a similar manner to establish the uncertainty for the system.

Data will be reported at the 95% confidence interval; as such a multiplication factor of 1.96 will be assessed to TPU.

6.5.3.6 Minimum Detectable Activity

- General – MDAs are assessed to determine a radioactive sample from a non-radioactive sample. An MDA will be assessed for each sample in a batch. Batch blank acceptance criteria is contingent upon being <MDA.
- Calculations

$$MDA = \frac{3.29 ((B/T_s) \times (1 + T_s/T_b))^{1/2}}{K} + \frac{3}{KT_s}$$

Where:

B = background count rate

Ts = sample count time

Tb = background count time

K = any factors that are used in the establishment of activity determination including volume, mass, efficiency, conversion factors, etc.

6.5.3.7 Batch QC Quantification

- General – for each batch of 20 samples or less, 4 form of quality control samples will be used: batch blank, laboratory control sample, matrix spike, and duplicate. Batch blank acceptance limits will be <MDA, LCS and MS will compare %R to amount added, and duplicate will utilize the RER value 0-3 to determine precision. Each analyte's SOP will have specific acceptance limits for the LCS and MS.

6.5.3.8 Radioactive Waste

- As defined in the Radiation Protection Plan, no radioactive waste shall be discarded in regular waste containers. Any sample or material suspect of radioactivity shall be analyzed and assessed prior to discarding. Discardment of any solid source into a radioactive container will be logged in a logbook with the isotope, activity, and date of disposal. Glassware suspect of radioactive contamination will be acid washed and rinsed prior to being sent for cleaning. The rinse will be captured and analyzed prior to disposal. Disposal down the drain of radioactive waste is permitted per GPL's radioactive license. Any discharge via sewer system shall be recorded with the activity, volume, and date of disposal.

6.5.3.9 Negative Activities

Reporting results will be real calculated values. Values <than 0 will be reported as a negative activity unless otherwise specified by the client. Recurrence of negative activities during particular procedures will be evaluated.

6.5.3.10 Environmental vs. High Level Samples

The laboratory shall do everything in its power to prevent cross contamination of samples during the preparation sequence. Samples exhibiting high-level amounts of activity will be segregated from those that are at environmental levels. If applicable, batch analysis shall not include both high-level and environmental samples. Backgrounds will be assessed on any detector subject to high-level samples prior to any other sample being analyzed.

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6.6 Measurement Uncertainty

All measurement processes contains a quantity of uncertainty that can be estimated. This uncertainty depends on the minor variances that are associated with the actual process, equipment, climatic conditions, etc. during the generation of each specific measurement. Measurement uncertainty does not imply doubt about the validity of the laboratory measurement, but attempts to quantitate the naturally occurring variation that occurs in every measurement process into a definable value.

Knowledge of the uncertainty associated with s specific analytical process implies increased confidence that the quantifies result falls within a range of values given by the quantitated value and the expanded uncertainty established using historical quality control data. Some published analytical methods contain an estimate of the uncertainty associated with that method; however, for most analytical quantitative processes laboratory control sample recoveries are used to generate expanded uncertainty values within the laboratory. This value in conjunction with the analyte quantitation of field samples helps to ensure the validity of the measurement result.

It is useful to recognize that measurement uncertainty of the laboratory data generated is likely to be much less than the uncertainty associated with the sample collection activities and the matrix of the samples. The estimation of uncertainty applied by the laboratory relates only to quantitative measurements conducted in the laboratory and does not relate to qualitative analyses performed. Uncertainty associated with sampling activities and related processes are not considered in the laboratory measurement uncertainty determination process. The measurement uncertainty value generated with in the laboratory should be considered the minimal uncertainty associated with the analytical process in a clean and interference-free matrix.

At the laboratory the expanded uncertainty for each analyte is calculated by multiplying the relative standard deviation of at least 50 quality control results by a factor of two. This corresponds to a confidence level of approximately 95%. The expanded uncertainty values reflect laboratory values for each given analysis. These values can then be used to calculate the confidence interval of quantitated analytes in each method. The 95% confidence interval for each analyte can be determined by:

95% Confidence Interval for Target Analytes:

Range= Analyte Concentration +/- (analyte concentration x expanded uncertainty)

Expanded uncertainty values will change with time and as more and more data is accumulated. Updated of these values will be completed at least annually as required by laboratory procedure. It is the responsibility of each individual who utilizes these values to ensure that the appropriate current values are used for their data.

Updated copies for the expanded uncertainty values can be obtained from the QAM.

7.0 Data Processing

7.1 Collection

Accuracy and completeness of data records are essential in maintaining the quality of laboratory results. Black ink is used for all entries. All entries are signed and dated. Corrections are made with a single line through the error, and it must be initialed and dated.

Data records are maintained for all transfers and processing of each sample from the time the sample is received until the results are reported and the sample is disposed of. The records kept for receipt, log-in, and sample custody have been discussed in Sections 6.3 and 6.4. Preparation of standard solutions is documented in solutions manager programs. Each stock material and solution is assigned a unique number. Prepared solution identification numbers are recorded on the analysis data sheets. The standard solution preparation log contains entries regarding the source material, which includes:

- Compound name
- Purity
- Manufacturer and lot number
- Date received
- Concentration, if in solution form
- Solvent, when appropriate
- Date consumed or disposed of
- Expiration date
- Solution identification number

The solution preparation is documented by the following information:

- Compound identification
- Source material (by number)
- Assigned solution number
- Date prepared
- Quantity weighed out or measured by volume
- Final volume after preparation
- Solvent used
- Final concentration
- Expiration date
- Date disposed of

Data for inorganic (nonmetal) compound analyses are recorded in bound notebooks assigned to each test. The required information for each analysis includes, but is not limited to: the analytical procedure; any procedure changes required; internal sample number; raw analytical data; standard solutions used; preparation of reagents when appropriate; signature and date. If an instrument printout is obtained for the analyses, the printouts are signed, dated and reviewed.

For metals analysis, a digestion log is maintained in a separate notebook in the digestion lab. The digestion is documented by record of internal sample number, client ID, analysis required or method quantity and identity of spiking solution used, initial sample volume, final sample volume initials of technician and date.

Printouts of results are obtained for cold vapor, and ICP and ICP/MS analysis. For cold vapor work, a separate calculation page is prepared electronically to reference the analysis date, instrument identification, internal sample ID, concentration corrected final results, identity of QC or spiked

samples, percent recovery obtained and any comments. Final calculation of results for ICP are recorded directly on the data system printout. Each data set is filed in the metals raw data file cabinet.

Data for organics extractions are recorded in bound notebooks. All details regarding the extraction are recorded on this form. The data includes the following entries: extraction method, sample matrix, extraction date, surrogate spiking solution number and concentration, matrix spiking solution numbers and concentration, internal sample identification number, sample amount, quantity of surrogate and matrix spike added, final extract volume, extract storage location and signature of chemist.

Analytical data from GC, GC/MS and HPLC instruments is generated by the computer data system. Data outputs include identification of the sample, identifications of compounds retention times, and comparisons to standards. Outputs are in tabular form (retention times, areas, mass listings, etc.) and in graphic form (chromatograms, spectrum, etc.). Outputs are in a standard format specified for each analysis type. Data produced are compared to information concerning the sample history, sample preservation, QC data, etc., to judge the validity of the results.

Paper Record Entries

Only laboratory analysts, department supervisors and the laboratory director are authorized to make record entries in the laboratory notebooks and logbooks. All entries must be made in black ink. All entries must be made in accordance with the applicable method SOP. Any corrections that need to be made in any laboratory notebook/logbook must be made by crossing out, with a single line, the old entry, and incorporate the new entry next to it. The old entry must remain readable, and the persons initials and the date of the correction must appear in the logbook. Only laboratory analysts, department supervisors and the lab

director (or his designee) are authorized to make corrections in laboratory logbooks.

Electronic Data Entry

All electronic data must be stored in well functioning, well maintained and routinely backed up data systems. All electronic data entries must be performed using the software specified in the applicable method SOP. Only laboratory analysts, department supervisors and the lab director (or his designee), are authorized to make electronic data entries and/or corrections.

When electronic data entry corrections are made by authorized personnel, the person making the correction must log in with their individual, unique, computer account using their unique password. Upon completion of the correction, a hard copy must be produced, showing the individuals unique computer account identification on the pages that the correction took place. The updated packages must be included in the applicable data package. Writing over data files is not an acceptable corrective action.

The Software Quality Assurance Plan (SQAP) is under separate cover which describes policies and practices of GPL for the development, procurement, modification, maintenance and use of all computer software used for generation, compilation, reduction or reporting of laboratory results. The SQAP is available upon request.

7.2 Data Review and Verification

GPL performs data review and verification on all data packages generated. Information concerning the sample history, sample preparation, quality control data and other factors are used in determining the validity of the results. Each sample's history from sample receipt to reporting must be documented. Procedures implemented in this documentation are described in the SOPs designated for chain-of-custody and document control. Dated and signed entries by appropriate personnel on all worksheets and logbooks are required. The progress of the samples is traced through the laboratory by use of the sample tracking system. Finally, quality control information is judged against set criteria, the criteria used are dependent upon the methodology, the client's requirements, and the eventual use of the data. For environmental analysis performed under Contract Laboratory Program protocol, whether for EPA or commercial clients, all quality control parameters including method blanks, surrogate spikes, matrix spikes and duplicates, sample duplicates, laboratory control samples (QCs), field blanks, trip blanks and storage blanks must meet CLP acceptance criteria. Where applicable, sample flags or qualifier codes shall be used to qualify data.

All data receive a 100% review by either the supervisor or a second analyst of equal or higher experience and responsibility, in accordance with written procedures and guidelines. This review ensures that the following requirements have been appropriately met.

GC/MS Section

The analyst and GC/MS supervisor review data to ensure the laboratory provides the following where appropriate:

- Calculates the recoveries of surrogate spikes and verifies that criteria are not exceeded
- Verifies that there are no contaminants in associated blanks outside acceptable limits
- Compares samples and duplicates for precision in data results
- Verifies calibration performance for acceptability
- Reviews and verifies instrument tuning
- Reviews internal standard areas response for acceptability
- Verify that holding time criteria have been met
- Ensure surrogate recovery has been completed and acceptance limits are not exceeded
- Ensure that all analyte compounds have been properly recorded
- Ensure accuracy of calculations on compound quantities, and
- Ensure spectra are included and have been correctly interpreted

The reviewer examines the entire sample data file to ensure that all data transcription and documentation included meet customer requirements. The organic section manager performs a final technical review to verify that the completed package conforms to all quality control criteria.

Upon completion of review, the sample data files are forwarded to the reporting group for compilation of the entire data package and the project manager performs the final review.

All Other Sections

- Verify that holding time criteria have been met
- Calibration met or exceeded a correlation coefficient of 0.997 (metals and inorganics = .995). If an average calibration factor was used for calculations, the relative standard deviation of the average was $\leq 25\%$. Standards used in the calibration curve cover the expected concentration ranges of the samples including the reporting limit. The lowest calibration standard should be at least 5-10 times higher than the MDL for any given techniques. All sample results were extrapolated within the range of the standard curve. Initial and continuing calibration verification checks conforms to the acceptance criteria defined in the method requirements.
- Method blanks were processed with each analytical batch and were acceptable.
- Results of duplicate samples and matrix spike duplicates were within the laboratory or contract-established precision control limits.
- Matrix spike recovery was within acceptable control limits.
- Laboratory control samples were analyzed according to frequency specified in the SOP or contract and the results obtained were within control limits.
- For organic compound analyses, surrogate spike recovery was within control limits.
- For GC and HPLC analyses, the compounds identified fell within the method defined retention time window. This retention time window is established as outlined in Section 6.5 and per the individual methods.
- Calculations have been accurately performed.

Data for the analyses provide a complete audit trail. Data notebooks and data sheets correctly reference the analytical method, the standard solutions used, internal numbers, original data values, sample results in correct units, calculation

formula for all conversions, signature of the analyst, and date. Instrument printouts must identify the person responsible for the data generation and the date of the run. The supervisor or other data reviewer signs the data sheet to document approval. If the complete review was performed by someone other than the supervisor, a spot check is performed by the supervisor. The supervisor checks a minimum of 10% of the data. No data may be reported without supervisor approval evidenced by signature on the data page. The section manager performs a final technical review to verify that the completed package conforms to all quality control criteria.

A tabulation of results is prepared by the supervisor or analyst and placed in the central project file. The tabulation is transcribed into the report format by assigned report writers. The report and complete project file go to the section manager for final check. The section manager's review covers the following points:

- Transcriptions are checked for accuracy and use of appropriate units.
- QC data are reviewed to assure that internal specification and contract requirements have been met.
- Nonconformance reports, if any, are reviewed for completion of corrective action and impact upon results. Information contained in the nonconformance report may need to be included in the narrative report to the client.
- Results seem reasonable when compared to historical information associated with the site and results for other parameters tested at the same time.

Upon completion of review, the report folders are forwarded to the reporting group for compilation of the entire data package. The project manager performs the final review, as based upon client requirements. A copy of the signed report package is retained in the project file for archiving.

When a project requires 24-48 hour turn around time, the laboratory analyst will upload the results into the database, the supervisor will verify the results to ensure it was uploaded properly. The project manger then will release the results and submit them to the client. Upon the client's request, hard copy of the data will be assembled and released within 72 hours from the initial data release date. The laboratory director or his designee will compare and verify the close agreement between the initial submission and the final results. If any discrepancies are found, the laboratory director will initiate immediate corrective action.

According to the EPA Contract Laboratory Program Statements of Work under certain circumstances data must be qualified. Qualification of data may occur for a number of reasons including blank contamination, inability to accurately quantitate the analyte, confirmation of previous results and others. Qualification of data performed by CLP Protocol shall follow the data flagging procedures as stated in the Statement of Work. Additionally, EPA CLP deliverable packages may be validated after submission to the client, by an independent contractor, as part of the overall Contract Laboratory Program.

Data evaluation, sample flagging procedures and method blank evaluation procedures are usually discussed in each analytical method SOPs.

The procedures for reporting analytical results are detailed in SOP G.12 "Standard Operation Procedure for Reports Generation".

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7.3 Record Storage

Data notebooks, instrument printouts, instrument log, sample chain-of-custody logs, files, and contracts are retained for a period of 5 years. If contract requirements deviate from this procedure, the lab will retain the data for the duration specified in the contract, but not less than five years. All data reports that are EPA CLP data will be retained for 10 years. Original SOPs, current and outdated, are archived on-site storage location. In the event that the laboratory transfers ownership or goes out of business, all the laboratory records will be either maintained or transferred according to clients' instructions.

All laboratory reports are archived by the Report Generation in either on-site or off-site storage locations. Reports are submitted to the archives in archive boxes. Each box is numbered. A cross-index of documents by workorder is maintained for expedient retrieval of information.

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7.4 Transcription

Transcription is a potential source of error. Therefore, all transcriptions are checked by a second person.

Two types of transcriptions are most common:

- Transcription of a value from a chromatogram or instrument printout to a data sheet for further calculation of a result. This transfer is checked by the data reviewer's supervisor prior to release of results.
- Transcription in the report preparation and typing stage. This transfer is checked by the project manager.

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7.5 Data Reduction

Data reduction includes all processes, which change either the form of expression or quantity of data values. The size or dimensionality of the data set is reduced.

To validate all reduction operations, all calculations or manipulations of data are recorded in the data. A description of the formula used must be provided.

GPL uses stand alone computers, computer data systems, and microprocessor controlled instrumentation to reduce raw data to final form, such as:

- Lachat omnion data system
- Hewlett-Packard chemstation used in conjunction with Enviroquant operating on the laboratory's network system
- The "ADAMS" data reduction system for metals data
- Thermo Jarrell Ash data system

Calculation of results is performed by these systems based on standard curve responses and is printed with each sample response and/or summarized in tabular form at the end of each analysis set.

When data calculations using linear regression are performed, the correlation coefficient, slope, and y-intercept values are recorded in the data.

The procedure for correct use of significant figures and rounding of numbers is defined in a SOP. The rounding rules cited in the USEPA Handbook of Analytical Quality Control in Water and Waste Water Laboratories are followed for all manual rounding of numbers.

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8.0 Data Quality Assessment

8.1 Introduction – Definition of Terms

Accuracy

Accuracy is defined as the degree of agreement of a measurement, X with an accepted true value, T. Two types of accuracy check samples are used, Laboratory Control Samples (blank spike) and the matrix spike. The formula used to calculate accuracy for the Laboratory Control Sample is:

$$\text{Accuracy} = (A / B) \times 100$$

Where A = Concentration measured; and

B = Concentration spiked

which is the same formula as used for percent recovery. For calculating accuracy in matrix spike analysis, a correction for background concentration found in the unspiked sample must be performed. The formula is:

$$\text{Accuracy} = ((A - B) / C) \times 100$$

where A = Spiked concentration measured

B = Unspiked concentration measured

C = Concentration spiked

Precision

Precision is a measure of the mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Analysis

precision is assessed through comparison of duplicate samples or duplicate matrix spike samples. The term expressing precision is Relative Percent Difference (RPD) and is calculated as follows:

$$RPD = ((A_1 - A_2) / ((A_1 + A_2) / 2)) \times 100$$

where A_1 = Rep1; and

A_2 = Rep2

where Rep1 and Rep2 are replicate analyses of the same sample; and,

$$RPD = ((MS - MSD) / ((MS + MSD) / 2)) \times 100$$

where MS = the Matrix Spike sample result; and

MSD = the Matrix Spike Duplicate result

where the matrix spike and matrix spike duplicate analyses are performed upon the same sample.

Representativeness

Representativeness expresses the degree to which data accurately and precisely represent an environmental or process condition.

Field sampling operations have a major impact on data representativeness. Factors including site selection, sampling tools, equipment cleaning procedures, sample preservation, and many others must be considered. Similarly, laboratory operations could impact representativeness if there were day-to-day fluctuations.

Accuracy and precision results of the daily quality control samples provide a measure of representativeness associated with laboratory operations.

Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount expected under correct normal conditions. To maximize completeness of laboratory analysis, it is essential to obtain a sufficient quantity of each sample to provide for original and repeat analyses should the original analysis fail to meet acceptance criteria. Our goal for completeness is 95%.

Comparability

Comparability expresses the confidence to which one data set can be compared with another. This indicator of quality is enhanced at GPL by the following controls:

- Standardized EPA approved methodology for sample preservation, holding and analysis.
- Consistent reporting units for each parameter in similar matrices.
- EPA- or NIST-traceable standards, when available.
- Frequent analysis of USEPA QC samples.

Sensitivity

The term sensitivity is used broadly here to describe the contract method detection/reporting limits established to meet project specific DQOs; and not limited to the definition which describes the capability of a method or instrument to discriminate between measurement responses. Several limits have been established to describe sensitivity requirements (i.e., IDL, MDL, PQL, CRDL, CRQL, etc.). Normally, instrument detection limits (IDLs), and method detection limits (MDLs) reported are typically based upon a reagent water matrix or purified solid and ignore sample matrix interferences and the resulting effects

on the limits. The CRDLs and CRQLs published within CLP methodologies are contractually based levels.

- Method Detection Limit. The method detection limit (MDL) is 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. Method Detection Limits for IC analyses and Method 353.2 analysis are required semi-annually as method stated. All other analyses are determined annually and are performed for all new tests and when changes in equipment are initiated. The procedure is defined in 40 CFR Part 136, Appendix B (Federal Register).
 1. An estimate of the detection limit is established.
 2. A minimum of seven replicates of blank water are spiked at a level 1 to 5 times the estimated detection limit.
 3. The spiked samples are processed through every step of the analytical method.
 4. The standard deviation for the seven samples is multiplied by 3.143 (students t value at 99% confidence at N-1 degrees of freedom) to obtain the MDL.

The validity of the MDL study is verified per CFR requirements by comparing the mean value of the measured MDL spikes to the calculated MDL. The MDLs shall be preparatory method-specific, and include any clean-up methods used.

For DOD projects, an MDL verification check shall be performed immediately following an MDL study. DOD requires that the MDL check sample be spiked at Approximately 2 times the current reported MDL.

- Method Reporting Limit. The method reporting limit is established at a factor of five to ten times the MDL for the majority of target analytes, but no lower than three times the MDL for any target analyte.

- The method reporting limit is set at the lowest standard used for the initial calibration curve (or low-level calibration verification standard) or higher for each target analyte. The lowest standard or low-level calibration verification standard must be at least three times the MDL or greater.

- All target analyte values detected and reported below the method reporting limit must be flagged as an estimated quantity (i.e., J-flag).

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8.2 Methods for Attaining Quality Control Requirements

Sample Batching

The basic unit for application of laboratory quality control is the batch. Samples shall be prepared, analyzed, and reported in batches and be traceable to their respective batches. Batch sizes are normally limited to twenty field samples of a similar matrix but can exceed this by incorporating additional QC samples. Each batch shall be uniquely identified within the laboratory. Samples taken from the same site would normally be grouped together for batching purposes within the constraints imposed by the method holding times. However, laboratories may find it necessary to group multiple clients samples into a single batch. Under these circumstances, additional batch QC samples may be needed that evaluate the effect of the matrix from each site on method performance. Field QC samples, i.e., trip blanks, rinsates, etc., shall not knowingly be used for batch QC purposes.

- **Preparation Batch**

The preparation batch shall be defined as samples of the same or similar matrix that is prepared together by the same person, or group of people within the same time period or within limited continuous time periods, which follow the same method, using the same type of equipment and same lots of reagents. The laboratory shall have sufficient quantities of extraction/digestion labware to meet these requirements. Each preparation batch shall contain the requisite number and type of calibration solutions, blanks, quality control samples, and regular analytical samples as defined by the analytical method. The use of clean-up methods would be included as part of the preparation batch. All field and batch specific QC samples within the batch should be subjected to all preparatory and clean-up procedures employed.

- Analysis Sequence

The analysis sequence or instrument run sequence shall be defined as samples that are analyzed together within the same time period or in continuous time periods on one instrument under the control of one continuing calibration verification. Analyses sequences would be bracketed by the appropriate continuing calibration verification standards and other QC samples as defined by the analytical method. In general, if an instrument is not used for periods of time or shut down (e.g., overnight, etc.), then a new analysis sequence shall be initiated. Each analysis sequence shall contain the requisite number and type of calibration solutions, quality control samples, and regular analytical samples as defined by the analytical method.

Quality Control Samples

Data quality is evaluated by the performance of quality control sample analysis, including:

- Method Blanks
- Surrogate Spikes
- Matrix Spikes and Duplicates (MS, MSD)
- Sample Duplicate Analysis
- Laboratory Control Samples (LCS) / Blank Spike Samples (BKS)
- Calibration Check Samples
- Field Blank Samples
- Trip Blank Samples
- Storage Blank Samples

When the method of analysis contains definitive performance and acceptance criteria for quality control and calibration samples, the laboratory adheres to these criteria, unless different criteria are specified in the client's Quality Assurance Project Plan, or the client expressly demands that different (predefined) criteria are met.

When the method contains guidelines for quality control and calibration samples, and includes advisory acceptance criteria, the laboratory adheres to these criteria, unless different criteria are specified in the client's Quality Assurance Project Plan, or the client expressly demands that different (predefined) criteria be met.

When the method contains no specific or advisory acceptance criteria, or lacks detailed information concerning calibration and quality control, the laboratory will adopt QC criteria as listed in section 8000 of SW846. The particular types and frequency of QC samples processed with production samples are determined by the requirements of the client. Most common needs are those presented in the Contract Laboratory Program Statement of Work (CLP-SOW), EPA SW846, state requirements, project requirements, customer requirements, and those requirements specified in our SOPs.

Information obtained from the above listed quality control samples is used to assess the quality of the data generated and is useful in identifying problems in the sampling process, in the shipment of samples, in the storage of samples, in the analysis of samples and in identifying problems, in the analysis of the samples caused by the samples themselves. Specifically:

- Method Blanks

A blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix. A purified solid matrix (e.g., Ottawa sand or other purified solid) blank is used for solid matrices. In certain methods (i.e., pest/PCB & BNA determinations) purified sand is used where applicable.

A method blank is defined as a volume of deionized, distilled laboratory water, or in some cases a purified solid matrix, which is carried through the entire analytical process. Data obtained from these samples will indicate the absence or presence of sample contamination during the analytical process. The method blank will be performed at least once with each preparation batch, with a minimum of once per 20 samples.

The acceptance criteria for method blanks are addressed by the individual method SOP and/or the initial protocol. When no criteria are given, the laboratory will accept no target analytes at concentrations greater than the MDLs present in the blank.

- Surrogate Spikes

Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Samples are spiked using a surrogate to monitor the preparation and analytical process of the samples. If the surrogate material(s) are not recovered in sufficient quantity from the sample, the preparation and/or analysis of the sample is suspected. When surrogates are used they are spiked into all samples including blanks. The acceptance ranges for surrogate recoveries are specified by:

- a. The specific project plan, or
 - b. The method requirements, or
 - c. The GPL applicable SOP
- Matrix Spikes and Duplicates

In matrix/spike duplicate analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction/digestion and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at the regulatory standard level or the estimated or actual method quantification limit. When the concentration of the analyte in the sample is greater than 0.1%, no spike of the analyte is necessary. Matrix Spike and Matrix Spike Duplicate analysis are performed to evaluate the effect of the sample matrix upon the methodology and the precision of the method with the particular matrix.

If matrix spike compounds are not adequately recovered or vary in recovery between duplicates some measure of matrix interference is suspected. The acceptable ranges for MS/MSD recoveries are specified by:

- a. The specific project plan, or
- b. The method requirements, or
- c. The GPL applicable SOP

The MS/MSD will be performed at least once with each analytical batch, with a minimum of once per 20 samples. The laboratory will perform matrix spike and duplicate on specific samples as identified by clients field operations. Otherwise, the selected samples for matrix spike and duplicate will be rotated among client samples so that various matrix interference may be noted and/or addressed.

- Sample Duplicate Analysis

A duplicate sample is a sample prepared by dividing a sample into two or more separate aliquots. Duplicate samples are considered to be two replicates.

Sample duplicate analysis is used to assess sample preparation and analytical method precision. The precision acceptance criteria are specified by:

- a. The specific project plan, or
- b. The method requirements, or
- c. The GPL criteria of $\leq 20\%$ RPD

The duplicate (when no MSD applies) will be performed at least once with each analytical batch, with a minimum of once per 20 samples.

- Laboratory Control Samples (LCS)/Blank Spike Samples (BKS)

A blank, which has been spiked with the analyte(s) from an independent source in order to monitor the execution of the analytical method, is called a LCS/BKS.

The LCS/BKS is analyzed to assess general method performance by the ability of the laboratory to successfully recover the target analytes from a control matrix. For aqueous analyses use analyte-free reagent water. For soil analyses, a purified solid matrix (e.g., Ottawa sand, sodium sulfate, or other purified solid) would typically be used. However, due to the difficulty in obtaining a solid matrix which is metals-free, analyte-free reagent water is taken through the appropriate digestion procedures for metals analyses. The LCS/BKS is spiked with all single-component target analytes before it is carried through the preparation, cleanup and determinative procedures. A subset of the (single-component) target analytes containing the specific analytes of interest can be substituted for the full list of target analytes if specified in project-specific contracts or work plans. When multi-component target analytes are reported, a separate LCS/BKS may be necessary if specified by project documents. For Method 8082, the LCS/BKS must be spiked with at least one PCB (e.g., 1016/1260 mixture), or any project-specified PCBs.

When samples are not subjected to a separate preparatory procedure (i.e., purge and trap VOC analyses, or aqueous Hg analysis), the CCV may be used as the LCS/BKS, provided the CCV acceptance limits are used for evaluation. The spiking levels for the LCS/BKS would normally be set between the low and mid-level standards. The results of the LCS/BKS are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples. The laboratory also maintain control limits for these samples to assess the precision and bias of an analytical method. The precision may be evaluated by comparing the results of the LCS/BKS from batch to batch, or by duplicate LCSs/BKSs.

- Calibration Check Samples

A Calibration Check Sample is used as a method of determining the accuracy of an instruments calibration, by verifying the instrument response to analyte amount. The source of the material must be independent of the material used to calibrate the instrument and must be of a known quality and concentration.

- Field Blank Samples

Field blanks are aliquots of analyte-free water or solvents brought to the field in sealed containers and transported back to the laboratory with the sample containers. Field blank submission is solely upon the clients' discretion and/or requirements.

Analysis of field blank samples can furnish some measure of information into the possibility of contamination of samples occurring in the field during the sampling process.

- Trip Blank Samples

Trip blanks are not opened in the field. They are a check on sample contamination originating from sample transport, shipping and from site conditions.

- Storage Blank (Refrigerator Blank) Samples

Storage blank (refrigerator blank) sample analysis is used to determine if sample contamination may have occurred during the storage of the samples at our laboratory facility. The storage blank is analyzed every 2 weeks. The acceptance criteria should be less than one half of the method reporting limit (same as the method blank requirement). The VOA department is responsible for maintaining, monitoring and recording storage blank data.

Storage blanks shall be stored in the same manner as the client samples.

Blind Quality Control Samples

The QA unit, as well as outside regulatory agencies, periodically formulates blind samples for submission to the laboratory for analysis. Sample sets usually contain blanks, and replicates of known concentration. Analysis of the data produced from these samples are used to assess quality of data produced by the laboratory, particularly laboratory precision and accuracy.

Quality Control Limits

Precision and accuracy acceptance limits for CLP (Contract Laboratory Program) organic and inorganic analyses are contract-mandated. GPL also offers a variety of analytical services using EPA approved methodologies. The QC requirements for accuracy and precision are mandated by the method and of course the clients' needs and the regulatory authority under which the work is being performed. In the October 31, 1984 F.R., it is recommended that the laboratory periodically update these control limits based on historical data. It is GPL's policy to update control limits semi-annually after every twenty new sample data points are accumulated.

Warning and control limits are based upon the following formula:

$$\text{Upper Control Limit (UCL)} = X + 3s$$

$$\text{Upper Warning Limit (UWL)} = X + 2s$$

$$\text{Lower Warning Limit (LWL)} = X - 2s$$

$$\text{Lower Control Limit (LCL)} = X - 3s$$

where:

$$X = \text{Mean Percent Recovery}$$

$$S = \text{Standard Deviation}$$

All QC sample results are tabulated following analysis and compared to the contract-mandated, method-mandated, or client-mandated control limits for precision and accuracy. Out-of-control results are cause for immediate generation of a nonconformance report as described in Section 9.5 and possible re-extraction and/or re-analysis. No outlying data are ever released until the laboratory has verified that unacceptable results are attributable to the sample matrix. An analysis may be considered out of control whenever, as a minimum, any one of the following conditions is demonstrated by a control chart used to monitor that analysis.

- Any one point is outside of the control limits.
- Any three consecutive points are outside the warning limits.
- Any eight consecutive points are on the same side of the plotted mean.
- Any six consecutive points are such that each point is larger (or smaller) than its immediate predecessor.
- Any obvious cyclic pattern is seen in the data points.

QC data is recorded by analytical methodology employed and instrumentation used. For all CLP analyses, precision and accuracy data are required to be tabulated and reported on the MS/MSD Form.

Policy

The management and staff of GPL makes every effort to generate data of the highest quality possible and will continue to apply state-of-the-art analytical methodologies to ensure that our data continues to be of the best quality available anywhere.

GPL makes every attempt to produce and deliver analytical data, which has been demonstrated to meet contract-, method-, or client-required quality control acceptance criteria. Should anomalies occur in the processing and/or analysis of samples, which affect that objective, this is fully documented in the data and described in the report narrative. Also, when required, a statement of the estimated uncertainty of the test results will be documented in the report narrative. In cases where method variances occur, GPL will present the method or SOP to the client for evaluation and approval, prior to the initiation of the sample analysis. When any aspect of the environmental testing work or the results of the work do not conform to its own procedures or the agreed requirements of the specific project, the nonconforming testing SOP shall be followed

Laboratory Policy for Method Performance Determination

GPL consistently answers the need of its clients to provide specialized testing and develop additional analytical methods to meet specific project requirements. The method performance is determined by establishing the following parameters:

- A calibration curve of at least 5 points is developed.
- Method detection limit study is conducted, using at least seven replicate runs. The level spiked will be at least 10X the minimum peak detection level of instrument used.
- The resulting MDL must be approved by the lab director, the QA manager, and the general manager. No MDL will be approved, having a detection limit higher than the level spiked.
- Documentation of the MDL study must be filed with the QA manager and the department supervisor, including all approval signatures.
- A precision and accuracy (P&A) study must be developed and approved by the lab director, the QA manager and the general manager. No P&A study will be approved unless the RSD is $\leq 20\%$, and the accuracy is determined to be 70-130%.
Exceptions will be handled and approved on a case by case basis, depending on the method and with the approval of lab director, QA manager and general manager.
- The P&A study will be filed with the QA manager and the department supervisor, including all approval signatures.

All of the above bullets in the method performance policy must be completed and approved by the QA manager and the lab director/general manager, before a new method is used on any samples. Details on performing and approving MDL, IDL and P&A studies are discussed in SOP, "Determination of Accuracy – Precision, Instrument and Method Detection Limits".

8.3 Data Quality Objectives and Analytical Data Categories

In the planning of projects for the investigation of environmental pollution Data Quality Objectives (DQOs) are established. Data Quality Objectives are qualitative and quantitative statements, which specify the quality of data, required to support decisions during remedial response activities. DQOs are applicable to all data collection activities including those performed for preliminary assessments/site investigations, remedial investigations, feasibility studies, remedial design, and remedial actions. The level of quality and detail will vary depending upon the intended use of the data.

To assist in the interpretation of data, the superfund program has developed the following two descriptive data categories:

- Screening data with definitive confirmation;
- Definitive data.

These two data categories are associated with specific quality assurance and quality control elements, and may be generated using a wide range of analytical methods. The particular type of data to be generated depends on the qualitative and quantitative DQOs developed during application of the DQO process. The decision on the type of data to be collected should not be made prior to completion of the entire DQO process.

8.3.1 Screening Data

Screening data are generated by rapid, less precise methods of analysis with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, instead of elaborate extraction/digestion and cleanup. Screening data provide analyte identification and quantification, although the quantification may

be relatively imprecise. At least 10% of the screening data are confirmed using analytical methods and QA/QC procedures and criteria associated with definitive data. Screening data without associated and confirmation data are not considered to be data of known quality.

Screening Data QA/QC Elements

- Sample documentation (location, date and time collected, batch, etc.);
- Initial and continuing calibration;
- Documentation of detection limits;
- Analyte(s) identification;
- Analyte(s) quantification;
- Analytical error determination: An appropriate number of replicate aliquots, as specified in the QAPP, are taken from at least one thoroughly homogenized sample, the replicate aliquots are analyzed, and standard laboratory QC parameters (such as variance, mean, and coefficient of variation) are calculated and compared to method-specific performance requirements specified in the QAPP;
- Definitive confirmation: at least 10% of the screening data must be confirmed with definitive data. As a minimum, at least three screening samples reported above the action level (if any) and three screening samples reported below the action level (or as non-detects, ND) should be randomly selected from the appropriate group and confirmed.

8.3.2 Definitive Data

Definitive data are generated using rigorous analytical methods, such as approved EPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined.

Definitive Data QA/QC Elements

- Sample documentation (location, date and time collected, batch, etc);
- Initial and continuing calibration;
- Documentation of detection limits;
- Analyte(s) identification;
- Analyte(s) quantification;
- QC blanks (trip, method, rinsate);
- Matrix spike recoveries;

GPL typically provides definitive data as required by our clients. Project managers work with our clients in determining the data quality level required for each project. Project managers have the responsibility to ensure that the proper analytical methodology is employed and that the appropriate data deliverables package is generated.

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9.0 Corrective Action

9.1 Introduction

The QA is responsible for conducting inspections (audits) of the quality systems, data generation, and support systems of the laboratory. The purpose of the internal audit is to assist management in identifying and correcting deficiencies and to reinforce acceptable practices. This ensures that services meet the requirements of the Laboratory Quality Assurance Program Plan as well as the requirements of the client.

These inspections help to ensure that the policies of the laboratory requiring production of high quality data are being followed, including laboratory standard operating procedures, instrument procedures, sample preparation procedures and data review policies. If discrepancies are found, corrective action is taken. Two types of audits are in place: systems and performance audits. Additionally, there are routine data audits, independent audits, and audits for subcontracted services.

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9.2 System Audits

A systems audit is an inspection and review of an entire data-generation and support system. Quality-related activities are reviewed, assessed, and compared against the quality assurance program requirements for compliance. The audit includes the evaluation of personnel, facilities, standard operating procedures (SOPs), and records. Systems audits generally follow performance audits (usually by state or client auditors, required for certification and contract awards), and may be instituted as part of corrective action monitoring programs. These audits are performed quarterly.

Systems audits may also focus on a single area or aspect of laboratory operations. These inspections may consist of an in-process inspection of a particular analytical procedure, review of data books or logbooks for compliance to SOPs, or an inspection of the laboratory facility. These audits may be performed at any time at the discretion of the QA manager. Management may also direct the initiation of an audit for cause.

Systems audits are documented in the form of an audit report. The audit report describes any findings of the audit, recommendations to correct the finding and identifies the person or persons responsible for correction implementation. The original of the audit report is maintained in a chronological file while a copy of the document is circulated to the laboratory supervisor, laboratory director and the president. Once circulation is completed and all items are responded to, the audit report is filed by quality assurance. Follow-up audits will be performed to verify correction implementation. Audit reports are considered confidential documents and shall not be shown to or discussed with those outside the company without the expressed consent of the laboratory director and the quality assurance manager.

If deficiencies are observed during a performance audit, the quality assurance manager evaluates the audit report and initiates a follow-up systems audit, with emphasis on actions necessary to correct the deficiencies. A corrective action report is completed, detailing all remedial actions to be taken, and issued to the laboratory director and the laboratory manager for approval. If corrective action cannot be taken immediately, the anticipated date of action is provided. Once approved, the report is forwarded to the performance auditing agency or client.

Many of the objectives of a routine systems audit are similar to those a client or independent auditor would hope to accomplish during an on-site laboratory evaluation and data audit. These goals ensure that:

- Necessary quality control (including corrective action measurement) is being applied.
- Adequate facilities and equipment are available to perform the client's required scope-of-work.
- Personnel are qualified to perform the assigned tasks.
- Complete documentation is available, including sample chain-of-custody.
- Proper analytical methodology is being applied.
- Acceptable data handling techniques area being used.
- Corrective actions identified in any previous on-site visits have been implemented, and
- The laboratory management continues to demonstrate a commitment to quality.

In response to performance audits, any corrective actions taken are noted with reference to the auditor's deficiency report and the lab's standard operating procedures. Should a quantitative or qualitative error be noted in a data audit, a blind Proficiency testing / Proficiency evaluation sample may be entered into the system to test affected parameters. Additionally, laboratory proficiency tests may be scheduled if method performance is in question. Specifics of these two programs are outlined in the following sections.

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9.3 Performance Audits

A performance audit is a planned independent check of the operation of a measurement system with the purpose of obtaining a quantitative measure of the quality of the data generated. In practice, this involves analysis of standard reference samples or materials, which are certified as to their chemical composition or physical characteristics.

GPL participates in various Proficiency Testing / Proficiency Evaluation programs for each analyte or analyte group. The proficiency testing / Proficiency evaluation programs is evaluated to obtain or maintain approval to analyze an analyte or analyte group. GPL establish, maintain, and document the proficiency testing / Proficiency evaluation programs

The QA submits the Proficiency Testing / Proficiency Evaluation samples to the laboratory periodically. These samples provide a check on all operations performed in the lab, including sample holding, extraction, analysis, data validation, and reporting. The blind Proficiency Testing / Proficiency Evaluation samples are prepared from certified proficiency sample providers. Results reported by the laboratory are submitted to the laboratory managers. Unacceptable results require both investigation and documentation of corrective action by the laboratory manager.

If deficiencies are observed during an on-site assessment, the quality assurance unit will document the response to each deficiency noted on the on-site audit findings. Copies of the completed reports are filed by QA.

The procedures for proficiency testing /proficiency evaluation is defined in SOP "Proficiency Testing/Proficiency Evaluation Procedure".

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9.4 Audits of Subcontractors

Analysis performed by subcontractors must conform with GPL quality control requirements. Subcontractors must meet the requirements of the GPL quality assurance program or have in place an equivalent program. Also, where applicable, the laboratory will cooperate with any program requirements concerning the use of subcontractors.

The QA is authorized when necessary to evaluate the QA program of the subcontractor through review of the laboratory's written quality assurance program plan, the quality assurance project plan (where applicable), quality control SOPs, typical SOPs, and latest applicable USEPA performance evaluation study results. An on-site audit of the facility can be performed as deemed necessary by the QA manager.

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9.5 Nonconformance Event Corrective Action and Documentation

Documentation of analytical problems and corrective action taken is an essential part of the data record for each project. Identification, implementation, and monitoring of the actions that could have prevented the analytical problem provide a method for improving the quality of laboratory performance. A nonconformance report sheet has been designed to document laboratory problems, corrective actions, impact on analytical results, and preventive actions for the future (Section 9.5 page 3).

The nonconformance report must show complete background information about the event, including: date and time; analysis and phase; the client name; the sample identification number; and a description of the event that occurred. The report must further include: the corrective action taken; indication of the status of the system; an assessment of impact on analytical results; and recommendations for preventive action.

The nonconformance report should be initiated by the person experiencing or noticing the discrepancy and completed by the supervisor. For example, the initiator may provide the description of the event and corrective action taken; the supervisor addresses the impact and details future preventive action.

Copies of the completed reports should be distributed to the project manager, the laboratory director, and the original copy to the QA. The project manager should review the nonconformance report and place a copy of the report into the project file. If the event has caused any impact on the analytical results, the project manager will meet with the QA manager and communicate with the client personally.

The section manager should check that corrective action has been appropriate, confirm analytical impact, and ensure the implementation and monitoring of preventive action.

The QAM should review the nonconformance reports and file for follow-up action. On an as needed basis, a QA meeting is held with the QA manager, project managers, and laboratory management to evaluate corrective action and preventive action effectiveness. All effective preventive action will be documented for all appropriate laboratory sections. The laboratory managers and supervisors of each area will be responsible for any SOP revision to reflect these preventive actions.

Initial preventive action plans, which are evaluated as being ineffective, will be investigated to identify the origin of the problem and the effective preventive action. The supervisor of the area where the initial nonconformance occurred and section manager will participate in the investigation. Progress of the investigation and monitoring of the effectiveness of preventive action is documented by the supervisor and the information is filed by QA.

Nonconformance Report

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9.6 Preventive Actions

Preventive actions are a pro-active process to determine the areas where potential improvements can be made to reduce the likelihood of future problems or complaints. Preventive actions may originate with any member of the laboratory, from analyst to Laboratory Director, and should be brought to the attention of Quality Assurance for inclusion in the next management review meeting agenda. Preventive actions can result from needed changes as instrumentation or procedures become outdated, as newer technology is created to improve the laboratory's throughput and data quality, or as a result of trends identified during control charting or data analysis/review, etc. Once issues are identified for possible preventive actions and Quality Assurance is informed, the issues are added to the next management review meeting agenda maintained by Quality Assurance. The issue will be discussed in the management meeting, including possible benefits and costs, and an action plan is formulated. Following the reception of all required supporting information from the action plan, the Laboratory Director is responsible for determining the overall need for the proposed preventive action, for assigning personnel to perform the preventive action tasks, and for determining the time frame in which the duties will be completed. Preventive action documentation may be maintained as minutes in management review meetings or may require further documentation as determined by the Laboratory Director.

If the situation becomes an actual nonconformance or the results of nonconformance prior to the resolution of the preventive action; the preventive actions taken will be assistive but the issue is then addressed using the procedure for nonconformance and that procedure will take precedence over the preventive action activities.

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9.7 Management Review

The laboratory's management team shall periodically, and at least annually, will conduct a review of the laboratory's quality system and environmental testing activities to ensure the continuing suitability and effectiveness, and to introduce necessary changes or improvements.

Management Review meeting, attended by all laboratory management staff are conducted at regular intervals, to review the laboratory quality assurance system and to verify that the system is suitable and effective in meeting the needs of the clients, regulatory agencies, and certification programs. Quality topics addressed may include proficiency testing, nonconformance/ corrective action reports, internal audit findings and corrective actions and Laboratory health and safety; hazardous; and radioactive materials management functions.

The meetings may be used to determine solutions to quality and/or laboratory issues. Management review meetings may address topics of special concern, suggested by the QAM, section management or laboratory Director. Special topics may also include strategic planning, equipment needs, staff duties and needs, and reviews of upcoming projects to determine facility and resource requirements and suitability. The SOP "Management Review" gives further information regarding the procedures.

Findings from management reviews and the actions that arise from the reviews will be recorded. The management will ensure that those actions are carried out within an appropriate and agreed timescale. The recorded documents is maintained by QAM

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9.8 Confidentiality and Complaints

9.8.1 Client Confidentiality and Proprietary Rights

GPL Laboratories, LLLP makes every effort to maintain client confidentiality and proprietary rights in every phase of the analysis process from sample receipt through archive. Documents provided to the laboratory are held in strict confidence by project management staff. Analysts are prohibited from discussing client information with other parties outside the laboratory. On-site assessors from client's facilities are prohibited from examining any documentation in regard to information regarding other clients. Documents pertaining to quality assurance and analytical requirements are reviewed with appropriate managers and staff through the project specific meetings and the project protocol worksheet. Project related information provided by clients is securely archived. The transmittal of final results is specified in the project file and followed unless the project manager assigned to the client/project makes specific changes to the project.

9.8.2 Customer Complaints

If clients have complaints regarding data reporting, interpretation, methodology, or billing, the GPL policy on handling client complaints is addressed in the SOP "Customer Complaints". Every effort is made to resolve the problem to the client's satisfaction. In the event that there is a substantial disparity between the client's demands and GPL policy, the issue is submitted to the General Manger/Laboratory Director for resolution. All complaints associated with results or QC problems are examined closely and a QA/QC Corrective Action record may be complete.

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10.0 Implementation Requirement and Schedule

The QAPP becomes effective on the first day after approval by the QA manager and laboratory director. Any questions regarding implementation should be addressed to the laboratory quality assurance manager or the laboratory director.

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11.0 References

Regulations

40 CFR 136.3e Required containers, preservation techniques, and holding times

40CFR 136 Guidelines establishing test procedures for the analysis of pollutants under the Clean Water Act

40 CFR 136 Methods for Organic Chemical Analysis of Municipal and
Appendix A Industrial Wastewater

40 CFR 136 Definition and procedures for the Determination of the Method
Appendix B Detection Limit

40 CFR 136 Inductively Coupled Plasma – Atomic Emission
Appendix C Spectrophotometer Method for Trace Element
Analysis of Water and Wastes. Method 200.7

40 CFR 141 National Primary Drinking Water Regulations

40 CFR 143 National Secondary Drinking Water Regulations

Manuals

EPA 600/4-79-020 Method for Chemical Analysis of Water and Wastes (1983)

EPA 600/4-79-019 Handbook for Analytical Quality Control in Water and Wastewater
Laboratories (1979)

EPA 540/R-93-071	Data Quality Objectives Process for Superfund, September 1993
SW846	Test Methods for Evaluating Solid Wastes, Third Edition (1986)
Standard Methods	Standard Methods for the Examination of Water and Wastes, 17 th and 18 th Editions, American Public Health Association
EPA QA/R-5	EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, November 1999
DOD QSM	DOD Quality System Manual for Environmental Laboratories, Version 3 Final, January 2006
NELAC	National Environmental Laboratory Accreditation Conference 2003 NELAC Standard
DOE QSAS	Department of Energy Quality Systems for Analytical Services Version 2.2

APPENDIX A
RESUMES – KEY PERSONNEL
(available upon request)

APPENDIX B

CERTIFICATIONS STATUS AS OF PUBLICATION DATE OF QAPP

(most current and detailed certification status is available in the QA office upon request)

CERTIFICATIONS/VALIDATION/ACCREDITATION

Army Corps of Engineers (MRD)
Organic/Inorganic/Explosives (Current)

Navy CLEAN – NFESC Evaluated (Current)

Air Force AFCEE/IRPIMS Deliverables/ERPIMS Deliverables

USATHAMA/AEC IRDMIS Deliverables

Chemical Agent Degradation Analysis Capability
(Full List-USATHAMA/AEC Methods)

USDA Permit For Importing of Foreign
Soils For Chemical Analysis (Current)

State of Alabama (Current)
State of Arkansas (Current)
State of California (Current)
State of Connecticut (Current)
State of Delaware (Current)
State of Florida (Current)
State of Kansas (Current)
State of Maine (Current)
State of Maryland (Current)
State of Massachusetts (Current)
State of Nevada (Current)
State of New Jersey (Current)
State of New York (Current)
State of North Carolina (Current)
State of North Dakota (Current)
State of Pennsylvania (Current)
State of Rhode Island (Current)
State of Tennessee (Current)
State of Utah (Current)
Commonwealth of Virginia (Current)

US EPA CLP Laboratory (Routine Analytical Services 1980-1991)
US EPA CLP Laboratory (Special Analytical Services 1992-1994)
US EPA CLP Laboratory (Direct Analytical Services – 1996)

GPL Laboratories, LLLP
7210A Corporate Court
Frederick, MD 21703-8386
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APPENDIX C
EQUIPMENT LIST

APPENDIX D
METHOD DETECTION LIMITS/METHOD REPORTING LIMITS
(available upon request)

APPENDIX E

STANDARD OPERATING TABLES OF HOLDING TIMES AND PRESERVATION
REQUIREMENTS FOR ROUTINE METHODS

APPENDIX F
STANDARD OPERATING PROCEDURE MANUAL INDEX
(as of publication date of QAPP)