QUALITY MANUAL

FOR

Southeast Alaska Laboratories, LLC

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SECTION 1- Foreword

The use of a quality systems approach is critical to the effective operations of South East Alaska Laboratories. (SEAL) To help enhance consistency, efficiency and effectiveness of day-to-day work, the SEAL has adopted a quality system model.

SEAL's Quality System (QS) provides guidance to: (1) design and develop processes, products, and services related to SEAL's mission and to critical management and administrative support services, and (2) continually improve and strengthen product and service quality.

This Laboratory Operations Manual contains the required information to implement quality principles and practices throughout SEAL's laboratories. This manual serves as a key element in the implementation of a Quality Management System (QMS). The anticipated audience for this manual includes those in the public, regulated industry, counterpart agencies, SEAL scientists and other State of Alaska staff members who wish to understand SEAL QMS.

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Training is an essential element of QS implementation. For SEAL, training is necessary prior to achieving accountability for the policies and procedures described in this manual. For others, general information about quality systems may be found on the Internet at: American Society for Quality, "Learn About Quality" (www.asq.org)

SECTION 2.0 - Mission Statement

South East Alaska Laboratories (SEAL) is a certified, full accredited/licensed laboratory that tests organic consumable products, including but not limited to, those that contain cannabinoids, at or above the minimum levels required by the State of Alaska to ensure that any product tested and then sold is safe for public consumption.

Our Mission is to ensure customer satisfaction, accuracy of results and public safety in a timely and effective manner while adhering to all local, state, federal laws and established/accepted best international, peer reviewed laboratory practices.

2.1 Background

SEAL is a licensed organic testing facility located in Juneau Alaska. It is registered and licensed with the Marijuana control board at both the State of Alaska and City and Borough of Juneau as a Cannabinoid testing facility. These regulations are issued under the authority of the Alaska Statute; and other laws. In order to fulfill its regulatory mission, SEAL must ensure that its laboratory work products are of appropriate level of quality and reliability.

SEAL laboratories operates a permanent facility in Juneau Alaska.

The location is: 5450B Jenkins Dr., Juneau AK 99801

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SECTION 3 – INTRODUCTION AND SCOPE

The purpose of this Operations Manual is to outline the quality system for Southeast Alaska Laboratories Laboratory. The Operations Manual defines the policies, procedures, and documentation that assure analytical services continually meet a defined standard of quality that is designed to provide clients with data of known and documented quality and, where applicable, demonstrate regulatory compliance. This Operations Manual sets the standard under which all laboratory operations are performed including the laboratory's organization, objectives, and operating philosophy. This Quality Manual uses the references from TNI Standards, 2009.

Quality control terms are generally defined within the section that describes the activity. Glossary TNI Standards, 2009, Volume 1, Module 2, Module 4 and Module 5 .

3.1 Scope of Testing

The laboratory scope of analytical testing services includes those listed in the current scope of testing retained by the Laboratory Director.

The management system covers activities in the laboratory's Analytical Laboratory Section.

The fields of activities include:

1. Potency Testing
2. Residual Solvents
3. Microbiological Testing

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3.2 Acronyms

A list of acronyms used in this document and their definitions are:

AA– Accrediting Authority

ANSI – American National Standards Institute

ASQC – American Society for Quality Control

ASTM – American Society for Testing and Materials

Blk – Blank

°C – degrees Celsius

cal – calibration

CAR - Corrective Action Request

CAS – Chemical Abstract Service

CCV – Continuing calibration verification

COC – Chain of custody

DO – Dissolved oxygen

DOC – Demonstration of Capability

EPA – Environmental Protection Agency

g/L – grams per liter

GC/MS – gas chromatography/mass spectrometry

ICP-MS – inductively coupled plasma-mass spectrometry

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ICV – Initial calibration verification

ISO/IEC – International Organization for Standardization/International Electrochemical Commission

lb/in2 – pound per square inch

LCS – Laboratory control sample

LFB – Laboratory fortified blank

MDL – method detection limit

mg/Kg – milligrams per kilogram

mg/L – milligrams per liter

MS – matrix spike (using this for mass spec and matrix spike could cause confusion)

MSD – matrix spike duplicate

NELAC – National Environmental Laboratory Accreditation Conference

NELAP – National Environmental Laboratory Accreditation Program

NIST – National Institute of Standards and Technology

PAR - Preventive Action Request

PT – Proficiency Test(ing)

PTOB –Proficiency Testing Oversight Body

PTPA – Proficiency Testing Provider Accreditor

QA – Quality Assurance

QC – Quality Control QM – Quality Manual

QAM – Quality Assurance Manager RL – Reporting level

RPD – Relative percent difference RSD – Relative standard deviation

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SOPs – Standard operating procedures spk -spike

std – standard

TNI - The NELAC institute

ug/L – micrograms per liter

UV – Ultraviolet

VOC – Volatile organic compound

WET – Whole effluent toxicity

SECTION 4 – ORGANIZATIONAL ROLES AND RESPONSIBILITIES

POLICY

The laboratory is a legally identifiable organization. Through application of the policies and procedures outlined in this chapter, the laboratory assures that it is impartial and that personnel are free from undue commercial, financial, or other undue pressures that might influence their technical judgment. The laboratory is responsible for carrying out testing activities that meet the requirements of the TNI Standard and that meet the needs of the client.

The laboratory is a commercial laboratory which operates in Juneau, Alaska. See Appendices for Organizational Chart.

4.1 Laboratory Organizational Structure

4.1.1 Overview

This section presents information regarding the basic roles and responsibilities for SEAL staff. The duties described below are not all-inclusive.

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4.1.1.1- Office of Laboratory Director

Establishes and manages a program to maintain quality and integrity for all Center laboratory studies and the processing of regulatory samples.

Responsibilities of the Office of the Center Director include:

Maintain a QA program to help ensure the quality and integrity of data produced by SEAL.

Provide the resources and commitment to implement the LQM.

Develop overall SEAL scientific policy and laboratory programs to support policy.

Review reports of SEAL QA activities which include internal audit reports, corrective/preventive actions, management reviews, and continual improvement efforts.

Maintain overall responsibility to ensure that deviations and complaints are addressed.

4.1.1.2- Laboratory Director:

The Laboratory Director shall audit the lab periodically and maintain written and properly signed records of each periodic inspection to ensure that quality standards are being met. The scope and frequency of these activities will be determined in consultation with senior management.

Periodically submit to the consultant written status reports, noting any problems and the corrective actions taken.

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Maintain and update the SEAL Laboratory Quality Manual.

Maintain copies of all pertinent documentation and records.

Ensure that a SEAL/State of Alaska Tracking System number has been assigned to each test upon approval.

Audit laboratories in accordance with a predetermined schedule.

Generate QA status reports, which include internal audit reports, corrective/preventive actions, and continual improvement efforts.

4.1.1.3- Lead Scientist

For each laboratory test, a scientist or other professional of appropriate education, training, and experience, or combination thereof, shall be identified as the lead scientist. The lead scientist has overall responsibility for the technical conduct of the test, as well as for the interpretation, analysis, documentation, and reporting of results. The lead scientist shall be responsible for the following:

Developing the testing protocol per State of Alaska Statue.

Ensuring that changes (amendments) to the project/protocol are documented and approved.

Maintaining pertinent documentation regarding all project/protocol activities.

Communicating on a regular basis with contractors who provide project/protocol support.

Maintaining results of relevant analyses as part of the raw data for the protocol.

Documenting and addressing circumstances that may affect the quality and integrity of the laboratory test.

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Preparing a final report in accordance with SEAL policies and State of Alaska Statute.

Ensuring that raw data, documentation, protocols, specimens, and final reports (if necessary) are archived and/or properly disposed of at the completion of a test.

Ensuring that the test is conducted in a safe manner and in accordance with the safety plan, safety procedures, and safety requirements.

Conducting the test in accordance with other relevant regulations.

4.1.1.4-Research Facility Management

For each laboratory test, management shall:

Designate a lead scientist before the test is initiated.

Replace the lead scientist promptly if it becomes necessary to do so during the conduct of a test.

Ensure that projects/protocols are properly amended to reflect changes in responsible personnel.

Ensure that personnel clearly understand the functions they are to perform.

4.1.1.5-The Laboratory Manager

Serves as project oversight on all contracts whose obligations are to provide all aspects of building maintenance for systems and equipment relating to laboratory operations. This includes, but is not limited to,

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room and fume hood airflow, cold box and structural maintenance (walls, ceilings, floors), water, gas, and electrical services.

Provides guidance for the maintenance of all SEAL-installed equipment. This includes offering technical assistance, making recommendations, and preparing requisitions necessary to contract for the required services.

Provides assistance and recommendations on space utilization and assists in relocations.

Receives and distributes laboratory and other supplies ordered for use.

Operates and maintains the FDA Scientific Supply Store.

Arranges for the shipment of appropriately packaged items, with the possible exceptions of radioactive materials and select agents and toxins, to their destination via GSA contract-approved transportation company or courier service.

Delivers gas cylinders and picks up empty gas cylinders upon request of laboratory personnel.

Provides for pick up, processing, and transportation of hazardous waste to a centralized location for disposal.

Receives and stores equipment and supplies for use in the limited access areas at designated facilities.

Ensures that all laboratory work conducted at SEAL is performed in compliance with all applicable occupational safety and health and environmental management regulations, standards and SEAL policies

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4.1.1.6- Board of Directors

The Board of Directors are investors who have purchased 1 or more share(s) of SEAL. The Board will meet bi-annually to receive updates on the financial outlook of SEAL as well as a general overview of the operations. Each member will have the same number of votes as the number of shares that he/she owns. The Board will have binding decision making authority on the following decisions: Hiring and salary of the consultant, further expansion of SEAL, distribution of dividends, and purchase of real property excluding equipment necessary for testing of product.

4.1.1.7-Consultant

The consultant is hired by the Board of Directors (BOD). He/she must have a scientific background and have a firm understanding of chemistry and testing. A bachelor's degree in chemistry or a related field is highly recommended. The consultant will be the liaison between the BOD and the CEO, lab director/lead scientist. He/she will be expected to report, in clear non-scientific language, the financial and scientific output of the lab. He/she will also be expected to facilitate conversations with local and state government agencies for purposes of licensing, testing and compliance. Additionally he/she will be responsible for the evaluation/performance review of the CEO. The consultant will be the only personnel over which the BOD has authority.

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POLICY

The organization and management structure of the laboratory, its place in the parent organization, and the relationships between management, technical operations, support services, and the quality

management system is defined through the aid of an organizational chart.

PROCEDURE

Senior management keeps the most current organizational chart on file. An organizational chart is available with this manual as a reference record and is considered the official record on the date it is marked. (See Appendix A)

4.1.2 Management and Personnel

Management and personnel are free from any undue internal and external commercial, financial and other pressures that may adversely affect the quality of their work. The integrity of test results is the responsibility of all personnel. Management ensures that employees are never instructed or forced to alter or falsify data.

POLICY

The laboratory avoids involvement in any activities that would diminish confidence in its competence, impartiality, judgment, or operational integrity.

PROCEDURE

To ensure confidence in laboratory operations, a formal quality assurance program is implemented. Technical competence is ensured through check sample programs. Impartiality is assessed through audits and approvals. Judgment is ensured through the hiring of qualified personnel and by continuously refining, upgrading, and improving personnel skills. Operational integrity is reviewed by management on a regular basis at management review meetings to ensure continued suitability and effectiveness of laboratory policies and procedures. Any problems are acted on immediately through corrective action procedures.

. 4.1.3 Potential Conflicts of Interest

The laboratory reports to the Technical Manager, who ensures there exists no adverse influence against the laboratory’s ability to operate in an independent manner and it is further assured by independent

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checks and/or audits from the Quality Manager and implementation of Section 16 of the Quality Manual.

The following list provides some guidelines on how employees avoid conflict of interest situations. Employees shall not:

1. Falsify records, prepare fraudulent reports, or make false claims
2. Seek or use privileged or confidential company information, or data from any customer, for any purpose beyond the scope of employment
3. Conduct non-laboratory business on laboratory time, or use company facilities or equipment to conduct outside interests in business, unless prior approval has been obtained
4. Solicit business on their own behalf (rather than the laboratory) from a customer Be employed by, or affiliated with, organizations whose products or services compete with laboratory products or services
5. Have employment that negatively affects or interferes with their performance of laboratory duties
6. Compete with the laboratory in the purchase, sale, or leasing of property or goods
7. Allow association, family, or friends to influence business decisions to their benefit - decisions must be made on a strictly business basis, always in the best interest of the laboratory
8. Make any decision that provides gains or benefits to the employee and/or others
9. Have personal financial dealings with an individual or company that does business with the laboratory which might influence decisions made on the laboratory's behalf

Firm adherence to this code of values forms the foundation of our credibility. Personnel involved in dishonest activities are subject to a range of disciplinary action including dismissal.

4.1.4 Responsibility and Authority

Management includes the Laboratory Director, Technical Manager, and the Quality Manager.

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POLICY

Management has overall responsibility for the technical operations and authority needed to generate the required quality of laboratory operations.

POLICY

Management's commitment to quality and to the Quality System is stated in the Quality Policy, which is upheld through the application of related policies and procedures.

Management ensures technical competence of personnel operating equipment, performing tests, evaluating results, or signing reports, and limits authority to perform laboratory functions to those appropriately trained and/or supervised.

PROCEDURE

The assignment of responsibilities, authorities, and interrelationships of the personnel who manage, perform, or verify work affecting the quality of tests is documented in section 17.

Management bears specific responsibility for maintenance of the Quality System. This includes defining roles and responsibilities to personnel, approving documents, providing required training, providing a procedure for confidential reporting of data integrity issues, and periodically reviewing data, procedures, and documentation.

Management ensures that audit findings and corrective actions are completed within required time frames.

Designated alternates are appointed by management during the absence of the Laboratory Manager, Technical Manager or the Quality Manager, and always if the absence is more than 15 days. If the absence of the Technical Manager exceeds thirty-five (35) consecutive calendar days, the primary accreditation body will be notified in writing.

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Management is responsible for defining the minimal level of education, qualifications, experience, and skills necessary for all positions (within the scope of state and federal law) in the laboratory and assuring that technical staff have demonstrated capabilities in their tasks.

Training is kept up to date as described in Section 17.4 by periodic review of training records and through employee performance review.

Only the Laboratory Director can approve departures from the organizational and management policies in this manual.

Only the Quality Manager can approve departures from quality management system procedures.

Only the consultant can approve departures from LLC SOP's or Laboratory Director directives.

Departures from test methods or technical standard operating procedures (SOPs) can only be approved by the Technical Manager.

SECTION 5 – QUALITY SYSTEM

The laboratory's Quality System is documented in this Quality Manual and associated documents and records serve as the quality plan for the laboratory. Other documents and records include:

1. Standard operating procedures
2. Quality control plans in test methods
3. Proposals
4. Project management schemes

The purpose of our Quality System is to ensure that all services and products satisfy the customer's requirements and have been designed, manufactured, and delivered under controlled conditions.

POLICY

The Quality System is established, implemented, and maintained by management. It is applicable to all the fields of testing and activities in which the laboratory is involved and undertakes. All policies, systems,

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programs, procedures and instructions are documented to the extent necessary to enable the laboratory to assure the quality of results generated. These documents are communicated to, understood by, available to, and implemented by the appropriate personnel.

PROCEDURE

The effectiveness of the Quality System is assessed in several ways:

1. By a program of planned internal audits, covering all aspects of the operation of the quality system
2. By regular management reviews of the suitability and effectiveness of the quality system
3. By analysis of potential and actual problems as shown by customer complaints and supplier and subcontractor assessments
4. By other methods approved from time to time by the Laboratory Manager or Quality Manager

5.1 Quality Policy

POLICY

The policies and objectives for laboratory operations are documented in this Quality Manual. The overall objectives are set out in the Quality

Policy Statement and reviewed during management review. The Quality Policy Statement is issued under the authority of the Laboratory Manager on the effective date.

Objectives

To ensure accurate, timely analytical and technical services, and to continuously meet or exceed the stated or implied expectations of our customers through day-to-day interactions.

A. Management commitment to good professional practice and quality of services provided to the customer: tests and calibrations are always carried out in accordance with stated standardized methods and customers' requirements. Requests to perform tests that may jeopardize an objective result or have a low validity are rejected.

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B. Standards of service include:

1. Customer Satisfaction
2. Accuracy of the Tests
3. Timely testing a results.

5.2 Additional objectives:

1. To establish the level of the laboratory's performance
2. To make test method changes to improve performance
3. To participate in proficiency testing or quality evaluation programs with peer laboratories
4. To ensure that all personnel are trained to a level of familiarity with the quality system appropriate to the individual's degree of responsibility
5. To improve and validate laboratory methodologies by participation in method validation collaborative tests To establish and report on quality savings

Quality Policy Statement

Southeast Alaska Laboratories Laboratory's management and employees are committed to consistently provide our customers with data of known and documented quality that meets their requirements. Our management is committed to continually improve the effectiveness of our quality system by meeting our objectives and complying with the requirements of the TNI Standard. Our policy is to use good professional practices, to maintain an effective quality system, to uphold the highest quality of service, and to maintain the confidentiality of our customers. Every Southeast Alaska Laboratories employee is committed to following all policies and procedures, maintaining demonstrated capability in their analytical areas and adhering to our standards for data integrity, ethics, and quality.

5.3 Quality Manual

POLICY

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Management ensures that the laboratory's policies and objectives for quality are documented by reference or by inclusion in the Quality Manual, and that the Quality Manual is communicated to, understood by, and implemented by all personnel concerned.

POLICY

Where the Quality Manual documents laboratory requirements, a separate SOP or policy is not required. C. D. E.

Excellence in the workplace is promoted by providing all employees with the knowledge, training, and tools necessary to allow for the completion of accurate and timely work.

Purpose of management system related to quality: to manage our business by meeting the needs of our customers.

Personnel: familiarize them with quality documentation and implement the policies and procedures in their work.

Management is committed to complying with TNI Standards and to continually improve the effectiveness of the management system: The objective of this Quality Manual is to document the compliant policies and associated procedures that are integrated into our daily activities. Continual improvements are established, implemented, and locked into the quality system.

PROCEDURE

All employees sign a form, kept with the Quality Manual, that states that they have read and understood the Quality Manual, including the quality policy. The Laboratory Director approves the Quality Manual and the Quality Manager keeps the signature of this approval on file.

The Quality Manual is maintained current and up-to-date by the Quality Manager.

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SECTION 6 – DOCUMENT MANAGEMENT

This Section describes procedures for document management, which includes controlling, distributing, reviewing, and accepting modifications. The purpose of document management is to preclude the use of invalid and/or obsolete documents.

The laboratory manages three types of documents, 1) controlled, 2) approved, and 3) obsolete.

1. A CONTROLLED DOCUMENT is one that is uniquely identified, issued, tracked, and kept current as part of the quality system. Controlled documents may be internal documents or external documents.
2. APPROVED means reviewed, and either signed and dated, or acknowledged in writing or secure electronic means by the issuing authority.
3. OBSOLETE DOCUMENTS are documents that have been superseded by more recent versions.

POLICY

All documents that affect the quality of laboratory data are controlled appropriate to the scope and depth required. Document means any information or instructions including policy statements, procedures, specifications, regulations, standards, other normative documents, manuals, test and/or calibration methods, charts, textbooks, posters, notices, memoranda, software, drawings and plans. These may be in various media, whether hard copy, electronic, digital, analog, photographic or written.

The documents to be controlled include:

1. Quality Manual
2. Standard Operating Procedures
3. Test methods
4. Forms

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6.1 Controlled Documents

POLICY

All documents issued to personnel in the laboratory as part of the quality management system are prepared and reviewed for use by authorized personnel prior to issue (i.e., reviewed by personnel knowledgeable in the documented activity). Reviewed documents are approved for use by management.

PROCEDURE

Documents are reviewed to ensure their contents are suitable and in compliance with the current quality systems requirements, and accurately describe current operations. The Quality Manager reviews the Quality Manual annually. Written procedures and documents are reviewed on a biennial basis by personnel knowledgeable in the documented activity.

Authorized editions of appropriate documents are available in the Quality Manager's Office and on the public drive where operations essential to the effective functioning of the laboratory are performed. Note:

Controlled internal documents are uniquely identified with 1) date of issue, 2) revision

6.2 Obsolete Documents

POLICY

All invalid or obsolete documents are promptly removed from the public drive and moved to identification, 3) page number, 4) the total number of pages (or a mark to indicate the end of the document), and 5) the signatures of the issuing authority (i.e. management). The "Controlled Document" colored ink stamp at the bottom of each page indicates controlled hard copies.

A master list of controlled internal documents is maintained that includes distribution, location, and revision history. A master list of

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controlled external documents is also maintained that includes title, author, copyright date, and date of publication, and location. The controlled document list is maintained by the Quality Manager. The controlled document list is updated after the documents have been reviewed.

POLICY

A master list identifying the revision history and distribution of documents in the quality management system is readily available in order to preclude the use of invalid and/or obsolete documents.

PROCEDURE

This procedure is done after documents have been revised and approved (on the "Date Effective").

6.2.1 Document Changes to Controlled Documents

Revisions to documents and procedures are accompanied by a form which details the appropriate additions, removals, and replacements.

6.2.1.1 Paper Document Changes

POLICY

Document changes require the same review and approval as the original review and approval.

POLICY

The document management process does not allow for handwritten modifications to documents.

PROCEDURE

All document changes must be approved. The designated personnel have access to pertinent background information upon which to base their approval.

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6.2.1.2 Electronic Document Changes

PROCEDURE

Electronic copies of documents are maintained under the authority of the Quality Manager on a network drive. Public access to electronic copies is on the server saved as a PDF file. A private drive on the server will be used to assure against unintended use. The documents are labeled, "OBSOLETE" and dated.

PROCEDURE

Obsolete documents retained for legal use or historical knowledge A reservation are appropriately marked and retained.

Obsolete master copies are clearly marked obsolete and archived for at least five years. All other draft copies and obsolete versions are destroyed to prevent inadvertent use.

6.3 Standard Operating Procedures

STANDARD OPERATING PROCEDURES (SOPs) are used to ensure consistency of application of common procedures. SOPs are written procedures that describe in detail how to accurately reproduce laboratory processes, and are of two types: 1) test method SOPs, which have specifically required details, and 2) general use SOPs which document the more general organizational procedures.

SOPs do not have to be formal documents with predefined section headings and contents. They can be less formal descriptions of procedures described in the Quality Manual or other documents.

POLICY

Procedures are peer-reviewed and authorized by management. Copies of all SOPs are accessible to all personnel. The issued date (see header) is the date the document was issued to the user for review. The effective date (see footer) is the date the document was effectively in place and followed.

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PROCEDURE

Each SOP indicates the effective date, the revision number, and the signature(s) of management.

6.3.1 Test Method SOPs

Test methods and standard operating procedures are controlled documents with limited distribution. They are kept in three-ring binders in the Quality Manager's Office labelled "Controlled Documents." The Table of Contents includes all current and obsolete procedures with the following information:

1. The code and title of each procedure
2. The revision number of each procedure
3. The effective date of each procedure
4. Review date – The date each procedure was last reviewed
5. The status of each procedure (current or dormant)

6.3.1.1- Procedure Codes

Unique codes are assigned for each procedure.

F – Forms used in the laboratory

Log – Logbook

QSP – Quality System Procedure

SOP - Standard Operating Procedure

SP – Spreadsheets

POLICY

The laboratory has SOPs for all test methods within its scope, and for procedures that are part of the Quality System that accurately reflect how the analytical process is performed. Where equipment manuals or published methods accurately reflect laboratory procedures in detail, a separate SOP is not required.

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POLICY

Any deviation from a test method is documented, including both a description of the change made and a technical justification. The deviation from a test method is reported to the client.

PROCEDURE

Each Test Method SOP is written with the following headings (as applicable):

- Identification of the method
- Applicable matrix or matrices
- Limits of detection and quantitation
- Scope and Application
- Summary of method
- Definitions
- Interferences
- Safety
- Equipment and supplies
- Reagents and standards
- Sample collection, preservation, shipment and storage
- Quality control
- Calibration and standardization
- Procedure
- Data analysis and calculations
- Method performance
- Pollution prevention
- Data assessment and acceptance criteria for quality control measures
- Corrective actions for out-of-control data
- Contingencies for handling out-of-control or unacceptable data
- Waste management
- References
- Tables, diagrams, flowcharts and validation data

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SECTION 7 – REVIEW OF REQUESTS, TENDERS AND CONTRACTS

Contract Review is a systematic activity carried out by the supplier, before signing the contract to ensure that requirements for quality are adequately defined, free of ambiguity, documented, and can be realized.

POLICY

The review of all new work assures that oversight is provided so that requirements are clearly defined, the laboratory has adequate resources and capability, and the test method is applicable to the customer's needs. This process assures that all work will be given adequate attention without shortcuts that may compromise data quality.

Contracts for new work may be formal bids, signed documents, verbal, or electronic.

7.1 Procedure for the Review of Work Requests

The Laboratory Director or designee determines if the laboratory has the necessary accreditations, resources, including schedule, equipment, deliverables, and personnel to meet the work request.

Upon client inquiry, the following information is obtained:

- Client Contact Information (Contact person, phone/fax/ email, Address)
- Objective/goals/ required information
- Requested analysis: Qualitative/semi-quantitative, limit of detection; Quantitative, range of concentration
- Costs (Expected costs, cost limits)
- Date of completion/ schedule
- Date of intermediate results/reports
- Deadline for final results/report
- Sampling Requirements (Client, Laboratory, other)
- Date of sampling, source/producer, responsible person, number of samples
- Description of sample(s)

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- Identification, special care for storage/transport/stabilization, pretreatment/preconditioning
- Methodology
- Description of methods used for sampling, sample preparation, measurement
- Standard method
- Generic method New/adapted method Validation required for method

The Laboratory Director or designee informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to the complete the work satisfactorily.

The client is informed of any deviation from the contract including the test method or sample handling processes. All differences between the request and the final contract are resolved and recorded before any work begins. It is necessary that the contract be acceptable to both the laboratory and the client.

7.2 Quotations for routine requests

Quotations for routine requests can be quickly provided through the standard price list. Quotations for non-routine requests take more time to cost out and are formally issued as a service quotation document. This is typically faxed or emailed to the client. Each new service quotation is assigned a unique number.

The review process is repeated when there are amendments to the original contract by the client. The participating personnel are given copies of the amendments.

7.2.1 Documentation of Review

Records are maintained for every contract or work request, when appropriate. This includes pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract.

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7.2.2 Acceptance of a Contract

A. The client sends back the service quotation with their signature within the acceptance period and/or a purchase order referring to the service quotation.
B. When a client submits samples without written authorization and the submission form requests, the previously discussed services and a confirmation work order are sent to the client informing them that the work will be performed as proposed.
C. When a client submits samples without written authorization and the submission form requests services that are different from the original service quotation, the client is contacted to clarify the required testing and to obtain written authorization to proceed.
D. When a client submits samples without any prior discussion i.e. required testing and pricing, the client is contacted, requirements are reviewed, a service quotation is issued to clarify request and provide pricing.

7.2.3 Contract Amendment

Amendments to a contract are reviewed in the same manner as previously outlined. If a contract needs to be amended after the work has commenced, the same contract review process is repeated and any amendments are communicated to all affected personnel.

SECTION 8 – SUBCONTRACTING OF TESTS

A SUBCONTRACT LABORATORY is defined as a laboratory external to this laboratory, or at a different location than the address indicated on the front cover of this manual, that performs analyses for this laboratory.

POLICY

Subcontracting is not allowed under Alaska Statue. Should subcontracting become necessary the laboratory will contact the State of Alaska for permission before proceeding.

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Should the State of Alaska give written permission for subcontracting due to work load, large contracts, contracts requiring extra technical expertise, or test requested is not an approved test is subcontracted to a technically competent laboratory the following procedure will be used.

Procedure

When subcontracting analytical services, the laboratory assures work requiring accreditation is placed with an appropriately accredited laboratory or one that meets applicable statutory and regulatory requirements for performing the tests.

A list of subcontractors is maintained.

The subcontracted laboratory demonstrates technical competence by possession or receipt of one or more of the following: Recognized technical accreditation by TNI or an Audit of the subcontractor's quality management system by TNI. A copy of the certificate and analyte list for subcontractors may be maintained as evidence of compliance.

The laboratory notifies the client of the intent to subcontract the work in writing.

When possible, the laboratory gains the approval of the client to subcontract their work prior to implementation, preferably in writing.

The laboratory performing the subcontracted work is identified in the final report. The laboratory assumes responsibility to the client for the subcontractor's work, except in the case where a client or a regulating authority specified which subcontractor is to be used.

It is the responsibility of the Quality Manager to assess and approve the competence level of subcontractor laboratories.

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SECTION 9 – PURCHASING SERVICES AND SUPPLIES

POLICY

Only services and supplies of the required quality are used. These quality requirements are detailed in the laboratory SOPs and will identify the appropriate minimum specifications when necessary.

PROCEDURE

Laboratory director reviews and approves the supplier of services and supplies and approves technical content of Purchase Requisition prior to ordering.

The criteria for evaluation may include, but is not limited to the following:

- References
- Accreditation
- Formal recognition

The records are maintained by the Purchasing department. A list of approved suppliers is kept in the Purchasing department.

POLICY

Purchase Requisitions are used for purchasing, receiving and storage of supplies.

PROCEDURE

Packing slips are checked against package content labels and matched with the Purchase Requisition if accepted. Once accepted, the packing slip is dated and initialed as evidence of compliance. Certificates of analysis (COA) are maintained on file in the Quality Manager's/laboratory director's office after the COA is checked to ensure the received item meets minimum specifications.

Chemicals are purchased with manufacturer's certificates where possible.

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If the chemical does not come with COA, the laboratory verifies the quality of the standards by comparing the new batch of standards to the old. The laboratory follows the manufacturer's recommendations on storage and shelf life.

Reagents are generally purchased from manufacturers who have a quality management system based on ISO 9001 or other required certifications. The grade of any reagent used (including water) is stated in the method together with guidance on any particular precautions to be observed in its preparation or use.

Where no independent assurance of the quality of procured goods or services is available or the supplier's evidence is insufficient, the laboratory ensures that purchased goods and services comply with specified requirements. Where possible and practical the laboratory ensures that goods are inspected, calibrated, or are otherwise in compliance with any standard specification relevant to the calibrations or tests concerned.

Consumable materials are stored according to the appropriate test method, SOP, work instruction, or manufacturer's instructions.

SECTION 10 – SERVICE TO THE CLIENT

Customer requests are clarified for the customers or their representatives. The customer or their representative will be afforded the right to monitor the performance of the laboratory in relation to the work performed, provided that the laboratory ensures confidentiality to other customers.

10.1 Client Confidentiality

POLICY

It is the policy of our laboratory to protect the confidential information and proprietary rights of our customer including the electronic storage and transmission of results.

PROCEDURE

All employees must sign an Employee Confidentiality Agreement. The signed agreement is retained in each employee's lab personnel file.

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Test results are only released to the customer. Release to someone other than the customer requires the express permission of the customer, except when the situation contravenes State or Federal Legislation and the results must be provided to the appropriate agency. The release of test results to anyone other than the customer requires the permission of the Laboratory Director or delegated signature of authority. Prior to release, all Laboratory reports are reviewed for accuracy by the Laboratory Director or designee.

Service to the customer includes:

- Affording the customer or the customer's representative

reasonable access to relevant areas of the laboratory for the witnessing of work performed for the customer; it is understood that such access should not conflict with rules of confidentiality of work for other customers or with safety.

- Preparing, packaging, and dispatching of test items needed by the customer for verification purposes.
- Maintaining of open contacts. The customer values advice and

guidance in technical matters, and opinions and interpretations based on results. Contact with the customer, especially in large assignments, should be maintained throughout the work. The laboratory should inform the customer of any delays or major deviations in the performance of the tests.

10.2 Feedback

POLICY

The laboratory seeks feedback from the customer.

PROCEDURE

Positive and negative feedback can be obtained passively through ongoing communications with the customer (e.g., review of test reports with customers) or actively through customer satisfaction

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surveys. The feedback is used to improve the quality management system, testing activities, and customer service.

SECTION 11 – COMPLAINTS

The purpose of this section is to assure that customer complaints are addressed and corrected. This includes requests to verify results or analytical data.

POLICY

Records are maintained of all complaints and follow-up. The laboratory reviews all complaints and determines appropriate action.

PROCEDURE

All customer complaints are documented by the person receiving the complaint and addressed by management.

Records of complaints include the following information:

- Details of the complaint
- Investigation
- Corrective action
- Follow-up verification

11.1 Record-Keeping

1. Record complaints on a Corrective Action Request (CAR) form.
2. Perform whatever immediate corrections can be made and record these details.
3. Forward to the concerned supervisor's attention.
4. Supervisor determines whether a corrective action needs to be taken to prevent recurrence.
5. A root cause analysis is performed to identify what the true cause of the complaint was and records are kept.
6. Take corrective action to prevent recurrence and record actions.
7. Perform follow-up verification to ensure the appropriate corrective action was effective and record verification.

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8. All CAR/PAR form records are kept in the Quality Manager's

Office.

11.2 Written Responses

1. Any required written responses to the clients are authored by the Laboratory Director.
2. Copies of the written response are attached to the CAR.
3. The Quality Manager conducts final review.

See Section 13 for additional Corrective Action procedures.

SECTION 12 – CONTROL OF NON-CONFORMING TESTING

NON-CONFORMING WORK is work that does not meet acceptance criteria or requirements. Non- conformances can include unacceptable quality control results or departures from standard operating procedures or test methods (See Section 23 Quality of Test Results). Requests for departures from laboratory procedures are approved by the Quality Manager and documented on a CAR form.

POLICY

The policy for control of non-conforming work is to identify the nonconformance, determine if it will be permitted, and take appropriate action. All employees have the authority to stop work on samples when any aspect of the process does not conform to laboratory requirements.

Nonconforming samples are documented on accompanying submission forms, i.e. LIMS, bench sheet, or logbook. Equipment problems are recorded in the appropriate equipment maintenance log. All non-conformances are recorded on the Corrective Action Request form.

PROCEDURE

1. Record the occurrence of the nonconforming event on the CAR form and/or the equipment maintenance logbook.

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2. Report the non-conforming event to the Quality Manager or Technical Director.

3. Suspend further work and begin an investigation to correct the non-conformance.

4. Notify the client of all incorrectly reported results, if applicable.
5. Correct non-conformance, if possible.
6. Initiate corrective action to prevent recurrence of the non-conformance.
7. Repeat the test or analysis, if possible.

The procedure ensures that:

- Responsibilities and authorities for the management of nonconforming work are designated and actions (including halting of work and withholding of test reports as necessary) are defined and taken into consideration when nonconforming work is identified.
- An evaluation of the significance of the nonconforming work is made.
- Correction is taken immediately, together with any decision about the acceptability of the nonconforming work.
- Where necessary, the customer is notified and the work is recalled.
- The responsibility for the resumption of work is authorized by the Quality Manager.
- Identification of nonconforming work or problems with the quality management system or with testing activities can occur at various locations within the quality management system and technical operations such as:
- Customer complaints
- Quality control
- Instrument calibration
- Checking of consumable materials
- Staff observations or supervision

- Test report checking

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- Management reviews
- Internal or external audits

Required Documentation for nonconforming work:

- CAR form
- Signed waiver of deviation when required by client agreement
- "Calibration Void – Do Not Use" label
- "Out of Service – Do Not Use" label

12.1 Root Cause Analysis

POLICY

Where evaluation indicates that nonconforming work could recur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures, the corrective action procedures given in Section 13 are followed to identify the root cause(s) of the problem and to prevent recurrence on nonconformance.

PROCEDURE

Identify situations warranting corrective action investigation. They include but are not limited to: Failure to comply with test method including all applicable procedures necessary to ensure the integrity and representative nature of the sample.

- Presentation of uncertain knowledge as to compliance with test methods including all applicable procedures necessary to ensure the integrity and representative nature of the sample.
- Failure or suspected failure in method performance as demonstrated by results provided by quality control samples.
- Lack of relevant evidence provided by quality audit, proficiency testing, or customer feedback.
- Lack of relevant evidence provided by data validation.
- Neglect to check the inherent property of the sample that compromises the testing.

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SECTION 13 – CORRECTIVE ACTION

CORRECTIVE ACTION is the action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

POLICY

Deficiencies cited in external assessments, internal quality audits, data reviews, complaints, or managerial reviews are documented and require corrective action. Corrective actions taken are appropriate for the magnitude of the problem and the degree of risk. Corrective action always begins with an investigation to determine root cause(s) of the problem.

POLICY

Where the identification of nonconformities or departures casts doubts on compliance of policies, procedures, regulations, quality standards, the appropriate areas of activity are promptly audited in accordance with Section 16.

PROCEDURE

Problems with the quality management system or technical operations of the laboratory may be identified through a variety of activities, such as control of nonconforming work, internal or external audits, management reviews, feed-back from customers, or staff observations.

Corrective action investigations are documented and required changes to operational procedures are implemented. The corrective action request, investigation and resolution are recorded on a CAR/PAR form.

All deficiencies are investigated and a corrective action plan developed and implemented if determined necessary. The implementation is monitored for effectiveness.

Specific corrective action protocols specified in test methods may override general corrective action procedures specified in this manual.

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The appropriate authorities for the implementation of a corrective action with their responsibilities include:

13.1 CAR Originator

✐ Initiate CAR/PAR when a need for corrective or preventive action is identified.

13.2 QA Manager

- ✐ Assign CAR/PAR number and update master CAR index.
- ✐ Review and assign CAR to supervisor.
- ✐ Approve proposed corrective action.
- ✐ Monitor CAR/PAR status to ensure complete and timely response.
- ✐ Approve close-out of corrective action.
- ✐ File completed CAR/PAR and CAR index.
- ✐ Track CAR/PAR completion dates.
- ✐ Provide copy of completed CAR to the CAR Originator.
- ✐ Train and support users of the CAR system.
- ✐ File copies of objective evidence which support verification and validation of CAR closure

13.3 Supervisor

- ✐ Review CAR and determine if corrective action is warranted.
- ✐ Assign CAR to the responsible personnel for action needed.
- ✐ Review corrective action to verify implementation.
- ✐ Review and evaluate client comments and generate CAR, if appropriate.

13.4 Personnel

✐ Investigate and determine root cause(s) of nonconformance. ✐ Identify and implement timely corrective or preventive action.

13.5 Selection and Implementation of Corrective Actions

ROOT CAUSE is the condition or event that, if corrected or eliminated, would prevent the recurrence of a deficiency. Once an deviation or non-conformance noted, the first action is an investigation to

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determine the root cause. Records are maintained of nonconformities requiring corrective action to show that the root cause(s) was investigated, and includes the results of the investigation.

POLICY

After determining the cause(s) of the problem, potential corrective actions are identified.

PROCEDURE

The most likely action(s) (this includes practical and/or reasonable) are selected and implemented to eliminate the problem and to prevent recurrence. Where uncertainty arises regarding the best approach for analysis of the cause of deviations that require corrective action, appropriate personnel will recommend corrective actions to be initiated. It should be noted that any corrective actions taken to eliminate the cause(s) of nonconformities or other departures are to a degree appropriate to address the magnitude of the problem and commensurate with the risks encountered (determine whether the benefit outweighs the cost).Controls are applied to prevent recurrence. The laboratory documents and implements the required changes resulting from corrective action investigations.

13.6 Monitoring of Corrective Action

POLICY

After implementing the corrective action(s), the laboratory monitors the results to ensure that the actions taken have been effective in overcoming the problems originally identified.

PROCEDURE

Monitoring is assigned to an appropriate individual such as the originator of the CAR/PAR or the originator's manager. Changes resulting from corrective action are documented by the originator in CAR/PAR, and standard operating procedures or work instructions (if applicable). The Quality Manager monitors the status to ensure corrective actions are complete in a timely manner.

13.7 Technical Corrective Action

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CAUSE ANALYSIS in corrective action investigates the root cause of the problem.

POLICY

Sample data associated with a failed quality control are evaluated for the need to be reanalyzed or qualified.

PROCEDURE

Unacceptable quality control results are documented, and if the evaluation requires cause analysis, the cause and solution are recorded. The analyst is responsible for initiating or recommending corrective actions and ensuring that deviations of quality control acceptance criteria are documented. Analysts routinely implement corrective actions for data with unacceptable QC measures. First level correction may include re-analysis without further assessment. If the test method SOPs addresses the specific actions to take, they are followed. Otherwise, corrective actions start with assessment of the cause of the problem.

Technical/Laboratory director reviews corrective action reports and suggest improvements, alternative approaches, and procedures where

needed. If the data reported are affected adversely by the nonconformance, the client is notified in writing.

The discovery of a non-conformance for results that have already been reported to the client must be immediately evaluated for significance of the non-conformance, its acceptability to the client, and determination of the appropriate corrective action.

13.8Exceptionally Permitting Departures from Documented Policies and Procedures

POLICY

The laboratory allows the release of non-conforming data only with approval by the appropriate Technical Director or their designee on a case-by-case basis. Planned departures from procedures or policies do not require audits or investigations.

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PROCEDURE

Permitted departures for non-conformances, such as QC failures, are fully documented and include the reason for the departure, the affected SOP(s), the impact of the departure on the data, and the data.

SECTION 14 – PREVENTIVE ACTION

Preventive Action is the action taken to eliminate the causes of potential non-conformity, defect, or other undesirable situation in order to prevent occurrence.

PREVENTIVE ACTION, rather than corrective action, aims at minimizing or eliminating inferior data quality or other non-conformance through scheduled maintenance and review, before the non- conformance occurs.

Preventive action includes, but is not limited to, review of QC data to identify quality trends, regularly scheduled staff quality meetings, annual budget reviews, annual managerial reviews, scheduled column trimming, running a new LIMS system in tandem with the old system to assure there is at least one working system, and other actions taken to prevent problems. Action may result from the review of operational procedures and analysis of data, this includes trend analysis, analysis of proficiency testing results, and risk analysis.

Records of preventive action include the following information:

- Details of potential nonconformities
- Investigation
- Preventive action
- Follow-up verification

POLICY

Preventive Analysis of data

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Opportunities for needed improvement and potential sources of nonconformities, either technical or with the quality management system shall be identified. If action is required, action plans are developed, implemented and monitored, to reduce the likelihood of occurrence of such nonconformities and to take advantage of the improvement opportunities. All employees have the authority to recommend preventive action procedures, however management is responsible for implementing preventive action.

POLICY

The preventive action procedure includes the initiation of such actions and application of controls to ensure that they are effective.

PROCEDURE

1. Select the potential problem (or process to be improved).

Note - when a team is being utilized, the team members must fundamentally agree that a potential problem exists. They must review the background and what drives the process and then clarify their thoughts by writing a problem statement. The quality tools that may be useful for this process include "Brainstorming", "Affinity Diagrams", "Interrelationship Diagram", "Voice of the Customer", and "Gap Analysis".

2.Describe the current situation by collecting baseline data, describing the process, confirming the problem statement, and setting improvement targets.

Note - the quality tools that may be useful for this process include

"Affinity Diagrams", "Bar Charts", "Control Charts", "Histograms", "Voice of the Customer", "Flowcharts", "Pareto Analysis", "Gap Analysis", and "Benchmarking/Completive Analysis".

3. Choose the most likely cause by conducting a root-cause analysis, prioritizing root causes, and using a cost/benefit analysis.

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Note - the quality tools that may be useful for this process include "Cause and Effect Diagram/Fishbone", "Pareto Analysis",

"Prioritization Matrix", "Spreadsheet", and "Value Analysis".

4. Develop a solution/action plan by identifying solutions, mapping solutions to root causes, conducting cost/benefit analysis, assess barriers to implementation, identifying countermeasures to barriers, and writing an action plan.

Note- the quality tools that may be useful for this process include

"Benchmarking/Completive Analysis", "Value Analysis", "Force-

Field Analysis", "Brainstorming", and "Gantt Chart/Action Plan".

5. Obtain management approval to implement improvements.

6. Implement the solution/action plan by conducting a test pilot study, evaluating test results, revising the action plan, implementing and monitoring the revised plan, and summarizing the results.

Note - the quality tools that may be useful for this process include

"Check sheets", "Control Charts", "Line Chart/Run Chart",

"Histograms", "Pie Charts", "Scatter Diagram", "Gannt Chart/ Action Plan", and "Work Breakdown Structure".

7. Review and evaluate results to determine whether solution did work through the completion of the action plan, if the best solution was selected, and if the correct cause was selected.

Note - the quality tools that may be useful for this process include

"Line Chart/Run Chart", "Gannt Chart/Action Plan", "Planning Matrix", and "Cause and Effect Diagram".

8. Reflect and action learning.

Note - the quality tools that may be useful for this process include

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"Lessons Learned", "Plan- Do-Check-Act (PDCA)", and "Planning

Matrix".

Records are logged, identified and kept on file as laboratory improvements.

SECTION 15 – CONTROL OF RECORDS

RECORDS are a subset of documents, usually data recordings that include annotations, such as daily refrigerator temperatures posted to a laboratory form, lists, spreadsheets, or analyst notes on a chromatogram. Records may be on any form of media, including electronic and hard copy. Records allow for the historical reconstruction of laboratory activities related to sample-handling and analysis.

Records apply to all quality and technical records. Quality records include audit reports, management review, corrective action requests, and preventive action requests. Technical records include observations, calculations, derived data, calibration records, personnel records, and test reports.

Note - forms are not records until they are completed.

Records are available to demonstrate conformance to requirements and effective operation of the

Quality Management System. Quality records from suppliers are also controlled.

Records may be in the form of any type of media, such as hard copy or electronic media.

Technical records are accumulations of data and information that result from carrying out tests and which indicate whether specified quality or process parameters are achieved. They may include forms, contracts, work sheets, work books, note books, instrument printouts, magnetic media, check sheets, work notes, control graphs, test reports, calibration certificates, customer's notes, papers and feedback, and test reports to customers.

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Typical records maintained in the laboratory include but are not limited to:

- Instrument and equipment maintenance logbooks
- Calibration record of instruments and analytical processes
- Records associated with test method quality control plans (e.g., control charts)
- Records associated with approved SOPs
- Spreadsheets used to calculate accuracy and precision of instruments
- Standard logbooks
- Sample logbooks

Laboratory Director or designee shall:

- ✐ Specify any alternative retention period
- ✐ Ensure that records specified in the Quality System are handled in accordance with the requirements of this document

Quality Manager shall:

- ✐ Ensure the unique indexing of records.
- ✐ Review material prior to storage.
- ✐ Assign storage location numbers for the material

Records custodian (all personnel) shall:

- ✐ Collect, store, and maintain records for the minimum retention period or the period designated by the Technical Manager
- ✐ Ensure that records retained are legible

POLICY

Southeast Alaska Laboratories maintains a record system appropriate to its needs. Southeast Alaska Laboratories records all laboratory activities, and complies with applicable standards or regulations as required. Original observations, calculations, derived data and sufficient information to establish an audit trail, calibration records, personnel records and a copy of each test report issued are retained for 5 years.

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The records for each test or calibration shall contain sufficient information to facilitate, if possible, identification of factors affecting the test uncertainty and to enable the test or calibration to be repeated under conditions as close as possible to the original. The records include the identity of personnel responsible for sampling, performing of each test and checking of results.

POLICY

Observations, data, and calculations are clearly and permanently recorded and identifiable to the specific job at the time they are made.

POLICY

Changes to test data are made so as not to obscure or delete the previous data entry.

PROCEDURE

Mistakes are crossed out with one line and the correct value entered alongside. Mistakes are not erased, made illegible, or deleted. All alterations to records are made by striking with one line, dated, reason of corrections and signed or initialed by the person making the correction. In the case of computer-collected data, similar measures are taken to avoid loss or change of original data.

PROCEDURE

The laboratory retains all original observations, calculations and derived data, calibration records, and a copy of the test report for a minimum of five years. Records are maintained in the designated archival area.

All records (obsolete SOPs), and test reports, are safely stored and held secure in locked areas, and in confidence to the customer. Records of all procedures to which a sample is subjected while in the possession of the laboratory are kept. Handwritten records must be legible and made with indelible ink immediately after an observation, after data is collected and/or after calculations are made.

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15.1 Records Management and Storage

POLICY

Records, including electronic records, are easy to retrieve, legible, and protected from deterioration or damage; held secure and in confidence; and are available to accrediting authorities for a minimum of five years.

All records are to be legible and shall be retained in such a way that they are readily retrievable in facilities that provide a suitable environment to prevent damage or deterioration and to prevent loss.

Access to records is secured through locked rooms and filing cabinets.

PROCEDURE

The retention times for records are generally set at 5 years. This provision changes if the dispute is in court; records are kept 5 years from the date of last deposition.

POLICY

The laboratory maintains a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting.

PROCEDURE

The master list of records is organized with the following information:

- Record No. / Form No.
- Record Name
- Filing Method (loose forms filed monthly, quarterly, semi-annual, annual or electronic)
- Active Files (files referred to within the work area) / Retention Period / Location
- Inactive Files (files referred to but not often and kept in storage) / Retention Period / Location
- Persons / Positions Responsible / Users

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POLICY

Archived information and access logs are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.

POLICY

In the event that the laboratory transfers ownership or goes out of business, records are maintained or transferred according to the clients' instructions.

POLICY

Electronic data is backed-up to prevent unauthorized access to or amendment of data/records on computers. All electronic records are backed-up daily. Access to protected records is limited to laboratory management or their designees to prevent unauthorized access or amendment. Data is password protected and controlled by the Laboratory Director or designee. Backups ensure integrity and availability of data/ information in the event of a system or power failure.

PROCEDURE FOR RECORD MANAGEMENT

Identification: Records are appropriately identified by a descriptive title clearly labeling the record.

Collection: The personnel/user identified for each record is responsible for collecting the record.

Indexing: Each record is assigned a unique name, number or alphanumeric identification, and date to distinguish it from other records with the same identification. This is referred to as the master index of records.

Accessing: Records are readily accessible to individuals requiring information contained in the record. Records are available to clients for the period agreed to per client agreements. Subcontractor's records, as specified by contract, are made available upon request.

Filing: Filing is considered the location where active records are kept. All records are physically or electronically filed by a method that enhances accessibility and retrieval by a user. If electronic files of raw data and

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other files are used, a full backup system is performed at least once a week.

Retention: A record's "retention" time refers to how long it is kept before it is either discarded or destroyed, or sent to off-site for long-term storage. Records are retained on- site for the minimum retention time 5 years and off site for 5 years. Records may be retained longer than the minimum retention time for the laboratory if court proceedings are held. Retention times for work area-specific records are determined by the laboratory and stated in their Quality System documents.

Maintenance: All records are filed and stored in an office or laboratory environment unless specific media and/or special environmental control are specified to prevent damage, deterioration, or loss.

Disposition: Records are disposed when the retention time has been exceeded. Disposition may be to discard or destroy the records, or as instructed by the Technical Manager. The Technical Manager may opt for long-term storage of records.

Storage: After the appropriate retention time, records are stored long term as defined by the Laboratory Director or designee. Each department is responsible for the boxing and accurate indexing of its storage material.

SECTION 16 – AUDITS AND MANAGEMENT REVIEW

AUDITS measure laboratory performance and verify compliance with accreditation/ certification and project requirements. Audits specifically provide management with an on-going assessment of the quality system. They are also instrumental in identifying areas where improvement in the quality system will increase the reliability of data. Audits are of four main types: internal, external, performance, and system.

Notification of clients for events that cast doubt on the validity of the results is completed immediately within 24 hours in writing.

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16.1 Internal Audits

POLICY

The laboratory conducts internal audits of its quality systems activities, including data integrity, and the use of trained and qualified personnel annually. The internal audit program involves periodic audits conducted according to a predetermined schedule.

All elements of this Quality Manual will be audited each year and all relevant laboratory records are available to personnel conducting the audit. These audits are performed to verify operations continue to comply with the requirements of this Quality Manual and are effective.

PROCEDURE

The Quality Manual, test procedures, TNI Standard, and laboratory results are verified for compliance. It is the responsibility of the Quality Manager to plan and organize audits as required by the schedule and requested by management. The internal auditing plan is available for the management review meeting. Trained and qualified personnel who are independent of the activity to be audited conduct audits. Personnel are not to audit their own activities except when it can be demonstrated that an effective audit will be carried out. Audits are performed through the aid of a checklist prepared in advance to minimize the possibility of overlooking any details during the audit.

Generally, the types of audits include:

- Quality management system
  - Processes and procedures
  - Products, services, and reports

All investigations that result in findings of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients. Clients are notified within 2 hours, in writing, when audit findings cast doubt on the validity of the data. The area audited, the audit findings, and corrective actions are recorded by the Quality Manager or designee.

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Audits are reviewed after completion to assure that corrective actions were implemented and effective.

Quality Manager shall:

- Establish and maintain an audit schedule.
- Ensure that all employees have received the required training
- Plan audit
- Perform audit or assign somebody to perform audit
- Conduct audit meetings
- Prepare audit report
- Reconcile any disagreements between employees and procedures audited and, when necessary, submit disagreements to the management for reconciliation

Employee/Auditor (Technical Assessor) shall:

- Review documentation
- Participate in the audit, including the verification of effectiveness of corrective actions taken
- Collect objective evidence to support findings, Corrective Action Request (CAR) verifications, and follow-up of observations from prior audits
- Record findings

Employee being audited shall:

- Inform area personnel of the time and scope of the audit
- Assign knowledgeable guide(s) to accompany each audit team
- Ensure that timely access to processes, products, and documentation needed by the auditor(s), including objective evidence is provided
- Attend audit meetings
- Sign audit report

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Internal Quality Audit Plan

The Quality Manager is responsible for developing the plan and schedule for internal quality audits.

The audit plan and schedule shall:

✐ Address all applicable elements of the Quality System ✐ Identify the depth and frequency of each audit based on prior audit history and operational status of the area to be audited

Audit Team Selection

The Quality Manager selects qualified employees for each area to be audited who are not directly responsible for the performance of the activity being audited.

Note - Employees shall have received training in audit methods and objectives.

Preparing for the Audit

Auditors prepare for an audit by reviewing the TNI Standards, the Quality Manual, relevant procedures, corrective action requests, and preparing questions and checklists.

Conducting and Reporting the Audit

The supervisor/employee negotiates the opening and closing meeting times with the manager/supervisor of the area being audited.

The Team Leader conducts the opening meeting with the managers/supervisors of the area being audited.

The Auditors:

✐ Interview appropriate personnel and determine whether actual practices conform to the requirements of the documented policies, plans, procedures, and work instructions, using the prepared checklist and notes

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- Verify the effectiveness of CAR/PAR closed since the last audit, complete the Follow-up Verification section of the CAR forms and forward CAR forms to the Quality Administrator; or reissue a new CAR/PAR if the corrective action was ineffective
- Follow-up on actions taken to address observations noted in previous audits
- Categorize findings into nonconformance's and observations

- Combine observations that show a pattern of non-conformance into a non-conformance
- Initiate CAR/PAR and provide information to the Team Leader

The supervisor/employee conducts a closing meeting. The Quality Manager reviews the audit findings.

The supervisor/employee completes and provides the audit report and the completed checklists to the Quality Manager.

The Quality Manager reviews the audit report to ensure that it is complete, concise, consistent, unambiguous, and that all nonconformance and/or observations are factual and traceable to elements of the TNI Standard.

The Quality Manager distributes the audit report to the audited work area's supervisor/manager within two weeks of the audit.

The supervisor/manager ensures that CAR/PAR issued are responded to in a timely manner and as required.

POLICY

When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of test or calibration results, timely corrective action is taken and customers are promptly notified in writing if investigations show that laboratory results may have been affected.

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PROCEDURE

Nonconformities that can be resolved easily are to be corrected immediately, ideally during the audit. Records are made on the audit checklist. Nonconformities that require a more involved resolution are recorded on a CAR/PAR and resolved as described in Section 13.

Corrective actions and customer modifications must be kept on record for each audit deviation that casts doubt as described in this section.

POLICY

Follow-up audits are performed to verify and record the implementation and effectiveness of the corrective action taken.

PROCEDURE

The follow-up audit is performed at a mutually acceptable time between the area implementing corrective action and the auditor. This time is determined when the CAR is issued.

16.2 External Audits

POLICY

It is the laboratory's policy to cooperate and assist with all external audits, whether performed by clients or an accrediting authority.

POLICY

All external audits are fully documented and tracked to closure.

PROCEDURE

Management ensures that all areas of the laboratory are accessible to auditors as applicable and that appropriate personnel are available to assist in conducting the audit.

Any findings related to an external audit follow corrective action

procedures. Management ensures that corrective actions are carried out within the timeframe specified by the auditor(s).

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. 16.3 Performance Audits

Performance audits may be Proficiency Test Samples, internal single-blind samples, double- blind samples through a provider or client, or anything that tests the performance of the analyst and method.

The policy and procedures for Proficiency Test Samples are discussed in Section 23.

. 16.4 System Audits and Management Reviews

POLICY

The Laboratory management team annually conducts a review of the laboratory's quality management system to ensure that: ✐ The Quality System continues to be effective and suitable – fulfilling the changing and future needs of the laboratory and its clients.

- ✐ The Quality Management System is updated as necessary.
- ✐ The results of Internal Audits are reviewed.
- ✐ The defined Quality system is being implemented and followed.

Records of review findings and actions are maintained in the

Quality Manager's office.

PROCEDURE

The quality system is reviewed annually, and findings are recorded. Managers assure that actions are performed within agreed time frames. The review takes account of:

- ✐ Suitability of policies and procedures
- ✐ Reports from managerial and supervisory personnel
- ✐ The outcome of recent internal audits
- ✐ Corrective and preventive actions
- ✐ Assessments by external bodies
- ✐ Results of inter-laboratory comparisons or proficiency tests
- ✐ Changes in the volume and type of work undertaken

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- Feedback from customers, including complaints and customer satisfaction surveys
- Recommendations for improvement
- Other relevant factors, such as quality control activities, resources and personnel training

A minimum period for conducting a management review is once a year. Results of the review feed into the laboratory planning system and include goals, objectives and action plans for the coming year.

A management review can be supplemented by consideration of related subjects at regular management meetings.

The minutes are taken and recorded to reflect the following items:

1. Date, attendees, and the individual completing the minutes.
2. A review of the minutes from the previous management review and any action items.
3. New items for review may include:
   - Business planning – forecasting
   - Internal audit status
   - Corrective actions
   - Preventive actions
   - Process improvements
   - Training (issues and needs)
   - Equipment (calibration and maintenance program)
   - Client complaints
   - Resources - personnel (absence, increasing work load, surprises from clients)
   - Resources - equipment (time, capacity, failure)
   - External audits
   - Quality policy statement
   - Client satisfaction
   - Quality goals
   - Proficiency results
4. Discussion may identify trends.

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5. Action items that are identified should be assigned and documented in writing to individuals and an appropriate completion date agreed upon. Any communication plan is noted.
6. Any other business discussed is recorded with action items as appropriate.

Findings from management reviews and the actions that arise are recorded in the minutes of the meeting. Management will ensure that the actions are discharged within an appropriate and agreed upon timeline.

POLICY

The laboratory continually improves the effectiveness of its management system through the use of the quality policy, quality objectives, and audit results, analysis of data, corrective actions, and management review.

PROCEDURE

The laboratory has implemented a continual improvement philosophy within the management system. Every employee in the laboratory is encouraged to suggest new ideas for improving services, processes, systems, productivity, and the working environment.

Opportunities for improvement of operations and processes are identified by managers on a continual basis from ongoing feedback on operations and through management reviews.

Opportunities for improvement of services are identified by anyone within the organization.

Inputs for improvement opportunities are obtained from the following sources:

Customer feedback

Market research and analysis

- Employees, suppliers, and other interested parties
- Internal and external audits of the management system
- Records of service nonconformities

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- Data from process and service characteristics and their trends
- Opportunities for improvement may also be identified on a special project basis.

The following are listed only as examples:

✐Improving usefulness of bench space ✐

Reducing excessive inspection/testing ✐

Reducing excessive handling and storage ✐

Reducing test/calibration failures

Opportunities for improvement from daily feedback on operational performance (i.e., internal audits, customer feedback, and test failures) are evaluated by the Technical Manager or designee. Typically, they are implemented through the corrective and preventive action system.

Opportunities for improvement from analysis of longer-term data and trends are evaluated and implemented through the management review process. They are prioritized with respect to their relevance for achieving quality objectives. When opportunities for improvement are no longer supported by the current policy and objectives, management establishes new quality objectives, and possibly change the policy. Longer-term improvement projects are initiated through the management review process, as well as the corrective and preventive action system.

Management evaluates Service improvement opportunities. They are implemented through the supervisor of the laboratory who ensures that the improvements are validated and appropriate level of quality control is performed on an ongoing basis.

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SECTION 17 – PERSONNEL, TRAINING, AND DATA INTEGRITY

17.1 Job Descriptions

POLICY

Job descriptions are available for all positions that manage, perform, or verify work affecting data quality, and are located in the Human Resource office.

PROCEDURE

Job descriptions include the specific tasks, minimum education and qualifications, skills, and experience required for each position.

17.1.1-Laboratory Director:

The Laboratory Director is in charge of all laboratory activities, and is the highest level manager. The laboratory director signs the Quality Manual.

Establishes and manages a program to maintain quality and integrity for all Center laboratory studies and the processing of regulatory samples.

Maintain a QA program to help ensure the quality and integrity of data produced by SEAL.

Provide the resources and commitment to implement the LQM.

Develop overall SEAL scientific policy and laboratory programs to support policy.

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Review reports of SEAL QA activities which include internal audit reports, corrective/preventive actions, management reviews, and continual improvement efforts.

Maintain overall responsibility to ensure that deviations and complaints are addressed.

The Laboratory Director shall audit the lab periodically and maintain written and properly signed records of each periodic inspection to ensure that quality standards are being met. The scope and frequency of these activities will be determined in consultation with senior management.

Periodically submit to the consultant written status reports, noting any problems and the corrective actions taken.

Maintain and update the SEAL Laboratory Quality Manual.

Maintain copies of all pertinent documentation and records.

Ensure that a SEAL/State of Alaska Tracking System number has been assigned to each test upon approval.

Audit laboratories in accordance with a predetermined schedule.

Generate QA status reports, which include internal audit reports, corrective/preventive actions, and continual improvement efforts.

17.1.2-Scientific Director

For each laboratory test, a scientist or other professional of appropriate education, training, and experience, or combination thereof, shall be identified as the lead scientist. The lead scientist has overall responsibility for the technical conduct of the test, as well as for the interpretation, analysis, documentation, and reporting of results. The lead scientist shall be responsible for the following:

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Developing the testing protocol per State of Alaska Statue.

Ensuring that changes (amendments) to the project/protocol are documented and approved.

Maintaining pertinent documentation regarding all project/protocol activities.

Communicating on a regular basis with contractors who provide project/protocol support.

Maintaining results of relevant analyses as part of the raw data for the protocol.

Documenting and addressing circumstances that may affect the quality and integrity of the laboratory test.

Preparing a final report in accordance with SEAL policies and State of Alaska Statute.

Ensuring that raw data, documentation, protocols, specimens, and final reports (if necessary) are archived and/or properly disposed of at the completion of a test.

Ensuring that the test is conducted in a safe manner and in accordance with the safety plan, safety procedures, and safety requirements.

Conducting the test in accordance with other relevant regulations.

17.1.3-Research Facility Management

For each laboratory test, management shall:

Designate a lead scientist before the test is initiated.

Replace the lead scientist promptly if it becomes necessary to do so during the conduct of a test.

Ensure that projects/protocols are properly amended to reflect changes in responsible personnel.

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Ensure that personnel clearly understand the functions they are to perform.

17.1.4-The Laboratory Manager

Serves as project oversight on all contracts whose obligations are to provide all aspects of building maintenance for systems and equipment relating to laboratory operations. This includes, but is not limited to, room and fume hood airflow, cold box and structural maintenance (walls, ceilings, floors), water, gas, and electrical services.

Provides guidance for the maintenance of all SEAL-installed equipment. This includes offering technical assistance, making recommendations, and preparing requisitions necessary to contract for the required services.

Provides assistance and recommendations on space utilization and assists in relocations.

Receives and distributes laboratory and other supplies ordered for use.

Operates and maintains the FDA Scientific Supply Store.

Arranges for the shipment of appropriately packaged items, with the possible exceptions of radioactive materials and select agents and toxins, to their destination via GSA contract-approved transportation company or courier service.

Delivers gas cylinders and picks up empty gas cylinders upon request of laboratory personnel.

Provides for pick up, processing, and transportation of hazardous waste to a centralized location for disposal.

Receives and stores equipment and supplies for use in the limited access areas at designated facilities.

Ensures that all laboratory work conducted at SEAL is performed in compliance with all applicable occupational safety and health and environmental management regulations, standards and SEAL policies

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17.1.5-Board of Directors

The Board of Directors are investors who have purchased 1 or more share(s) of SEAL. The Board will meet bi-annually to receive updates on the financial outlook of SEAL as well as a general overview of the operations. Each member will have the same number of votes as the number of shares that he/she owns. The Board will have binding decision making authority on the following decisions: Hiring and salary of the consultant, further expansion of SEAL, distribution of dividends, and purchase of real property excluding equipment necessary for testing of product.

17.1.6-Consultant

The consultant is hired by the Board of Directors (BOD). He/she must have a scientific background and have a firm understanding of chemistry and testing. A bachelor's degree in chemistry or a related field is highly recommended. The consultant will be the liaison between the BOD and the CEO, lab director/lead scientist. He/she will be expected to report, in clear non-scientific language, the financial and scientific output of the lab. He/she will also be expected to facilitate conversations with local and state government agencies for purposes of licensing, testing and compliance. Additionally he/she will be responsible for the evaluation/performance review of the CEO. The consultant will be the only personnel over which the BOD has authority.

17.1.7-Technical Director(s)

Day to day supervision of technical laboratory operations is the responsibility of the Technical Directors who are full-time members of the staff and who assure reliable data through the following activities: monitoring quality control, corroborating the analysis performed, and signing demonstrations of capability.

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The Technical Directors certify that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited.

17.1.8 Quality Manager

The Quality Manager has the authority and responsibility for ensuring that the quality system is implemented and followed.

The Quality Manager has direct access to the Laboratory Director and is independent of operations where the Quality Manager has oversight.

The Quality Manager:

- is the focal point for the quality system and has oversight of quality control data.
- evaluates data objectively and performs assessments without managerial influence.
- arranges for, or conducts, internal audits annually; and,
- notifies laboratory management of deficiencies (or opportunities for continuous improvement) and monitors corrective actions.
- keeps the Quality Manual current.
- signs the demonstrations of capability.

17.2 Data Integrity and Ethics

DATA INTEGRITY is the result of the processes that together assure valid data of known and documented quality. Data integrity and ethics procedures in the laboratory include training, signed, and dated integrity documentation for all laboratory employees, periodic monitoring of data integrity, and documented data integrity procedures.

POLICY

Technical managers uphold the spirit and intent by supporting integrity procedures, by enforcing data integrity procedures, and by signing and dating the data integrity procedure training forms.

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POLICY

Data integrity procedures and evidence of inappropriate actions are reviewed annually or through regularly scheduled internal audits, and are updated by management.

POLICY

The mechanism for confidential reporting of ethics and data integrity issues is (1) unrestricted access to senior management, (2) an assurance that personnel will not be treated unfairly for reporting instances of ethics and data integrity breaches, and (3) anonymous reporting.

POLICY

Employees are required to understand, through training and review of quality systems documents, that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences such as immediate termination, or civil/criminal prosecution.

POLICY

Any potential data integrity issue is handled confidentially until a follow-up evaluation, full investigation, or other appropriate actions have been completed and the issues clarified. Inappropriate activities are documented, including disciplinary actions, corrective actions, and notifications of clients, if applicable. These documents are maintained for a minimum of 5 years. PROCEDURE

Any determination for detailed investigation of data integrity issues must be communicated to senior management. Allegations are investigated and remain confidential to the extent necessary. Documentation for all investigations that result in findings of inappropriate activity include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

Data integrity procedures are reviewed annually and are periodically monitored through in- depth data review, records review, or other thorough check processes.

17.3 Data Integrity and Ethics Training

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POLICY

Data integrity training is provided for all employees initially upon hire and annually thereafter.

PROCEDURE

Attendance at an initial data integrity training (part of new employee orientation) and the annual refresher training is recorded with a signature attendance sheet or other form of documentation that demonstrates all staff have participated and understand their obligations related to data integrity.

Training records regarding data integrity and ethics are signed and dated by senior management.

When contracted technical or support personnel are used, management is responsible for ensuring that they are trained to the laboratory's quality system and data integrity procedures, competent to perform the assigned tasks, and appropriately supervised.

Topics covered are provided in writing and provided to all trainees.

17.4 General Training

POLICY

All personnel are appropriately trained and competent in their assigned tasks before they contribute to functions that can affect data quality. It is management's responsibility to assure personnel are trained.

POLICY

Only trained personnel are authorized to perform specific tasks.

POLICY

Training records are kept on individual training forms in the Quality Manager's Office.

PROCEDURE

New staff members are given introductory training and orientation upon arrival. The manager introduces the new employee to a general

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set of training requirements that must be completed prior to any further activities in the laboratory. Attendance at training sessions is documented on signature sheets.

The introductory training consists of:

1. An overview of the company's history and its business philosophy
2. A walk-through of the facility
3. A review of the company's personnel/orientation manual
4. A review of the safety and chemical hygiene plan and a safety equipment walk- through/usage review
5. Laboratory Ethics

17.4.1 Safety Training

All Laboratory Personnel will receive: Monthly Laboratory Safety Training

Unsatisfactory results require re-training. The trainee is deemed competent by the trainer when they produce satisfactory results. The trainer performs a follow-up evaluation to determine the effectiveness of training within one month after training.

The department head is responsible for ensuring effective training. Upon completion of initial training, all analysts are required to prove proficiency prior to analyzing client samples.

17.5 Customer Complaints

Lab Personnel, Supervisor and other employees likely to receive complaints are trained in complaint handling procedures applicable to their functions. These employees are trained in the relevant policies and procedures of the Quality Manual.

17.6 Change Control

All employees are to be advised that they perform their jobs as instructed or as covered by standard operating procedures (SOP's).

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They are NOT allowed to change tasks covered by SOP's until the change is approved according to the document control section of the Quality Manual.

Further procedures are defined on an ad hoc basis at the time training needs are identified for each employee.

17.7 Documentation

Comprehensive training of all laboratory staff is vital to the continued development of the staff as well as the continued maintenance of the laboratory's quality program. All staff training will be conducted under the direction of the Technical Manager. All activities relative to training will be thoroughly documented and maintained in each employee's personnel training file. Included in this file are copies of academic transcripts and/or degrees, resumes, job descriptions, all in-house training records and all documents pertaining to external training (e.g. seminars, instrument manufacturer training courses, etc.).

All classroom and on-the-job training are recorded on the training log sheet by the supervisor or trainer for the employee. Records should include orientation, Quality Manual review, and appropriate SOPs and test methods.

17.8 Competence and Qualification

POLICY

Management ensures the competency of all specific equipment operators, those performing tests and/or calibrations, those evaluating results and signing test reports. Appropriate supervision is provided for employees undergoing training. Personnel performing specific tasks are qualified on the basis of appropriate education, training, experience and/or demonstrated skills, as required.

In addition, personnel responsible for the opinions and interpretations included in test reports also have:

1. Relevant knowledge of the technology used for the manufacturing of the items, materials, products tested, or the

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way they are used or intended to be used and of the defects or degradation that may occur during or in service
2. Knowledge of the general requirements expressed in the standards
3. An understanding of the significance of deviations found with
4. regard to the normal use of the items, materials, or products concerned

PROCEDURE

Management defines the minimum levels of qualification and experience necessary for all posts within the laboratory. Continued competence is monitored and where this is not achieved, the need to retrain personnel is considered and provided.

17.8.1 Training Policies and Procedures

Where a method or technique is not in regular use, verification of personnel performance before they undertake tests, will be necessary.

POLICY

Management will formulate the goals with respect to the education and the skills of the laboratory personnel. The training program is relevant to the present and anticipated tasks of the laboratory. The effectiveness of the training actions taken is evaluated.

PROCEDURE

The skills and knowledge are defined in the job description for each job function. Management compares the job description to the skills and knowledge of the new incumbent to determine the training needs.

Training in the laboratory must include all methods or parts of methods and techniques that personnel are asked to perform. Minimally, the analyst must demonstrate competency through observation by management and verification using replicate and/or check samples. For technicians who perform only parts of the method, confirmation of competency may be verified by observation only. Re-verification of all personnel must be performed annually on all methods or techniques pertinent to their job description.

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In some cases it may be appropriate to define competence related to a particular technique or instrument rather than methods. If so, it will be necessary to define for each method, the necessary technique-based competence required together with any additional requirements.

Employees

POLICY

Competent permanent or contractual employees are employed in the laboratory. The Technical Manager ensures that contractual, additional technical employees, and key support personnel are supervised and work in accordance to the policies and procedures of this Quality Manual.

PROCEDURE

Testing must be either performed or supervised by an experienced person qualified to degree level. Personnel have relevant practical work experience before being allowed to perform accredited work.

SECTION 18 – ACCOMMODATIONS & ENVIRONMENTAL CONDITIONS

POLICY

Laboratory facilities are appropriate and provide security to attain correct performance of tests and/or calibrations. This may include, but not limited to, energy sources, lighting, heating, ventilation and any other environmental conditions. Appropriate care is taken to ensure that the environment does not invalidate the results or adversely affect the required quality of any measurement. Particular care is taken when sampling, tests and/or calibrations are undertaken at sites other than a permanent laboratory facility. The technical requirements for accommodation and environmental conditions that can affect the results of tests and calibrations are documented.

POLICY

Critical environmental conditions are monitored, controlled and recorded as required by the relevant specifications, methods, and procedures or where they may influence the quality of the results. As appropriate to the technical activities concerned. Tests and calibrations

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are stopped when the environmental conditions jeopardize the results of the tests and/or calibrations.

POLICY

Effective separation between neighboring areas is made when the activities are incompatible. Measures are taken to prevent cross contamination.

POLICY

Access to and use of areas affecting quality of the tests and/or calibrations is defined and controlled by restriction of areas to authorized personnel only.

POLICY

Measures are taken to ensure good housekeeping in the laboratory. Special procedures are prepared when necessary.

PROCEDURE

Laboratory space is arranged to minimize cross-contamination between incompatible areas of the laboratory.

SECTION 19 – TEST METHODS AND METHOD VALIDATION

POLICY

A method is validated before it is put into use. All methods are published or documented.

19.1 Demonstration of Capability (DOC)

A DEMONSTRATION OF CAPABILITY (DOC) is a procedure to establish the ability of the analyst to generate data of acceptable accuracy and precision.

POLICY

The laboratory confirms that it is capable of generating data of acceptable accuracy and precision on all methods before employing them.

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PROCEDURE

The DOC is documented on the DOC spreadsheet form, and these completed forms are kept in the training files for each analyst in the Quality Manager's Office.

A DOC is performed for each substance to be analyzed whenever the method, analysts, substance to be analyzed, or instrument type is changed.

The Technical Director certifies that technical staff members in their area of expertise are trained and authorized to perform all tests for which we are accredited by signing the DOC form.

19.2 On-Going (or Continued) Proficiency

After the demonstration of capability is completed, on-going proficiency is maintained and demonstrated annually through the analysis of either single-blind samples, performing another DOC, or use of four consecutive laboratory control samples compared to pre-determined acceptance limits for precision and accuracy. This is documented in the training file of each analyst.

19.3 Initial Test Method Evaluation

For chemical analyses, the INITIAL TEST METHOD EVALUATION involves the determination of the Limit of Detection (LOD), confirmation of the Limit of Quantitation (LOQ), an evaluation of precision and bias, and an evaluation of the selectivity of the method.

19.3.1 Limit of Detection (LOD)

The LIMIT OF DETECTION (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte-and matrix specific and may be laboratory-dependent. (TNI Standard Glossary 2009).

19.3.2 Limit of Quantitation (LOQ)

The LIMIT OF QUANTITATION (LOQ) is an estimate of the minimum amount of a substance that can be reported with a specified degree of confidence. (TNI Standard Glossary 2009).

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POLICY

If an LOD study is not performed, concentrations less that the Limit of Quantitation are not reported. If results are not reported outside of the calibration range (low), the LOD determination is not required.

POLICY

The lowest calibration standard is equal to the LOQ.

POLICY

The LOQ will always be greater than the LOD.

PROCEDURE

LODs are determined from a quality system matrix using all sample processing steps, and are verified annually or when there is a change in the test method or instruments affects sensitivity.

The LOQ is no less than 3 times the method detection level or 10 times the LOD. The LOQ may also be established by the low calibration standard.

The LOQ is verified using a quality systems matrix sample spiked at 1-2 times the determined LOQ that returns a concentration within the acceptance criteria for accuracy, according to the requirements of the method or client data quality objectives.

19.3.3 Precision and Bias

PRECISION is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms.

BIAS is the systematic error that contributes to the difference between the mean of a significant number of test results and the accepted reference value.

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POLICY

Precision and bias are determined for standard and non-standard methods.

PROCEDURE

Precision and bias are determined for standard methods through the performance of a Demonstration of Capability.

Precision and bias using non-standard, modified standard or laboratory-developed methods are compared to the criteria established by the client (when requested), the method, or the laboratory.

Replicate spikes in a quality system matrix are analyzed according the procedures outlined in the TNI Standard, V1M4-2009 1.7.3.2. where applicable.

19.3.4 Selectivity

SELECTIVITY is the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.

POLICY

The laboratory evaluates selectivity through procedures defined in the test method SOPs.

POLICY

The range and accuracy of the values obtainable from validated methods (e.g., the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object) as assessed for the intended use is relevant to the customer's needs.

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PROCEDURE

Validation includes the specification of the requirements, determination of the characteristics of the methods, the comparison of the requirements with the values of the characteristics of the method, and a statement on the validity.

As method development proceeds, regular review by assign chemist is required to verify that the needs of the customer are still being fulfilled. Changing requirements requiring modifications to the development plan are approved and authorized.

19.4 Estimation of Uncertainty

ESTIMATION OF UNCERTAINTY consists of the sum (combining the components) of the uncertainties of the numerous steps of the analytical process, including, but not limited to, sample plan variability, spatial and temporal sample variation, sample heterogeneity, calibration/calibration check variability, extraction variability, and weighing variability.

PROCEDURE

The laboratory estimates uncertainty using the standard deviation calculated from routine quality control samples.

19.5 Laboratory-Developed or Non-Standard Method Validation

POLICY

Laboratory developed, modified standard methods, and non-standard methods require method validation.

METHOD VALIDATION is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled (TNI Standard 2009).

POLICY

Where applicable, the laboratory validates non-standard methods, laboratory- designed/developed methods, standard methods used outside their published scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use.

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POLICY

The range and accuracy of the values obtainable from validated methods (e.g. the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross- sensitivity against interference from the matrix of the sample/test object), is assessed for the intended use and whether it is relevant to the clients' needs.

PROCEDURE

The laboratory's method validation procedures include, at a minimum, the steps described in TNI-EL-V1M4-2009, 1.5 (TNI 2009). The laboratory records method validation results, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.

19.6 Control of Data

POLICY

All calculations and all relevant data are subject to appropriate checks in a systematic manner.

POLICY

Commercial off-the-shelf software (e. g. word processing, database and statistical programs) used within the designed application range is considered sufficiently validated when in-house programming is not used.

PROCEDURE

The laboratory assures that computers and software are protected, maintained, and secure through measures such as documentation, locked access, and control of the laboratory environment.

The laboratory assures that computers, user-developed computer software, automated equipment, or microprocessors used for the acquisition, processing, recording, reporting, storage, or retrieval of test data are:

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a) documented in sufficient detail and validated as being adequate for use
b) protected for integrity and confidentiality of data entry or collection, data storage, data transmission and data processing;
c) maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of environmental test data; and
d) held secure including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.

SECTION 20 – EQUIPMENT

20.1 General Equipment Requirements

POLICY

The laboratory is furnished with all items for sampling, measurement and test equipment required for the correct performance of the tests and/or calibrations (including sampling, preparation of test and/or calibration items, processing and analysis of test and/or calibration data). When equipment is used outside the laboratory's permanent control, it ensures that the requirements of this Quality Manual are met.

POLICY

Equipment and software used for testing, calibration and sampling are capable of achieving the accuracy required and comply with specifications relevant to the tests and/or calibrations concerned. Calibration programs are established for key quantities or values of the instruments where these properties have a significant effect on the results. When received, equipment, including that used for sampling, is checked to establish that it meets the laboratory's specification requirements, complies with the relevant standard specifications, and is checked and/or calibrated in accordance with section 21 before use.

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POLICY

Equipment is used in an environment appropriate to its proper performance. All equipment required by a test is described in each method, including the equipment's tolerances.

POLICY

Equipment is operated only by authorized personnel.

POLICY

The SOP# for Calibration and Verification, is utilized as an established plan for safe handling, transport, storage, use and maintenance (including calibration) of measuring equipment, and appropriate use of correction factors to ensure proper functioning and in order to prevent contamination or deterioration.

PROCEDURE

Up-to-date instructions on the use and maintenance of equipment (including any relevant manuals provided by the manufacturer of the equipment) are readily available for use by laboratory personnel.

All equipment is calibrated or checked before being placed into use to ensure that it meets laboratory specifications and the relevant standard specifications.

Test equipment, including hardware and software, are safeguarded from adjustments which would invalidate the test results measures by limiting access to the equipment and using password protection where possible.

Equipment that has been subject to overloading, mishandling, given suspect results, or been shown to be defective or outside specifications is taken out of service, isolated to prevent its use, or clearly labeled as being out of service until it has been shown to function properly. If it is shown that previous tests are affected, then procedures for non-conforming work are followed.

When equipment is needed for a test that is outside of permanent control of the laboratory, the lab ensures the equipment meets the

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requirements of this manual prior to its use by inspecting or otherwise testing it.

Routine testing work is completely discontinued on equipment that even shows minor nonconformance. Not only do we do this for ethical reasons in support of our customer, but also minor nonconformance is often indicative of major breakdowns in expensive equipment. These breakdowns need to be avoided wherever possible.

Out of service equipment is clearly marked.

The laboratory examines the effect of the defect or departure from specified limits on previous test and/or calibrations and institutes the "Control of Nonconforming Testing" procedure as outlined in section 12.

Each item of equipment and the software used for testing and significant to the results is uniquely identified and records of equipment and software are maintained. This information includes the following:

a) identity of the equipment and its software;
b) manufacturer's name, type identification, serial number or other unique identifier;
c) checks that equipment complies with specifications of applicable tests;
d) current location;
e) manufacturer's instructions, if available, or a reference to their location;
f) dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration;
g) maintenance plan where appropriate, and maintenance carried out to date; documentation on all routine and non-routine maintenance activities and reference material verifications;
h) any damage, malfunction, modification or repair to the equipment;

i) date received and date placed into service (if available); and

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j) condition when received, if available (new, used, reconditioned).

k) measuring and testing equipment is uniquely identified through an asset number/identification.

20.2 Support Equipment

SUPPORT EQUIPMENT includes, but is not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, volumetric dispensing devices, and thermal/ pressure sample preparation devices.

POLICY

All support equipment is maintained in proper working order and records are kept of all repair and maintenance activities, including service calls.

PROCEDURE

All raw data records are retained to document equipment performance. These records include logbooks, data sheets, or equipment computer files.

All support equipment is calibrated or verified annually over the entire range of use using NIST traceable references where available. The results the calibration of support equipment are within specifications or (1) the equipment is removed from service until repaired, or (2) records are maintained of correction factors to correct all measurements.

Support equipment such as balances, ovens, refrigerators, freezers, and water baths are checked with a NIST traceable reference if available, each day prior to use, to ensure they are operating within the expected range for the application for which the equipment is to be used.

Mechanical volumetric dispensing equipment, including burettes (except Class A glassware), is checked for accuracy quarterly.

Glass micro-liter syringes have a certificate attesting to the established accuracy. If the certificate of accuracy for glass micro-liter syringes is

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not available, the accuracy of the syringe is demonstrated upon receipt and documented.

For chemical tests that use autoclaves, the temperature, cycle time, and pressure is documented by use of chemical indicators or temperature recorders and pressure gauges.

For microbiology analyses, records for autoclaves used in the laboratory are required for the following:

- initial performance of the autoclave functional properties (supplier by the installer);
- temperature demonstration of sterilization continuous monitoring device or maximum registering temperature;
- for every cycle, record date, contents, maximum temperature reached, pressure, time in sterilization mode, total run time, and analysts initials;
- quarterly check of autoclave timing device against a stopwatch; and
- annual maintenance check to include a pressure check and calibration of temperature device.

20.2.1 Support Equipment Maintenance

Regular maintenance of support equipment, such as balances and fume hoods is conducted at least annually. Maintenance on other support equipment, such as ovens, refrigerators, and thermometers is conducted on an as needed basis.

Records of maintenance to support equipment are documented in Instrument Maintenance Logs. Each piece of support equipment does not necessarily have its own logbook. Maintenance logbooks may be shared with equipment that is housed in the same laboratory area.

20.2.2 Support Equipment Calibration

Calibration requirements for analytical support equipment are found in the in the laboratory procedure for support equipment.

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POLICY

Equipment requiring calibration is labeled to indicate the calibration status and/or operational status and the date when re-calibration is due when appropriate.

Measuring equipment that has failed calibration or is deemed out of service is labeled with an out of service tag.

POLICY

When equipment goes outside the direct control of the laboratory for a period, the laboratory ensures that the function and calibration status of the equipment are checked and validated and shown to be satisfactory before the equipment is returned to service.

PROCEDURE

The procedures used to check and ensure that the function and calibration status of the equipment are satisfactory before the equipment is returned to service are outlined in the manufacturer's equipment manual. Any additional quality control checks are outlined in the "Quality Control Plan" section of the appropriate test method.

20.3 Analytical Equipment

20.3.1 Maintenance for Analytical Equipment

POLICY

All equipment is properly maintained, inspected, and cleaned.

PROCEDURE

Maintenance of analytical instruments and other equipment may include regularly scheduled preventive maintenance or maintenance on an as-needed basis due to instrument malfunction and is documented in Instrument Maintenance Logs, which become part of the laboratory's permanent records.

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20.3.2 Initial Instrument Calibration

Initial instrument calibration and continuing instrument calibration verification are an important part of ensuring data of known and documented quality. If more stringent calibration requirements are included in a mandated method or by regulation, those calibration requirements override any requirements outlined here or in laboratory SOPs. Generally, instrument calibrations are provided in test methods.

POLICY

All initial instrument calibrations are verified with a standard obtained from a second source traceable to a national standard when commercially available. If a second source is not available, a standard prepared from a separate lot may be used as long as the manufacturer can demonstrate the lot was prepared independently from other lots purchased.

POLICY

If the reference or mandated method does not specify the number of calibration standards to use, the minimum number is five not including blanks or a zero standard.

POLICY

Any samples that are analyzed after an unacceptable initial calibration are re- analyzed or the data are reported with qualifiers, appropriate to the scope of the unacceptable condition.

POLICY

Quantitation is always determined from the initial calibration unless the test method or applicable regulations require quantitation from the continuing calibration.

POLICY

The lowest calibration standard is the lowest concentration for which quantitative results can be reported without qualification. The lowest calibration standard is equal to the Limit of Quantitation and is greater than the limit of detection.

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POLICY

The highest calibration standard is the highest concentration for which quantitative results can be reported.

POLICY

Data reported that are greater than the highest calibration standard without dilution are considered to be an estimate and are reported with a qualifier code and explained in the case narrative.

PROCEDURE

Initial instrument calibration includes calculations, integrations, acceptance criteria, and associated statistics referenced in the test method SOP.

Sufficient raw data records are collected to allow reconstruction of the initial instrument calibration. These include, at a minimum, calibration date, test method, instrument, analysis date, analyte names, analysts signature or initials, concentration and response, calibration curve or response factor, or unique equation or coefficient used to reduce instrument responses to concentration. Calibration date and expiration date (when recalibration is due) is recorded for equipment requiring calibration, where practicable.

Acceptance criteria are listed in individual method SOPs.

Corrective actions are performed when the initial calibration results are outside acceptance criteria. Calibration points are not dropped from the middle of the curve unless the cause is determined and documented. If the cause cannot be determined, the calibration curve is re-prepared. If the low or high calibration point is dropped from the curve, the working curve is adjusted and sample results outside the curve are qualified.

Results that are less than the lower calibration standard are considered to have increased uncertainty, and are either reported with a qualifier code or explained in the case narrative.

Results that are greater than the highest calibration standard are either diluted to within the calibration range, or considered to be an

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estimate; and are reported with a qualifier code and explained in the case narrative

20.3.3 Continuing Instrument Calibration

POLICY

The validity of the initial calibration is verified prior to sample analysis by use of a continuing instrument calibration verification (CCV) standard.

POLICY

Corrective action is initiated for continuing instrument calibration verification results that are outside of acceptance criteria.

PROCEDURE

Continuing instrument calibration verification is performed at the beginning and end of each analytical batch, except for instances when an internal standard is used. For methods employing internal standards, only one verification is performed at the beginning of the analytical batch.

Continuing instrument calibration verification is performed whenever it is expected that the analytical system may be out of calibration or might not meet verification acceptance criteria.

Continuing instrument calibration verification is performed when the time period for calibration or the most recent calibration verification has expired.

Continuing instrument calibration verification is performed for all analytical systems that have a calibration verification requirement.

Calibration is verified for each compound, element, or other discrete chemical species.

The calculations and associated statistics for continuing instrument calibration are included or referenced in the test method SOP.

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Sufficient raw data records are retained to allow reconstruction of the continuing instrument calibration verification. Continuing instrument calibration verification records connect the continuing verification date to the initial instrument calibration.

Acceptance criteria for continuing instrument calibration are listed in individual method SOPs.

20.3.4 Unacceptable Continuing Instrument Calibration Verifications

If routine corrective action for continuing instrument calibration verification fails to produce a second consecutive (immediate) calibration verification within acceptance criteria, then a new calibration is performed or acceptable performance is demonstrated after corrective action with two consecutive calibration verifications.

For any samples analyzed on a system with an unacceptable calibration, some results may be useable if qualified and under the following conditions:

a) If the acceptance criteria are exceeded high (high bias) and the associated samples are below detection, then those sample results that are non-detects may be reported as non-detects.

b) If the acceptance criteria are exceeded low (low bias) and there are samples that exceed the maximum regulatory limit, then those exceeding the regulatory limit may be reported.

POLICY

When intermediate checks are needed to maintain confidence in the calibration status of equipment, these checks are carried out periodically according to defined procedure.

PROCEDURE

Internal quality control checks are specified in individual test methods that are located in the appropriate laboratory areas thereby providing procedures for intermediate checks.

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POLICY

Test and calibration equipment, including hardware and software, are safeguarded from adjustments that invalidate test and/or calibration results/status.

PROCEDURE

Safeguards against adjustment for laboratory equipment include:

✐ Detailed SOPs and manufacturer's manuals on the operation of the equipment

- ✐ Policies permitting only fully trained and competent personnel to operate equipment
- ✐ Access to the laboratory is restricted to authorized personnel

Safeguards against adjustment for software include:

- ✐ Password protection for important files and packages
- ✐ Access to the laboratory is restricted to authorized personnel

SECTION 21 – MEASUREMENT TRACEABILITY

Measurement quality assurance comes in part from traceability of standards to certified materials.

POLICY

Test and/or calibration equipment for subsidiary measurements (e.g., for environmental conditions) having a significant effect on the accuracy or validity of the result of the test, calibration, or sampling are calibrated and/or verified before being put into service.

PROCEDURE

The calibration program includes a system for selecting, using, calibrating, checking, controlling, and maintaining:

- ✐ Measurement standards
- ✐ Reference standards used as measurement standards

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- ✐ Measuring and test equipment used to perform tests and calibrations

Procedures are documented where appropriate. All measurements that play a defining role in testing accuracy are based directly or indirectly on reference standards, reference materials, certified reference materials, or other standards or materials having appropriate traceability.

POLICY

The program for calibration equipment is designed and operated to ensure that calibration measurements are traceable to the System International (SI) units of measurement.

PROCEDURE

All equipment that affects the quality of test results are calibrated according to the minimum frequency suggested by the manufacturer, by regulation, by method, or as needed.

Clients can verify that required uncertainty is achieved by reviewing the internal quality control data on the test reports.

PROCEDURE

Traceability of measurement is assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability. The calibration certificates issued by these laboratories show that there is a link to a primary standard or to a natural constant realizing the SI unit by an unbroken chain of calibrations. The calibration certificates contain the measurement results including the measurement uncertainty and/or a statement of compliance with an identified metrological specification.

Maintain certificates of all reference standards, measuring equipment, or certified reference material used in ensuring traceability. Where traceability to national standards of measurement is not applicable, the laboratory provides satisfactory evidence of correlation of results, for example by participation in a suitable program of inter-laboratory comparisons or proficiency testing.

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Reference standards, such as thermometers and weights, are traceable to a national or international standard (e.g., NIST).

POLICY

The traceability requirements apply to measuring and test equipment with measuring functions used, unless it has been established that the associated calibration uncertainty contributes little to the total uncertainty of the test result. When this situation arises, the laboratory ensures that equipment used can provide the accuracy of measurement needed.

PROCEDURE

The extent to which the traceability requirements are followed depends on the relative contribution of calibration uncertainty to the total uncertainty. If calibration is the dominant factor, the requirements are strictly followed. If, however, calibration is not one of the major contributors to the total uncertainty, other ways for providing confidence may be used.

POLICY

Where traceability to SI units of measurement is not possible and/or not relevant, other means for providing confidence in the results are applied:

- The use of suitable reference materials certified to give a reliable characterization of the material
- Mutual-consent standards or methods which are clearly specified and agreed upon by all parties concerned
- Participation in a suitable program of inter-laboratory comparisons or proficiency testing

PROCEDURE

Reliable characterization involves an estimate of recovery.

The laboratory participates in proficiency testing and/or check sample programs. The list of programs is maintained by the Laboratory Director or Technical Manager. .

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21.1 Reference Standards

REFERENCE STANDARDS are standards of the highest quality available at a given location, from which measurements are derived.

POLICY

Reference standards are obtained or calibrated by a body that can provide traceability to national or international standards. Such reference standards

of measurement held by the laboratory are used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

PROCEDURE

Reference standards are obtained from the National Institute of

Standards and Technology (NIST), if applicable.

The following reference standards are sent out to be calibrated to a national standard:

a) Class 1 weights are sent out for calibration every year.
b) Reference thermometers are sent out for calibration every

year.

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21.2 Reference Materials

REFERENCE MATERIALS are substances that have concentrations that are sufficiently well established to use for calibration or as a frame of reference.

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POLICY

Reference materials, where commercially available, are traceable to national standards of measurement, or to Certified Reference Materials, usually by a Certificate of Analysis.

POLICY

Internal reference materials, such as working standards or intermediate stock solutions, are checked as far as technically and economically possible.

PROCEDURE

Purchased Reference Materials require a Certificate of Analysis where available. Otherwise, purchased reference materials are verified by application to a certified reference material, interlaboratory comparison, and/or demonstration of capability.

Internal Reference Materials, such as working standards and intermediate stock solutions, are checked with secondary standards. a) Internal thermometers are checked annually against the NIST certified reference thermometer.

b) Working class weights are checked against Class 1 weights yearly.
c) Class A pipettes are verified for accuracy gravimetrically twice a year.

POLICY

Checks needed to maintain confidence in the calibration status of reference, primary, transfer or working standards and reference materials are carried out according to defined procedures and schedules.

PROCEDURE

The control check standards used to verify the accuracy of all the other standards are prepared independently from all the other standards used to establish the original calibration.

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These control check standards are preferably prepared from a separate lot # or source. It is the responsibility of the Supervisor to establish and maintain the individual schedule for each SOP and/or test method.

. 21.3 Transport and Storage of Reference Standards and Materials

POLICY

The laboratory handles and transports reference standards and materials in a way that protects their integrity.

PROCEDURE

Reference standard and material integrity is protected by separation from incompatible materials and/or minimizing exposure to degrading environments or materials. Reference standards and materials are stored according to manufacturer's recommendations and separately from working standards or samples.

Additional procedures may be necessary when reference standards and reference materials are used outside the permanent laboratory for tests, calibrations, or sampling. Proper conditions are established for housing, handling, and care of reference standards/reference materials. All information needed to properly identify references appears on their housing or containers.

. 21.4 Labeling of Reference Standards, Reagents, and Materials

POLICY

Reference standards and materials are tracked from purchase, receipt, and storage through disposal.

POLICY

Expiration dates can be extended if the reference standard or material's integrity is verified.

POLICY

Reagent quality is verified upon receipt using the Certificate of Analysis.

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PROCEDURE

Records for all standards, reagents, reference materials, and media include:

1. The manufacturer/vendor name (or traceability to purchased stocks or neat compounds)
2. The manufacturer's Certificate of Analysis or purity(if supplied)
3. The date of receipt
4. Reference to the method of preparation
5. Date of preparation
6. Recommended storage conditions
7. An expiration date after which the material shall not be used.
8. preparer's initials (if prepared)

In methods where the purity of reagents is not specified, analytical reagent grade is used. If the purity is specified, that is the minimum acceptable grade. Purity is verified and documented according to Section 9, Purchasing, Services, and Supplies.

All containers of standards, reagents, or materials, whether original or prepared, are labeled with an expiration date.

All containers of prepared standards and reference materials have a preparation date and unique identifier. This laboratory uses LIMS and a standard preparation logbook.

Standard preparation records are kept in laboratory notebooks and indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date, and preparer's initials.

Prepared reagents are verified to meet the requirements of the test method through internal QC measures.

POLICY

All Reference standards, reagents, and materials are inspected upon receipt.

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The complete traceability of all laboratory critical supplies is imperative. The unique ID number must be recorded on every chemical, in the appropriate logbook, and all paperwork to be filed.

The unique laboratory number shall be used to refer to the chemical throughout the laboratory. All logbook references will use it and, therefore, it will be possible to track the reagent from receipt to complete use or expiration and disposal.

The laboratory shall use the chemicals in the order of receipt. The sequential numbering will aide in this task.

Any chemical not properly labeled shall not be used. It shall be brought to the attention of the Quality Manager immediately.

Any delivery not containing all appropriate paperwork, especially the certificate of analysis, shall be held after labeling. The Quality Manager shall be notified and attempt to contact the manufacturer. The chemical may not be put into storage or used until released by the Quality Manager.

SECTION 22 – SAMPLE MANAGEMENT

POLICY

The Laboratory Director or designee is responsible for the sampling plan and procedures for any laboratory sampling of substances, matrices, materials or products for subsequent testing or calibration. The sampling plan and procedures are available at the location where sampling is performed. Sampling plans are based on appropriate statistical methods whenever reasonable. The sampling process addresses the factors to be controlled to ensure validity of the test and calibration results.

22.1 Sample Receipt

POLICY

Where the customer requires deviations, additions or exclusions from the sampling procedure, these are recorded in detail with the appropriate sampling data and included in all documents containing

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test and/or calibration results, and communicated to the appropriate personnel.

PROCEDURE

When samples are received at the laboratory, their condition is documented, they are given unique identifiers, and they are logged into the sample tracking system.

The physical appearance and temperature of all test items is observed and recorded upon receipt. Any deviations from specifications or observations are discussed with the customer as to the suitability of the sample. Cross-contamination is the most critical issue from broken, leaking samples for both qualitative and quantitative tests.

POLICY

Upon receipt of the test, any abnormalities or departures from normal or specified conditions, as described in the relevant test method, are recorded. When there is any doubt as to the suitability of an item for test, or when an item does not conform to the description provided, or the test required is not specified in sufficient detail, the laboratory consults the customer for further instructions before proceeding and keeps a record of the discussion.

PROCEDURE

Conform to applicable regulations or contractual arrangements. The condition of sample may include or relate to damage, quantity, preparation, packaging, or temperature. Preparation may include addition of chemical preservative, removal of moisture, and isolation of portion of sample to be tested, homogenization, or sub-sampling.

Procedure is in place to ensure that elapsed time between sampling and testing does not exceed test method specifications (holding time).

22.2 Sample Acceptance

POLICY

The minimum conditions a sample must meet on receipt are designated in the sample acceptance policy and procedure. If the

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required conditions are not met, the client is contacted prior to any further processing.

The sample acceptance policy is available to sample collection personnel, and emphasizes the need for use of water resistant ink, use of appropriate containers, adherence to holding times, sample volume requirements, and what to do with compromised samples.

Sample submission sheets (Chain of Custody) from the field are maintained with the samples. Upon receipt in the laboratory, chain of custody is scanned and filed using State Of Alaska adopted chain of custody software.

If the checks performed upon sample receipt indicate the criteria are not met, then 1) the sample is rejected as agreed with the client, 2) the decision to proceed is documented and agreed upon with the client, 3) the condition is noted on the Chain of Custody form and/or lab receipt documents, and 4) the data are qualified in the report.

22.3 Sample Identification

POLICY

Samples, including subsamples and extracts are uniquely identified in a permanent chronological record (such as a sample receipt log book or database) to prevent mix-up and to document receipt of all sample containers.

POLICY

Test items are systematically identified as they arrive at the laboratory. The identification is retained throughout the life of the item in the laboratory. The system is designed and operated so as to ensure that items cannot be confused physically, or when referred to in records or other documents. The system accommodates a sub-division of groups of items and the transfer of items within and from the laboratory when appropriate.

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PROCEDURE

Samples are assigned sequential numbers that reference more detailed information kept in sample receiving log.

The following information is collected in the chain of custody.

a) Client or project name
b) Date and time of sampling
c) Date and time of receipt at lab
d) Unique laboratory identification number
e) Unique field identification number (may be same as lab #)
f) Initials of recorder
g) Analyses requested
h) Comments regarding rejection (if any).

Sample labelling indicates the unique identification and conforms to applicable legal requirements. Where conformity of possession of a test sample must be maintained for other purposes, the laboratory establishes and documents a system for appropriate chain-of-custody (samples may be used in a court of law for evidentiary purposes).

22.4 Sample Storage

POLICY

The sample storage procedure defines the appropriate facilities for avoiding deterioration, loss or damage to the test item during storage, handling and preparation and testing; instructions provided with the item are followed. When items have to be stored or conditioned under specified environmental conditions, these conditions are maintained, monitored, and recorded. Where a test item is to be held secure (e.g., for reasons of record, safety or value, or to enable complementary test to be performed later), the laboratory has arrangements for storage and security that protect the condition and integrity of the secured item concerned.

PROCEDURE

Where test items are to be returned into service after testing

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(e.g., for non-destructive testing or human beings subject to medical tests), special care is required to ensure that they are not damaged or injured during the handling, testing or storing/ waiting processes. A sampling procedure and information on storage and transport of samples, including all information that may influence the test or calibration result, is provided to those responsible for taking and transporting the samples.

The laboratory establishes whether the sample has received all necessary preparation or whether the customer requires preparation to be undertaken or arranged by the laboratory. Proper requirements for packaging, environmental conditions, and separation from incompatible materials are observed. Where samples have to be stored or conditioned under specific conditions, these conditions are maintained, monitored, and recorded, where necessary.

Where a sample, or portion of a sample, is to be held secure (e.g., for reasons of record, safety, or value, or to enable check tests to be performed later), the laboratory has storage and security arrangements that protect the condition and integrity of the sample.

Storage conditions are monitored for any required criteria, verified, and the verification recorded in logbooks. This includes temperature monitoring and holding times.

Samples are held secure, as required. Samples are stored apart from standards, reagents, food or potentially contaminating sources, and such that cross-contamination is minimized. All portions of samples, including extracts, leachates, or any product of the sample is maintained according to the required conditions.

22.5 Sample Disposal

POLICY

Samples are disposed of according to Federal, State and local regulations. Procedures are available for the disposal of samples, leachates, and extracts. All samples will be rendered useless and sent to a landfill unless prohibited by federal, state of local regulations.

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22.6 Sample Transport

POLICY

Samples that are transported under the responsibility of the laboratory, where necessary, are done so safely and according to storage conditions. This includes moving bottles within the laboratory. Specific safety operations are addressed outside of this document.

22.7 Sampling Records

POLICY

Sampling plans are based, whenever it is reasonable or requested by the client, on appropriate statistical sampling methods.

POLICY

The sampling policy outlines the procedures for recording relevant data and operations relating to sampling that forms part of the testing or calibration that is undertaken. These records include the sampling procedure used, the identification of the sampler, environmental conditions (if relevant) and any diagrams or other equivalent means to identify the sampling location as necessary, and, if appropriate, the statistics upon which the sampling procedures are based. Subsampling within the laboratory is performed according to test method SOPs.

Relevant sampling data are recorded, including the sampling procedure used, 2) the identification of the sampler, 3) environmental conditions (if relevant), 4) the sampling location, and 5) the statistics upon which the sampling procedures are based.

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SECTION 23 – QUALITY OF TEST RESULTS

23.1 Essential Quality Control Procedures

POLICY

All essential quality control elements are collected and assessed on a continuing basis.

POLICY

The qualities of test results are recorded in such a way that trends are detectable, and where practicable, are statistically evaluated.

POLICY

For test methods that do not provide acceptance criteria for an essential quality control element or where no regulatory criteria exist, acceptance criteria are developed. Control limits are developed using the mean, plus or minus 3 standard deviations; or static limits such as +/- 15 percent. These limits can be found in Control Charts in the Quality Manager's office.

POLICY

Quality control procedures are utilized to monitor the validity of test results. These procedures are for each test method utilized in the laboratory. The resulting data are recorded so that trends are detectable (and where practicable, statistical techniques are applied to the reviewing of the results). This monitoring is planned and reviewed and may include, but not limited to, the following:

a) Regular use of certified reference materials and/or internal quality control using secondary reference materials
b) Participation in inter-laboratory comparisons or proficiency testing programs
c) Replicate tests or calibrations using the same or different methods

d) Re-testing or re-calibration of retained items
e) Correlation of results for different characteristics of an item The Quality Manager maintains a list of all the current proficiency testing programs the laboratory participates in, monitors the results, and notifies the appropriate personnel of both problematic and successful results.

Written procedures to monitor quality controls including acceptance criteria are located in the test method SOPs, except where noted, and include such procedures as:

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a) use of laboratory control samples and blanks to serve as positive and negative controls for chemistry methods;
b) use of laboratory control samples to monitor test variability of laboratory results;
c) use of calibrations, continuing calibrations, certified reference materials and/or PT samples to monitor accuracy of the test method;
d) measures to monitor test method capability, such as limit of detection, limit of quantitation, and/or range of test applicability, such as linearity;
e) use of regression analysis, internal/external standards, or statistical analysis to reduce raw data to final results;
f) use of reagents and standards of appropriate quality;
g) procedures to ensure the selectivity of the test method;
h) measures to assure constant and consistent test conditions such as temperature, humidity, rotation speed, etc., when required by test method;
i) use of sterility checks for equipment, media and dilution water for microbiology; and

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j) use of positive and negative culture controls for microbiology.

23.2 Internal Quality Control Practices

Analytical data generated with QC samples that fall within prescribed acceptance limits indicate the test method is IN CONTROL.

QC samples that fall outside QC limits indicate the test method is OUT OF CONTROL (non- conforming) and that corrective action is required or that the data are qualified.

POLICY

Detailed QC procedures and QC limits are included in text method standard operating procedures (SOPs), or where unspecified in the SOPs, are detailed elsewhere.

POLICY

Quality control data are analyzed and, where they are found to be outside pre-defined criteria, planned action is taken to correct and to prevent incorrect results from being reported.

POLICY

All QC measures are assessed and evaluated on an on-going basis, so that trends are detected.

PROCEDURE

The following general controls are used:

Positive and Negative Controls such as:

a) Blanks(negative)

b) Laboratory control sample (positive)
c) Reference Toxicants (positive)
d) Sterility checks and control cultures (positive and negative).

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Selectivity is assured through:

a) absolute and relative retention times in chromatographic analyses;
b) two-column confirmation when using non-specific detectors;
c) use of acceptance criteria for mass spectral tuning (found in test method SOPs);
d) use of the correct method according to its scope assessed during method validation; and
e) use of reference cultures (positive and negative) from a recognized manufacturer (where applicable).

Consistency, Variability, Repeatability, and Accuracy are assured through:

b) proper installation and operation of instruments according to manufacturer's recommendations or according to the processes used during method validation;
c) monitoring and controlling environmental conditions (temperature, access, proximity to potential contaminants);
d) selection and use of reagents and standards of appropriate quality;
e) cleaning glassware appropriate to the level required by the analysis. Cleaning procedures not provided in test method SOPs are provided in a separate SOP. For microbiology, glassware care includes use of borosilicate glassware, use of detergents designed for laboratory use, testing each day for alkaline or acid residue with bromothymol blue, and conduct of the Inhibitory Residue test when the detergent is changed.
f) following SOPs and documenting any deviation, assessing for impact, and treating data appropriately;
g) testing to define the variability and/or repeatability of the laboratory results, such as replicates;
h) use of measures to assure the accuracy of the test method, including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

.

i) use of duplicate plate counts on positive samples (microbiology only).

Acceptance or rejection criteria are created according to laboratory policy where no method or regulatory criteria exist. Acceptance criteria define the boundary for the appropriate response from laboratory personnel, such as corrective action, reporting with qualifiers, reanalysis, review, and others.

Test Method Capability is assured through:

a) establishment of the limit of detection where appropriate;
b) establishment of the limit of quantitation or reporting level; and/or
c) establishment of the range of applicability such as linearity;

Data reduction is assured to be accurate by:

a) selection of appropriate formulae to reduce raw data to final results such as regression;
b) periodic review of data reduction processes to assure applicability;
c) microbiological calculations, data reduction, and statistical interpretations specified by each test method.

23.3 Method Blanks

POLICY

Contaminated blanks are identified according to the acceptance limits in the test method SOPs or laboratory documentation.

POLICY

Samples associated with a contaminated blank are evaluated as to the appropriate corrective action for the samples (e.g. reprocessing or data qualifying codes).

PROCEDURE

Our laboratory identifies a blank as contaminated when analyte results are greater than the reporting limit and greater than 1/10 of that found in any sample, or where the contamination affects the sample results according to test method requirements or client objectives.

When a blank is determined to be contaminated, the cause must be investigated and measures taken to minimize or eliminate the problem.

Data that are unaffected by the blank contamination (non-detects or other analytes) are reported unqualified.

Sample data that are suspect due to the presence of a contaminated blank are reanalyzed, qualified, or deleted.

23.4 Laboratory Control Samples

LABORATORY CONTROL SAMPLES (LCS) are prepared from analyte free water, and spiked with verified and known amounts of analytes for the purpose of establishing precision or bias measurements.

POLICY

Laboratory control samples are analyzed at a frequency mandated by method, regulation, or client request, whichever is more stringent.

PROCEDURE

The results of laboratory control samples (LCS) are calculated in percent recovery or other appropriate statistical technique that allowscomparison to established acceptance criteria. The laboratory documents the calculation in the test method SOPs. The individual LCS is compared to the acceptance criteria as published in the mandated test method, or where there are no established criteria, the laboratory establishes limits.

23.5 Matrix Spikes and Matrix Spike Duplicates

MATRIX SPIKES (MS and MSD [duplicates]) are samples fortified with a known amount of analyte to help assess the effect of the matrix on method performance.

POLICY

The MS/MSD results are used to help assess the effect of the sample matrix on method performance.

PROCEDURE

The laboratory procedure for MS/MSD includes spiking appropriate analytes at appropriate concentrations, calculating percent recoveries and relative percent difference (RPD), and evaluating and reporting the results.

Where there are no established criteria, the laboratory uses the mean plus or minus three standard deviations as the control limits for MS/MSD.

For MS/MSD results outside established criteria corrective action is documented or the data are reported with appropriate data qualifying codes.

23.6 Surrogate Spikes

SURROGATES are substances with chemical properties and behaviors similar to the analytes of interest used to assess method performance in individual samples.

POLICY

Surrogates are added to all samples (in test methods where surrogate use is appropriate) prior to sample preparation or extraction.

PROCEDURE

Surrogate recovery results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory uses the mean plus or minus three standard deviations as surrogate control limits.

For surrogate results outside established criteria, data are evaluated to determine the impact. Corrective actions include qualifying the data as appropriate.

23.7 Proficiency Test Samples or Interlaboratory Comparisons

POLICY

The laboratory participates in proficiency test (PT) samples twice per year.

POLICY

The laboratory institutes corrective action procedures for failed PT samples.

POLICY

The laboratory does not share PT samples with other laboratories, does not communicate with other laboratories regarding current PT sample results, and does not attempt to obtain the assigned value of any PT sample from the PT provider.

PROCEDURE

Proficiency Testing (PT) or Proficiency Evaluation (PE) samples are treated as typical samples in the normal production process where possible, including the same preparation, calibration, quality control and acceptance criteria, sequence of analytical steps, number of replicates, and sample log-in. PT samples are not analyzed multiple times unless routine samples are analyzed multiple times.

23.8 Data Review

POLICY

The laboratory reviews all data generated in the laboratory for compliance with method, laboratory and, where appropriate, client requirements.

POLICY

All data review is documented.

Procedure

Initially, the analyst reviews data for acceptability of quality control measures and accuracy of the final result(s).

After the initial review, a second review is performed by either a Supervisor or Department Manager in the respective laboratory department, or by a peer analyst trained in the procedure that was used to produce the data batch. The second reviewer considers all manual transfers and calculations of data in detail and spot checks all electronic transfers of data. Final reports are compared to raw data either directly or through several reviewed steps.

SECTION 24 – REPORTING OF RESULTS

POLICY

The result of each test carried out is reported accurately, clearly, unambiguously, and objectively and complies with all specific instructions contained in the test method.

POLICY

Data are reported without qualification if they are greater than the lowest calibration standard, lower than the highest calibration standard, and without compromised sample or method integrity.

24.1 Test Reports

POLICY

The report format has been designed to accommodate each type of test performed and to minimize the potential for misunderstanding or misuse.

POLICY

Test reports are issued as electronic data (format) transfer or hard copy if requested.

POLICY

The testing laboratory does not issue calibration certificates. However, the laboratory often receives calibration services from a calibration laboratory and needs to be familiar with the information on a calibration certificate. A calibration certificate shall not contain any recommendation on the calibration interval except where this has been agreed with the customer or it is to be used by the laboratory itself.

PROCEDURE

Each test report generated contains the following information:

a) a title, such as Test Report or Test Results;
b) the name and address of the laboratory, the location of the laboratory if different from the address, and the phone number and name of a contact person;
c) unique identification of the test report on each page and a pagination system that ensures that each page is recognized as part of the test report and a clear identification of the end of the report, such as 3 of 10;
d) the name and address of the client;
e) the identification of the test method used;
f) an unambiguous identification of the sample(s), including the client identification code;
g) the date of sample receipt when it is critical to the validity and application of the results, date and time of sample collection, dates the tests were performed, the time of sample preparation and analysis if the required holding time for either activity is less
    a. than or equal to 72 hours;
h) reference to the sampling plan and procedures used by the laboratory where these are relevant to the validity or application of the results;
i) the test results with failures identified, units of measurement, an indication of whether results are calculated on a dry weight or wet weight basis;
j) the name, function, and signature or an equivalent electronic identification of the person authorizing the test report, and the date of issue;

k) a statement to the effect that the results relate only to the samples;
l) at the laboratory's discretion, a statement that the report shall not be reproduced except in full without written approval of the laboratory; m) certification that the results are in compliance with the TNI Standards if accredited to be in compliance or provide reasons and/or justification if they do not comply.

24.2 Supplemental Test Report Information

When necessary for interpretation of the results or when requested by the client, test reports include the following additional information:

a) deviations from, additions to, or exclusions from the test method, information on specific test conditions, such as environmental conditions, and any non-standard conditions that may have affected the quality of the results, and any information on the use and definitions of data qualifiers;
b) a statement of compliance/non-compliance when requirements of the quality systems are not met, including identification of test results that did not meet TNI sample acceptance requirements, such as holding time, preservation, etc.;
c) where applicable and when requested by the client, a statement on the estimated uncertainty of the measurement;
d) where appropriate and needed, opinions and interpretations

When opinions and interpretations are included, the basis upon which the opinions and interpretations are documented. Opinions and interpretations are clearly marked as such in the test report.

Opinions and interpretations included in a test report may comprise, but not be limited to the following:

- Opinion on conformity of the results with requirements
- Fulfilment of contractual requirements
- Recommendations on how to use the results
- Guidance to be used for improvements

In many cases it is appropriate to communicate the opinions and interpretations by direct dialogue with the customer. This dialogue is written down in the Case Narrative of the report. additional information which may be required by specific methods or client may include qualification of results with values outside the working range.

For test reports that contain the results of sampling, the following is provided if necessary for the interpretation of the results:

a) the date of sampling;
b) unambiguous identification of the material sampled;
c) the locations of the sampling, including diagrams, sketches, or photographs;
d) a reference to the sampling plan and procedures used;
e) details of any environmental conditions during sampling that may affect the interpretations of the test results;
f) any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

24.3 Testing Obtained from Subcontractors

Subcontractors must be fully licensed and accredited by the State of Alaska and local Test governments. Subcontractors will only be used with express write consent of the State of Alaska. Results obtained from test performed by subcontractors are clearly identified on the test report by subcontractor name and/or accreditation number.

The test results from subcontractors are reported in writing or electronically. A copy of the subcontractors report is be made available to the client if requested.

24.4 Electronic Transmission of Results

POLICY

All test results transmitted by telephone, fax, telex, e-mail, or other electronic means comply with the requirements of this

Quality Manual.

POLICY

Electronic signatures are stored on a private drive accessible to Laboratory Management only. The Quality Manager maintains a hard copy of signatures of all employees for verification.

PROCEDURE

The Quality Manager is responsible for obtaining and tracking signatures and initials of all employers.

Employees' electronic signatures are saved on a private drive as a pdf file which are only accessible by the Laboratory Management.

The permission to use signatures on the reports is released by the Quality Manager and Technical Manager.

24.5 Amendments to Test Reports

POLICY

Material amendments to a test report after it has been issued are made only in the form of another document or data transfer, which includes the statement "Supplement to Test Report, serial number... [or as otherwise identified]", or an equivalent form of wording. Such amendments meet all the requirements in this Quality Manual.

PROCEDURE

Amended test reports are titled, "Supplement to Test Report Serial Number" or an equivalent form of wording to assure they can be differentiated from other test reports.

When it is necessary to issue a complete new report, the new report is uniquely identified and contains a reference to the original that it replaces.

Testing Procedures

Jessica Dreibelbis, Chief Executive Officer

Foreword

The testing of marijuana for (1) THC, THCA, CBD, CBDA and CBN potency; (2) harmful microbials including Escherichia coli (E. Coli) or salmonella; (3) residual solvents; (4) poisons or toxins; (5) harmful chemicals; (6) dangerous molds, mildew, or filth; (7) pesticides is new to the State of Alaska.

South East Alaska Laboratories (SEAL) intends to be the first testing laboratory in South East Alaska and one of the first three laboratories in the state to begin testing. The testing requirements are codified in Title 3, section 306 of the Alaska Administrative Codes. Specifically, the code states:

1. Approval of testing facility. (a) A person seeking a marijuana testing facility license must first obtain the approval of the board by showing competence to perform each test the licensee will offer as an independent third-party testing facility, including tests to identify (1) THC, THCA, CBD, CBDA and CBN potency; (2) harmful microbials including Escherichia coli (E. Coli) or salmonella; (3) residual solvents; (4) poisons or toxins; (5) harmful chemicals; (6) dangerous molds, mildew, or filth; (7) pesticides. SEAL looks forward to future communications with the board to show our competence to perform these tests.

3 AAC 306 also states:

Specific tests and procedures

The tests required for each marijuana type or marijuana product, are as follows:

1. Potency Testing

Potency testing is required on marijuana bud and flower, marijuana concentrate, and a marijuana product, as follows: (A) the required cannabinoid potency test must at least determine the concentration of THC, THCA, CBD, CBDA and CBN cannabinoids; a marijuana testing facility may test and report results for any additional cannabinoid if the test is conducted in compliance with a validated method; (B) a marijuana testing facility shall report potency test results as follows: (i) for a potency test on marijuana and marijuana concentrate, marijuana testing facility shall list for each required cannabinoid a single percentage concentration that represents an average of all samples within the test batch; alternatively, the sum of THC and THCA may be reported as total THC; the sum of CBD and CBDA may be reported as total CBD; (ii) for a potency test on a marijuana product, whether conducted on each individual production lot or using process validation, marijuana testing facility shall list for each cannabinoid the total number of milligrams contained within a single retail marijuana product unit for sale;

(iii) for testing whether the THC content is homogenous, the marijuana testing facility shall report the THC content of each single serving in a multi-unit package; the reported content must be within 20 percent of the manufacturer's target; for example, in a 25 milligrams total THC package with five servings, each serving must contain between four and six milligrams of THC;

The marijuana testing facility shall determine an edible marijuana product to have failed potency testing if (i) an individually packaged edible retail marijuana product contained within a test lot is determined to have more than 50 milligrams of THC within it; or (ii) the THC content of an edible marijuana product is not homogenous;

2. Microbial testing
microbial testing for the listed substances on the listed marijuana products is required as follows: Substance Acceptable Limits Per Gram Product to be Tested –

1.1. Shiga-toxin producing Escherichia coli (STEC)- Bacteria less than 1 colony forming unit (CFU/g) flower; retail marijuana Products; water- and food-based concentrates

1.2. Salmonella species – bacteria less than 1 colony forming unit (CFU/g)

1.3. Aspergillus fumigatus, Aspergillus flavus, Aspergillus niger - fungus less than 1 colony forming unit (CFU/g)

3. Residual Solvent Testing
Testing for the listed residual solvents and metals on the listed marijuana products is required as follows:

3.1Substance Acceptable Limits Per Gram Product to be Tested Butanes less than 800 parts per million (PPM) solvent-based concentrates Heptanes less than 500 parts per million (PPM) Benzene less than .025 parts per million (PPM) Toluene less than 1 parts per million (PPM) Hexane less than 10 parts per million (PPM) Total Xylenes (meta-xylenes, para-xylenes, or ortho-xylenes) less than 1 parts per million (PPM)

This manual will detail how South East Alaska Laboratories intends to meet the state's requirements. Specifics such as flow rate, pressure settings, etc., have been intentionally omitted as these will depend on the specific availability of separation columns and are individually machine/unit specific.

Specifically, the State of Alaska requires the following tests:

Manual
(a) An applicant for a marijuana testing facility license and a licensed marijuana testing facility shall have a written manual of standard operating procedures with detailed instructions explaining how to perform each testing method the applicant or marijuana testing facility uses and minimum standards for each test. The written manual of standard operating procedures must be available to each employee of the marijuana testing facility at all times. The written manual of standard operating procedures must cover at least (1) sample preparation for each matrix that will be tested; (2) reagent, solution, and reference standard preparation; (3) instrument setup, if applicable; (4)

standardization of volumetric reagent solutions, if applicable; (5) data acquisition; (6) calculation of results; (7) identification criteria; (8) quality control frequency; (9) quality control acceptance criteria; and (10) corrective action protocol. (b) The scientific director of a marijuana testing facility shall approve, sign, and date each standard operating procedure, and each revision to any standard operating procedure. Laboratory testing of marijuana and marijuana products.

HPLC for the determination of cannabinoid potency.

1.1 Overview

High Performance Liquid Chromatography (HPLC). This method (HPLC) is applicable to cannabis flower for the measurement of Δ9-THC, Δ9-THC-A, CBD, CBD-A and CBN cannabinoids. It is also applicable to "concentrates" although sample preparation (extraction of cannabinoids from the plant) is not necessary for concentrates. Finally, it is applicable to "edibles" except that sample preparation will vary depending on the edible. Extracting cannabinoids from a brownie, for example, will be a different process than extracting it from a hard candy.

Analysis of cannabinoid potency by HPLC

Revision
1.0

Effective Date
8/6/16

Prepared By

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APPROVED BY:

Laboratory Director	Date
Quality Assurance Officer	Date

1.0 Scope and Application

2.0 Summary

3.0 Demonstration of Capabilities

4.0 Limit of Detection

5.0 Procedure

6.0 Calculations

7.0 Quality Control

8.0 References

9.0 Appendix

1.0 Scope and Application

1.1. This method is used for the determination of cannabinoids in cannabis via High Performance Liquid Chromatography (HPLC). This method is applicable to cannabis flower for the measurement of Δ9-THC, Δ9-THC-A, CBD, CBD-A and CBN cannabinoids. Testing for additional cannabinoids via this method must first be validated appropriately, as well as any additional matrices. This method is applicable in the range of 0.2µg/mL to 100µg/mL.

2.0 Summary

High Performance Liquid Chromatography (HPLC). This method (HPLC) is applicable to cannabis flower for the measurement of Δ9-THC, Δ9-THC-A, CBD, CBD-A and CBN cannabinoids. It is also applicable to "concentrates" although sample preparation (extraction of cannabinoids from the plant) is not necessary for concentrates. Finally, it is applicable to "edibles" except that sample preparation will vary depending on the edible. Extracting cannabinoids from a brownie, for example, will be a different process than extracting it from a hard candy.

3.0 Demonstration of capability

The analysts must make an initial demonstration of capability and generate acceptable accuracy and precision with the method before analyzing any samples. Any changes or modifications to this SOP require the analyst to repeat the demonstration of capability.

A quality control sample shall be obtained from an outside source.

The four quality control samples shall be analyzed according to the test method either concurrently or over a period of days.

Run the aliquots after analyzing all required calibration standards. Ensure that the reagent blank does not contain any analyte at a concentration greater than the reporting limit (RL).

Using all of the results, calculate the mean recovery (x) and the Percent Relative Standard Deviation.

An acceptable DOC has a mean percent recovery between 80% and 120%. The relative percent standard deviation <20%.

4.0 Limit of Detection (LOD):

4.1 LOD's must be determined annually. Also, LOD's must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

4.2 A MB, LCS, DUP and CCV must be run with every batch. A batch includes at most twenty samples.

4.2.1 Method Blanks (MB)-Will be performed at a frequency of one per batch of samples per matrix type per extraction or preparation method. The results of this analysis will be one of the QC measures to be used to assess batch acceptance. The source of contamination must be investigated and measures taken to correct minimize or eliminate the problem if: 1) The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch or 2) The blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

4.2.2 Lab Control Sample (LCS)/Initial Calibration Verification (ICV) - Are performed at a frequency of one per batch of 20 or less of samples per matrix type, and sample preparation. The results of analysis are used to assess batch acceptance. The LCS must have a percent recovery of 80%-120% to be acceptable.

4.2.3 Sample Duplicates (DUP) - Are performed at a minimum of 1 in 20 samples per matrix type per sample preparation. The duplicate sample is chosen at random. The selected samples are rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. The RPD for water samples is ±10%.

4.4 Continuing Calibration Verification (CCV): After 10 samples and/or at the end of analysis run, prepare a CCV to verify calibration. The concentration must vary to verify different parts of the curve. The CCV must have a recovery of ±20% to be acceptable. If a CCV fails, any sample associated with the CCV must be re-analyzed.

4.5 If the MB or LCS fails, the analysis will be rerun.

4.6 If the MB and LCS pass but the DUP and/or MS fail, the data is reported with qualifiers.

4.7 All data must be recorded in the Cannabis % Moisture data logbook.

5.0 Procedure

5.1 Weigh 1 g of randomly sampled flower for each analysis into a desiccated, pre-tarred crucible. Dry the sample 24 hours at 35°C in a forced ventilation oven.

5.2 Weigh the dried sample again and record the dry weight.

5.3 Homogenize sample for 1 minute in the blender. Weigh out 200 mg of the ground, homogenized sample into a glass vial.

5.4 Bring the sample to 10 ml with 9:1 methanol:chloroform. Sonicate for 1 minute.

5.5 Filter 300µl of the sample through a syringe filter into an amber glass vial. Bring up to 1.5 ml volume with 9:1 methanol:chloroform.

5.6 Prepare MB, LCS, CCB, and CCV as described in the QC section. Add spike to MS and MSD sample as described above.

5.7 Enter samples into chromatography software and load autosampler in the appropriate order.

5.8 The standard compounds may be separated using a binary gradient in 12 minutes including equilibration time. The mobile phases are 10 mM aqueous ammonium formate and methanol. The flowrate is 1.5 mL/min. The program starts at 60% methanol, ramps to 75% at 6 minutes, to 95% at 9 minutes, immediately drops back to 60% at 9.01 minutes and re-equilibrates until 12 minutes. The separation takes place on a 3µ 50 mm X 4.6 mm C18 column at 40° C. A single wavelength at 228 nm is monitored for the analysis. Injections of 5 µL are used for standards and samples.

6.0 Calculations

6.1 Individual cannabinoid values are expressed as w/w %. Calculate total content of THC (THC_{tot}), CBD (CBD_{tot}) and CBG (CBG_{tot}), using formulae which adjusted for the differing molecular weight of the cannabinoid and carboxylic conjugative components of each cannabinoid [32]:

$$THCtot = THC + THC - A * (314.46/358.47)$$

$$CBDtot = CBD + CBD - A * (314.46/358.47)$$

$$CBGtot = CBG + CBG - A * (316.48/360.48)$$

7.0 Quality Control

All sample runs must be accompanied by a method blank (MB), laboratory control sample (LCS), a method spike and method spike duplicates (MS and MSD), and closing calibration blank (CCB) and closing calibration validation (CCV).

7.1 Method blank for this method shall consist of 10 ml of a 9:1 methanol/chloroform mixture added to a VOA vial. This mixture should be prepared fresh for each run under a chemical hood by pipetting 9ml of methanol into a labelled vial and adding 1 ml of chloroform to the vial. Prior to loading the autosampler, load an aliquot of 1.5 ml into a 2 ml autosampler vial.

7.2 The laboratory control sample (LCS) will be made daily from 1.0 mg/mL stock solutions obtained from Cerilliant. One stock solution contains Δ9-THC, cannabinol (CBN) and cannabidiol (CBD) (Catalog #T-108). Separate stock solutions for Δ9-THC-A (Catalog T-093), and CBD-A (Catalog C-144), also in 1.0 mg/mL will be used to make up the LCS. To preserve standard, samples will be loaded into a 300 µL low volume autosampler vial. Add 50 µL each of the T-108, T-093, and C-144 stock solutions and bring it up to volume (300 µL) with a 9:1 mixture of methanol and chloroform. The LCS solution can also be made in larger volumes fresh each day and aliquoted out as needed for each run.

7.3 MS and MSD. The method spike is prepared by adding 50µl of each of the three standards directly to a randomly chosen sample. The MSD is prepared exactly the same, added to a duplicate of the MS sample. An MS/MSD should be included in every 20 samples.

7.4 CCB is identical to the method blank. Prepare a sample of 9:1 methanol:chloroform and add to an autosampler vial. One CCB should be included for every 10 samples.

7.5 CCV is identical to the LCS and should be included once for every 10 samples.

7.6 Standard QC for a sample run. **QUALITY CONTROL (See QC Summary Tables in Appendix A)**

8.0 References

8.1 Swift W, Wong A, Li KM, Arnold JC, McGregor IS: Analysis of cannabis seizures in NSW, Australia: Cannabis potency and cannabinoid profile. PLoS One. 2013, 8: e70052-10.1371/journal.pone.0070052.
16.2 De Backer B, Debrus B, Lebrun P, Theunis L, Dubois N, et al. (2009) Innovative development and validation of an HPLC/DAD method for the qualitative and quantitative determination of major cannabinoids in cannabis plant material. J Chromatogr B Analyt Technol Biomed Life Sci 877: 4115–4124. doi: 10.1016/j.jchromb.2009.11.004
Cannabis Inflorescence: Standards of Identity, Analysis, and Quality Control, Revision 2014.

9.0 Appendix

9.1 Expected peaks and elution time:

1.20 Method Performance

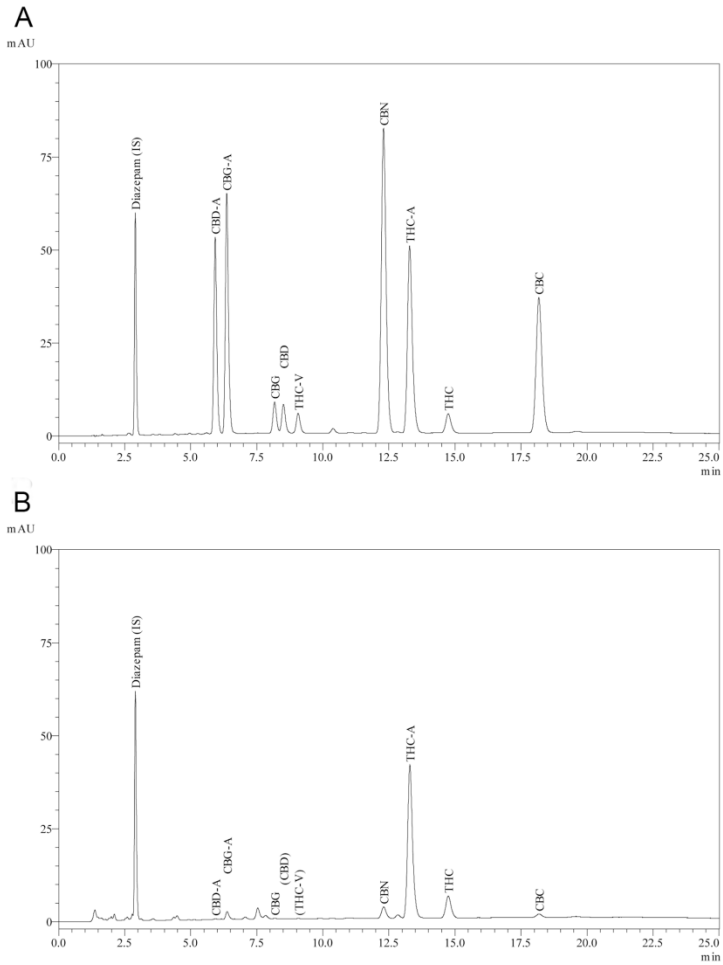
1.21 Pollution prevention

1.22 Waste Management

1.23 References

1.23.1 Swift W, Wong A, Li KM, Arnold JC, McGregor IS: Analysis of cannabis seizures in NSW, Australia: Cannabis potency and cannabinoid profile. PLoS One. 2013, 8: e70052-10.1371/journal.pone.0070052.

1.23.2 De Backer B, Debrus B, Lebrun P, Theunis L, Dubois N, et al. (2009) Innovative development and validation of an HPLC/DAD method for the qualitative and quantitative determination of major cannabinoids in cannabis plant material. J Chromatogr B Analyt Technol Biomed Life Sci 877: 4115–4124. doi: 10.1016/j.jchromb.2009.11.004



Cannabis Inflorescence: Standards of Identity, Analysis, and Quality Control, Revision 2014.

1.24Appendix

1.25 Expected peaks and elution time:

Microbial Testing- qPCR for the detection of microbial pathogens (adapted from Medicinal Genomics PathogINDICAtor protocol)

Revision

0

Effective Date

7/27/16

Prepared By

Southeast Alaska Laboratories, LLC

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315-778-1469

APPROVED BY:

Laboratories Director	Date
Quality Assurance Officer	Date

1.0 Scope
2.0 Reagents and Materials
3.0 Procedure

1.0 Scope

This is a PCR based assay that is contamination free and provides an internal plant DNA control for every reaction. It is a simple two-step protocol, which is flexible and is automation compatible.
This assay uses a multiplexing strategy with an internal plant DNA reaction control to ensure accurate detection of microbial species for every reaction. This multiplexing strategy verifies the performance of the assay when detecting pathogens, resulting in the minimization of false negatives due to reaction set-up errors or failing experimental conditions.

2.0 REAGENTS AND MATERIALS

2.1 qPCR Master Kit (stored at -15 to -20oC) [Medicinal Genomics #420002].
This kit should contain:
- Decontamination Enzyme (10 Units/µL)
- Decontamination Diluent
- Decontamination Buffer (10x)
- qPCR Master Mix (5x)

2.2 Total Pathogenic E.coli (STEC) Detection Assay (Medicinal Genomics #420104), Total Pathogenic E.coli (STEC), Positive Control (Medicinal Genomics #420304), Salmonella Detection Assay (Medicinal Genomics #420101), Salmonella Positive Control (Medicinal Genomics #420301), Yeast & Mold Detection Assay (Medicinal Genomics #420103), Yeast & Mold Positive Control (Medicinal Genomics #420303)
2.3 BioRad (or similar) Real-Time PCR System w/computer
2.4 96 well optical qPCR plates (Agilent AriaMx 96 well plates, Agilent # 401490, 401491, or 401494 or Fisher Scientific 96-Well Armadillo PCR Plate, Fisher # AB2396)
2.5 Adhesive optical seal for qPCR plates (Agilent adhesive plate seals, Agilent # 401492 or USA Scientific TempPlate® RT Optical Film # 2978-2100)
2.6 Multi-channel pipette P50 or P20
2.7 Single channel pipette P10, P20 and P200
2.8 Filtered pipette tips for P10, P20, P50, and P200

2.9 Crushed ice or cold racks (96 well PCR Cryogenic Rack, VWR #89004-570 and 1.5ul Tube Benchtop Cryogenic Racks, VWR #89004-558 or similar)
2.10 Freezer, -20oC
2.11 Table top mini plate centrifuge (Fisher Scientific #14-100-143 or similar)
2.12 Table top mini tube centrifuge (VWR® Mini Centrifuge #10067-588 or 6-place personal microcentrifuge for 1.5/2.0 ml tubes # 2631-0006, or similar)
2.13 Table top Vortex Genie (Scientific Industries #SI-0236 or Similar)
2.14 10% bleach

3.0 Procedure
3.1 Begin with a 10% bleach wipe down of the workspace, including the bench top and all equipment being used (except the Agilent AriaMx instrument or Notebook PC).
3.2 Remove decontamination reagents and positive controls for the tested assays from the -20oC freezer.
3.3 Decon Buffer and positive controls – Allow tubes to defrost at room temperature. Once defrosted, place tubes on ice.
3.4 Decon Enzyme and Decon Diluent – Place directly on ice.
3.5 Before preparing the reaction, invert or vortex the tubes and spin-down the reagents.
3.6 Decon Buffer and Positive Controls – Vortex quickly followed by a pulse spin down in a microcentrifuge.
3.7 Decon Enzyme and Decon Diluent – Invert the tube 5 times, followed by a pulse spin-down in a microcentrifuge. 3.3. Return all reagents to the ice. Do NOT vortex.
3.8 Begin with making 1Unit/µL stock of Decon Enzyme.
3.9 Label a 1.5ml tube with "1:10 Decon Enzyme"
3.10 Make a 1:10 dilution of 10 Unit/µL Decon Enzyme with Decon Diluent.
For every 1µL of 10 Unit/µL Decon Enzyme dilute with 9µL of Decon Diluent. Note: It is best to add the largest volume reagent first, in this case Decon Diluent
3.11. Once combined, slowly tip mix or invert the tube 5 times. 4.4. Pulse spin-down in a microcentrifuge.
3.12 Return all reagents to the ice.

3.13 Make a master mix using the table below, labeling a 1.5ml tube as "Decon MM"

Reagent	1 Reaction	24 Reactions (plus 15% excess)	48 Reactions (plus 10% excess)
Decon Buffer	1µL	27.6µL	52.8µL
Diluted Decon Enzyme (1Unit/µL)	1µL	27.6µL	52.8µL
H_2O	3µL	82.8µL	158.4µL
Total	5µL	138µL	264µL

3.14 The number of reactions must include an assay specific positive control and negative control for all assays to be tested. (i.e. When testing Yeast & Mold, E.coli, and Salmonella there will need to be 3 assay specific Positive Controls and 3 Negative Controls).
3.15 For the positive control, make a 1:10 dilution.
1ul of Positive Control (found in the kit) dilute with 9ul of H2O(found in the kit).
For the negative control, use H2O (found in the kit).
Note: It is best to add the largest volume reagent first, in this case the H2O.
3.16 Place the extraction plate on the magnet.
3.17 Use a new 96 well optical qPCR plate and label the plate "Decontamination Plate_[date]". Add 5ul of Decon MM to each corresponding sample well, positive control well, and negative control well in the qPCR plate.
Note: It may be helpful to label each of the corresponding column wells to accurately dispense the correct samples.
3.18 Remove the seal from the Extraction Plate and transfer 5uL of each sample into the corresponding well in the qPCR plate. Keep the extraction plate on the magnet when aspirating the 5uL.
3.19 Add 5uL of the diluted Positive Control to their corresponding well. Then add 5uL of water to the corresponding Negative Control well.
Note: ALWAYS use a fresh tip for every liquid transfer into the qPCR plate.
3.20 Tip mix all wells by pipetting up and down 2 times.

3.21 Seal the plate with the adhesive seal, making sure to completely seal the plate wells using a pen or flat object to slide back and forth along the seal.
3.22 Spin down for at least 1 minute in the plate microcentrifuge.
3.23 Label the plate as Decontamination Plate [date].
3.24 Place the sealed plate onto the qPCR instrument, positioning the A1 well in the top left corner.

3.25 Starting the qPCR reaction: Remove qPCR Reagents and Assay tubes from the -20oC freezer. Bring qPCR Master Mix and Assay Probe Mix tubes – Allow tubes to defrost at room temperature. Once defrosted, immediately place tubes on ice.
3.26 Before preparing the reaction, invert or vortex and spin-down the reagents.
3.27 Assay Probe Mix tubes – Vortex quickly followed by a pulse spin-down in a microcentrifuge.
3.28 qPCR Master Mix – Invert the tube 5 times, followed by a pulse spin-down in a microcentrifuge.
3.29 Return all reagents to the ice.
Note: Do not vortex the qPCR Master Mix at any point during the protocol.
3.30 Make a separate master mix in a 1.5mL tube for each different Assay Probe Mix being tested (all probe mixes contain the internal plant control, SCCG probe mix), labeling them [Assay Name] MM.
Note: It is best to add the largest volume reagent first, in this case H2O.

Reagents	1 Reaction	24 reactions (plus 5% excess)	48 reactions (plus 5% excess)
qPCR Master Mix	5µL	126µL	252µL
Assay Probe Mix (Assay Specific)	1µL	25.2µL	25.2µL
H2O	9µL	226.8µL	453.6µL
Total	15µL	378µL	756µL

3.31 3.1. Once combined invert the tube 5 times to combine the Assay MM tubes 3.2. Pulse spin-down in microcentrifuge.
3.32 Place MM tubes on ice until used in step 5.
3.33 Remove the seal from the Decontamination Plate carefully (pay attention not to spill any liquid). Add 15µL of probe specific MM to each of the corresponding detection assay wells in the spun-down decontamination plate.
3.34Tip mix all wells by pipetting up and down 2 times.
3.35 Seal the plate with the adhesive seal, making sure to completely seal the plate wells using a pen or flat object to slide back and forth along the seal.
3.36 Spin-down for at least 1 minute in the plate microcentrifuge.
Note: Check for bubbles at the bottom of the wells (bubbles on the surface of the liquid is acceptable). If bubbles remain in the bottom of the wells, spin-down for another minute.
3.37Re-label the plate as qPCR Plate_[date].
3.38 Place the sealed plate onto the qPCR instrument, positioning the A1 well in the top left corner.
3.39 Use the following thermal program: Hot start at 95°C for 5minutes, followed by 40 cycles of 95°C for 15 seconds and 65°C for 90 seconds.

Isolation of microbial DNA from Cannabis

Revision

0

Effective Date

7/27/16

Prepared By

Southeast Alaska Laboratories, LLC

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99801

315-778-1469

APPROVED BY:

Laboratories Director	Date
Quality Assurance Officer	Date

1.0 Scope
2.0 Reagents and Equipment
3.0 Procedure
4.0 Troubleshooting

1.0 Scope

This DNA isolation process uses magnetic particles to isolate and purify both plant and microbial DNA from a raw, homogenized cannabis sample. DNA can be isolated from a single sample or a large batch in under 1 hour. Hands-on time is less than 45 minutes.
To enable minimal laboratory overhead, all organic solvents have been replaced with non-caustic reagents and 70% EtOH. Magnet plates are available for purchase from Medicinal Genomics (part #420202).

2.0 Reagents and Equipment

2.1 SenSATIVAxTM Plant/Microbial DNA Purification Kit contains 200 reactions (Medicinal Genomics #420001) or 1000 reactions (Medicinal Genomics #420206) worth of reagents.
Kit should include:
- MGC Lysis Buffer (Store at Room Temperature, 20oC to 28oC)
- MGC Binding Buffer (Store at 2-8oC)
- MGC Elution Buffer (Store at Room Temperature, 20oC to 28oC)

2.2 Whirl-Pak bags (Nasco #B01385WA)
2.3 Solo Cups or Beaker (optional)
2.4 MGC Enrichment Broth, store at 2oC-8oC (Medicinal Genomics #420205)
2.5 1.5 mL Eppendorf tubes (Multiple Suppliers)
2.6 Adhesive optical seal for qPCR plates (Bio-Rad Microseal® # MSB-1001 or USA Scientific TempPlate® RT Optical Film # 2978-2100)
2.7 Multi channel pipettes P20 and P300, or P50 and P1000 (optional)
2.8 Single channel pipettes P20, P200, & P1000
2.9 Filtered pipette tips for P20, P50, P200, & P1000
2.10 Eppendorf tube rack
2.11 Scientific scale (milligram)
2.12 Incubator, that can reach 37oC (VWR® Personal Size Incubator # 97025-630, or similar)
2.13 Table top mini tube centrifuge (VWR® Mini Centrifuge #10067-588 or 6-place personal microcentrifuge for 1.5/2.0 ml tubes # 2631-0006, or similar)
2.14 96 well plate magnet (Medicinal Genomics #420202)
2.15 96 well extraction plate (Perkin Elmer #6008290)

2.16 able top Vortex Genie (Scientific Industries #SI-0236 or Similar)
2.17 10% Bleach
2.18 70% Ethanol (EtOH) (American Bioanalytical product # AB00844-01000)

3.0 Procedure:
3.1 Extraction #1 Protocol (for CFU Threshold Assays):
Begin with a 10% bleach wipe down of the workspace, including the bench top and all equipment being used.
3.2 Remove the MCG Binding Buffer and the MGC Enrichment Broth from the 2-8oC refrigerator (it should come to room temperature before use).
3.3 Before weighing out the sample to be tested, make sure that the entire sample is broken up and thoroughly homogenized. A well-homogenized sample will ensure more accurate testing.
3.4 Label a new Whirl-Pak bag with the "[sample name] [date]". After homogenization, weigh out 0.22-0.28g, and put into the labeled Whirl-Pak bag. Make sure to add all of the sample material to one side of the mesh layer inside the Whirl-Pak bag. If processing multiple plant samples, be sure to change gloves between each, to ensure there is no cross contamination of flowers during the weighing process.
3.5 Add 3.55mL of MGC Enrichment Broth to Whirl-Pak bag.
Note: MGC Enrichment Broth is a growth medium and the perfect condition for microbes to grow. Due to this, it is best to pour the approximate amount of MGC Enrichment Broth into another sterile tube or container as to not contaminate the whole bottle. Nothing should go into this bottle. Return it to the 2-8oC refrigerator immediately after use.
3.6 Close the Whirl-Pak bag by folding the top over three times.
3.7 Mix the homogenized plant material in MGC Enrichment Broth for at least 1 minute with your fingers, one sample at a time.
3.8 Prepare and label a 1.5mL tube with the "[sample name] [date]". Aspirate 285 µL from the side of the filter bag, free of plant debris, and dispense into the 1.5mL tube.
a. If using a presence/absence test (E.coli, Salmonella or Aspergillus), save and incubate the Whirl-Pak bag at 37oC for 16-24 hours, and proceed to the Extraction #2 set up. Incubate a full 24 hours for Aspergillus testing.
3.9 Add 15µL of MGC Lysis buffer to the 285µL sample.
3.10 Vortex for 5 seconds, and incubate on the bench for 2 minutes.
3.11 After 2 minute incubation, spin for at least 30 seconds in a bench top mini centrifuge.

3.12 Remove the 200µL of supernatant from the 1.5ml tube containing the centrifuged sample, being careful not to disturb the pellet at the bottom of the tube. Place the 200µL in a labeled 96 well extraction plate labeled with "Extraction Plate Day1 [date]" or "Extraction Plate Day2 [date]".
Note: Pellet size will vary depending on trichome density.
3.13 Vortex MGC Binding Buffer thoroughly before use, be sure that the magnetic particles are completely re-suspended in buffer at least 30 seconds.
3.14 Add 200µL of MGC Binding Buffer (this liquid is very viscous) to the 200µL sample, and pipette tip mix 15 times.
a. Incubate the plate on the bench for at least 5 minutes.
Note: Be careful to avoid adding too many bubbles by pipetting gently when tip mixing. This is extremely important as to not contaminate the wells in proximity.
3.15 Place the extraction plate onto the 96 well plate magnet plate for at least 5 minutes.
12. After 5 min incubation, remove as much of the 400ul of the supernatant as possible. Be careful not to disturb or aspirate the beads.
a. Add 400µL of 70% ethanol (EtOH) with the extraction plate still on the magnet plate.
b. Wait at least 30 seconds, and remove all the EtOH.
Note: Take the pipet tip to the bottom center of the well to remove liquid.
3.16 Again, add 400µL of 70% EtOH with the extraction plate still on the magnet plate. Wait at least 30 seconds and remove all the EtOH.
Note: If EtOH still remains in the wells, go back in with a smaller pipet tip to remove the excess.
3.17 After all the EtOH has been removed let the beads dry at room temperature on the magnet plate for at least 15 minutes. Be sure to remove all EtOH, as any leftover can inhibit qPCR results.
3.18 Remove the extraction plate from the magnet plate, and add 50µL of MGC Elution Buffer.
a. Tip mix approximately 15 times or until the beads are completely re-suspended.
Note: The re-suspensions may appear varied in their appearance, but the result will be the same.
b. Incubate the plate for at least 1 minute on the bench before returning the plate to the magnet plate.
c. Let the plate sit on the magnet for at least 1 minute before transferring the eluent to a new extraction plate labeled with "Final Extract Day 1 [date]" or "Final Extract Day 2 [date]".

Note: To save space and consumables, both day 1 and day 2 extracts can be stored in separate wells on the same extraction plate.
Seal the plate with the adhesive seal, making sure to completely seal the plate wells using a pen or flat object to slide back and forth along the seal. Store at -20oC until ready to perform qPCR protocol.

3.18 Extraction #2 Protocol (Presence/Absence) After an 16-24 hour incubation at 37oC has occurred remove 285µL from the side of the filter bag free of plant debris, and put into a 1.5mL tube and repeat steps 5-16.
Note: If using the Aspergillus Specific Detection Assays, you must incubate for the full 24 hours to insure proper growth.

4.0 Troubleshooting:

Symptom	**Reason**	**Solution**
Clumpy/Grainy Beads	Over-manipulation of plant with MGC Enrichment Broth	Over manipulation of the plant can cause the release of extra cellular debris therefore clogging the beads with extra material. To ensure this does not occur, only manipulate the plant material for 1 minute.
	Too many tricomes and/or insufficient spinning	Some plants produce more tricomes than others resulting in carry-over into extraction plate. To ensure this doesn't happen, it may be necessary to spin the tube for longer than the recommended 30 seconds. Also, be sure not to disturb the pellet. If the pellet is disturbed or tricomes are still visible, re-centrifuge the tube and

		try again.
Bead Loss	Insufficient time on the magnet	Make sure the supernatant has fully cleared before removing. Failure to do so will result in bead loss, which will result in DNA loss.
	Insufficient pipetting	Make sure no beads are aspirated during supernatant removal; dispense back supernatant, and attempt again with a smaller volume after beads have re-settled..
Extra elution volume	Insufficient removal of Ethanol	Make sure ALL ethanol is removed before drying. This may require a second or third aspiration. Carry-over ethanol can cause inhibition in qPCR.

Residual Solvent Analysis- Residual Solvent Analysis of Butanes, Heptanes, Benzene, Toluene and Hexane.

Control#_____

OG-0000.00

Residual Solvent Analysis by GCMS

Revision
0

Effective Date
7/19/16

Prepared By

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APPROVED BY:

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Laboratory Director Date

Quality Assurance Officer	Date

1.0 SCOPE AND APPLICATION

2.0 SUMMARY

3.0 TERMS AND DEFINITIONS

4.0 HEALTH AND SAFETY

5.0 APPARATUS AND MATERIALS

6.0 REAGENTS

7.0 SAMPLE COLLECTION/ PRESERVATION/ HOLDING TIMES

8.0 QUALITY CONTROL (See QC Summary Tables in Appendix A)

9.0 DEMONSTRATION OF CAPABILITY

10.0 Limit of Detection (LOD)

11.0 CALIBRATION AND STANDARDIZATION

12.0 PROCEDURE

13.0 REFERENCES

1.0 SCOPE AND APPLICATION

This procedure will be used for all extracts, hash, and other samples requiring analysis for residual solvents.

2.0 SUMMARY

Determination of the amounts of residual solvents by headspace GCMS.

3.0 TERMS AND DEFINITIONS

3.1 No new terms in this SOP

4.0 HEALTH AND SAFETY

4.1 Lab coat, gloves and eye protection should be worn during the preparation of samples and standards for this procedure.

5.0 APPARATUS AND MATERIALS

5.1 GCMS with capability of sample introduction by headspace Shimadzu QP-2010 Ultra with HS-20 or similar instrument.
5.2 10 mL headspace vials with caps and crimper
5.3 analytical balance readable to 0.1 mg
5.4 micro pipets w/tips
5.5 15 mL graduated centrifuge tubes
5.6 1.8 mL autosampler vials

6.0 REAGENTS

6.1 standard reference materials :

- iso-butane
- n-butane
- methanol
- pentane
- ethanol
- acetone
- isopropanol
- methylene chloride
- n-hexane
- ethyl acetate
- chloroform
- benzene
- toluene
- DMSO

7.0 SAMPLE COLLECTION/ PRESERVATION/ HOLDING TIMES

7.1 Samples should be collected and received as described in the collection and chain of custody SOPs.

8.0 QUALITY CONTROL (See QC Summary Tables in Appendix A)

1. The analysts must make an initial demonstration of capability and generate acceptable accuracy and precision with the method before analyzing any samples. Any changes or modifications to this SOP require the analyst to repeat the demonstration of capability.

9.0 Demonstration of Capability:

9.1 A quality control sample shall be obtained from an outside source.

9.2 The four quality control samples shall be analyzed according to the test method either concurrently or over a period of days.

9.3 Run the aliquots after analyzing all required calibration standards. Ensure that the reagent blank does not contain any analyte at a concentration greater than the reporting limit (RL).

9.4 Using all of the results, calculate the mean recovery (x) and the Percent Relative Standard Deviation.

9.5 An acceptable DOC has a mean percent recovery between 80% and 120%. The relative percent standard deviation <20%.

10.0 Limit of Detection (LOD):

10.1 LOD's must be determined annually. Also, LOD's must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

10.2 A MB, LCS, DUP and CCV must be run with every batch. A batch includes at most twenty samples.

10.2.1 Method Blanks (MB)-Will be performed at a frequency of one per batch of samples per matrix type per extraction or

preparation method. The results of this analysis will be one of the QC measures to be used to assess batch acceptance. The source of contamination must be investigated and measures taken to correct minimize or eliminate the problem if: 1) The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch or 2) The blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

10.2.2 Lab Control Sample (LCS)/Initial Calibration Verification (ICV) - Are performed at a frequency of one per batch of 20 or less of samples per matrix type, and sample preparation. The results of analysis are used to assess batch acceptance. The LCS must have a percent recovery of 80%-120% to be acceptable.

10.2.3 Sample Duplicates (DUP) - Are performed at a minimum of 1 in 20 samples per matrix type per sample preparation. The duplicate sample is chosen at random. The selected samples are rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. The RPD for water samples is ±10%.

10.3 Continuing Calibration Verification (CCV): After 10 samples and/or at the end of analysis run, prepare a CCV to verify calibration. The concentration must vary to verify different parts of the curve. The CCV must have a recovery of ±20% to be acceptable. If a CCV fails, any sample associated with the CCV must be re-analyzed.

10.4 If the MB or LCS fails, the analysis will be rerun.

10.5 If the MB and LCS pass but the DUP and/or MS fail, the data is reported with qualifiers.

10.6 All data must be recorded in the Cannabis % Moisture data logbook.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Prepare the LCS per manufacturer's instructions. Concentrations may vary between lots.

11.2 Prepare the following standards using 100 mL volumetric flasks: (Final volume is 100 mL with water.)

Amount of Stock Cannabis Solution	Concentration Cannabis
0 mL	0 mg/L
2 mL	20 mg/L
5 mL	50 mg/L
7.5 mL	75 mg/L
10 mL	100 mg/L
15 mL	150 mg/L

11.3 The correlation for linear regression must be at least 0.995. Print graph for each calibration curve to be included in the data review packet. If the correlation is <0.995 then the calibration must be redone before any samples can be analyzed.

11.4 Initial Calibration Verification (ICV): The LCS is from a secondary source, and is used to verify the calibration. The acceptable limits are 80-120%. If the ICV fails, the problem must be corrected before samples are analyzed.

12.0 PROCEDURE

12.1 **GCMS acquisition method:**

- GC column: Rxi-624SIL-MS, 30 m x 0.25 mm ID, 1.4 µm DF
- Temperature program: 35°C (hold 3.0 min) to 300°C at 30°C/min (hold 3.0 min)
- Injection: split 1:50
- Linear Velocity: 33.0 cm/sec
- MS scan: 20 to 400 m/z, 2.0 min. solvent delay, acq. stops at 8 min.
- Ion Source Temperature: 225°C
- Interface Temperature: 250°C
- Headspace incubation: 140°C for 10 minutes
- Headspace Sample Line Temp: 150°C
- Headspace Transfer Line Temp: 150°C
- Headspace Pressurizing Gas Pressure: 25 kPa
- Headspace Pressurizing Time: 1.0 min
- Headspace Injection Time: 0.5 min

12.2 Accurately weigh between 5 and 10 mg of sample into a 10 mL headspace vial and record the weight.

1. Crimp a cap onto the vial.
2. Label the vial with the sample identification and the weight. Mark directly on the vial, do not use tape or a label.

12.3 Accurately prepare a stock solution of butane in DMSO in the range of 5,000 µg/mL:

1. Transfer approximately 6 mL of DMSO to a 15 mL graduated centrifuge tube. Cap the tube and tare it on the analytical balance.
2. Transfer approximately 0.5 mL of liquid butane to a 1.8 mL autosampler vial and cap the vial. Allow the vial to cool until it reaches thermal equilibrium.
3. Uncap the centrifuge tube of DMSO, pour the butane into it, and immediately recap the tube. The top surface of the DMSO will freeze.
4. Weigh the tube and repeat the previous two steps until approximately 0.5 gram of butane has been added. Record the exact mass of the butane that has been added.
5. Allow the DMSO to thaw and invert the tube repeatedly to mix the butane with the DMSO.
6. Uncap the tube, bring to 10 mL with DMSO, and immediately recap the tube. Invert several times to mix.
7. Calculate the mass/volume concentration of butane in the solution. Calculate the volume of this solution that would be required to create a 1000 µg/mL standard when diluted to 10 mL.

12.4 Prepare a 1,000 µg/mL stock solution by adding the volume of the butane stock standard calculated in the last step to a 15 mL graduated centrifuge tube, adding ~4 mL DMSO, and then volumetrically adding each solvent to the flask using a micro-pipet according to the table below. Tare the tube prior to addition of each solvent and measure and record the mass of each solvent addition.

Compound	**Density**	**Volume required (µL)**
Methanol	0.79	12.6
Pentane	0.63	16.0
Ethanol	0.79	12.6
Acetone	0.79	12.6
Isopropanol	0.79	12.6
Methylene Chloride	1.33	7.5
n-Hexane	0.66	15.2

Ethyl Acetate	0.90	11.1
Chloroform	1.48	6.7
Benzene	0.88	11.4
Toluene	0.87	11.5

12.5 After the addition of neat solvents, bring the volume to 10 mL with DMSO, cap the tube, and mix by gently inverting several times.

1. Use the weights of each of the analyte sents added to the standard to calculate the exact concentration of each in the standard. These will be close to 1,000 µg/mL.
2. Standards at lower concentrations may be prepared by diluting the 1,000 µg/mL stock solution with DMSO into 1.8 mL autosampler vials.
3. Transfer 10 µL of each of the standards into separate 10 mL headspace vials with a micro pipet. Crimp caps onto the vials and label them. Mark directly on the vials, do not use tape or a labels. Standards in headspace vials may not be reused after the septa have been punctured.
4. The standards may be kept in 1.8 mL autosampler vials tightly capped with unpunctured septa at -5 °C.

12.6 Instrument Initialization

1. Start the data acquisition program from the *GCMS Real Time Analysis* icon on the desktop.
2. After the program has initialized, select the *Data Acquisition* icon from the purple Assistant Bar on the left side of the program window. If the *Data Acquisition* icon is not visible, select the *Top* arrow icon to display the *Real Time* top level selections.
3. Select *Open Method File…* from the *File* menu and load the *Residual Solvents.qgm* method from the *C:/GCMSsolution/Data/Project1/Method* directory.
4. Select *Select Project(Folder)…* from the *File* menu. If necessary, create a directory for the current date using the existing format, and select the current date.
5. Select *Save Method File As…* from the *File* menu and save the method file into the directory just specified for the current date.
6. Select *Download Initial Parameters* from the *Acquisition* menu. This will download the method parameters to the instrument.
7. Select the *Top* arrow icon on the Assistant bar and then select *Tuning.*
8. Verify that the MS status is green *Ready* at the top of the Instrument Monitor on the right side of the program window. Wait for it to show *Ready* if necessary.
9. Select the green *Start Auto Tuning* icon on the Assistant Bar. The auto tune process will start and take approximately two minutes. Save the tuning report as a PDF file if prompted.
10. Select the *Spectrum* tab above the three large peaks displayed in the center of the program window.
11. Verify that the peaks at ~18 amu (water), ~28 amu (nitrogen), and 32 amu (oxygen) at the left end of the spectrum are less than half the intensity of the peak at ~69 amu. If any of them are greater than half the intensity of the 69 peak, there may be a vacuum leak and the laboratory manager should be consulted before proceeding.
12. Verify that the *Detector* voltage listed to the right of the spectrum is less than or equal to 1.5 kV. If the detector voltage exceeds 1.5 kV notify the laboratory manger before proceeding.

13. Select *Save Tuning File As…* from the *File* menu and save the tuning file into the *Tune1* directory with the format *yy-mm-dd.qgt* with the current date, *i.e.*, an auto tune carried out on March 17, 2015 would be saved as *15-03-17.qgt.*
14. Select the *Top* arrow icon on the Assistant Bar and then select the *Batch Processing.* Alternatively, batch processing may also be selected using the tab at the bottom of the main program display area.
15. Select *Open Batch File…* from the *File* menu and load *residual solvents sample template.qgb* batch file from the *C:/GCMSsolution/Data/Project1/Batch* directory.
16. Select *Save Batch File As…* from the *File* menu and save the file to the directory for the current date with the format *mm-dd-yyyy_GCMS_RS_(nn).qgb* with *nn* being the sequential number of the batch files for a given day, *i.e., 03-17-2015_GCMS_RS_(01).qgb* for the first batch file on that date and *03-17-2015_GCMS_RS_(01).qgb* for the second batch file for that date.

12.7 Data Acquisition

1. Display the Batch Table if necessary by selecting *Batch Processing* from the Assistant Bar or the *Batch Table* tab at the bottom of the main window.
2. Select the *Settings* icon on the Assistant Bar. Select the *Folder* tab in the *Settings* dialogue box and select the directory for the current date as the specified folder to use. Check *Use the same folder*.
3. Select the *Data Filename* tab and verify that the *Selected Items* list includes only *Sample ID* and the *Auto-increment Format* is set to *01, 02, …* . Close the *Settings* dialog box.
4. Clear any entries in the *Vial#, Sample ID, Customer/Sample Name, weight used, description, Sample Amt., Dil Factor, Method File* and *Tuning File* columns of the batch table.
5. Fill in the batch table for the sample to be run:
6. *Vial#* - position of the sample vial in the autosampler rack
7. *Sample ID* – laboratory ID, *i.e., EXT 15-0001.*
8. *Customer/Sample Name* – customer and sample name
9. *weight used* – weight of the sample in the vial in grams

10. *description* – oil, shatter, wax, etc.
11. *Sample Amt.* – weight of the sample in the vial in milligrams
12. *Dil. Factor* – 10
13. *Sample Type* – 0:Unknown
14. *Method File – Residual Solvents.qgm* from the current folder
15. *Tuning File* – tuning file from the current date.
16. Include mixed standards with the samples in the batch table as necessary.
17. Add lines to the batch table as needed or delete unused lines by right-clicking on the row number of the table and selecting the appropriate action.
18. Select *Save Batch File* from the *File* menu.
19. Place the vials into the appropriate locations in the sample rack and then execute the batch file by selecting the *Start* icon with the green arrow from the Assistant Bar.

12.8 Calibration

1. Select the *Calibration Curve* icon from the Assistant Bar. If the *Calibration Curve icon* is not visible, select the *Top* arrow icon todisplay the *Postrun* top level selections.
2. Select the *Method* tab (second from the left) at the bottom of the list of files near the left side of the program window. If the list of files is not visible, select *Data Explorer* from the *View* menu.
3. Select the directory of files listed to the current date if necessary, and then double click on *Residual Solvents.qgm* to load that method.
4. Select the *Param's* tab at the bottom of the calibration table on the right side of the program window.
5. Select *Edit* at the upper right of the calibration table and edit the concentrations to reflect the actual concentrations in the standards. Select the *View* button to the left of the *Edit* button when finished. If additional concentration levels need to be added select *Quantitative Parameters…* from the *View* menu, select the *Quantitative* tab, and change the *# of Calib. Levels* as needed.
6. The various calibration levels will appear in the lower center area of the program window. Data files of standards used in the previous calibration will be listed under the calibration levels. Delete these files by right clicking on them and selecting *Delete.*

7. Select the *Data* tab (farthest to the left) at the bottom of the Data Explorer. The data files from the current day will be listed.
8. Drag the data files from the standards to the appropriate levels in the calibration list. If more than one standard was run for a calibration level, drag all of them to the list and the responses will be averaged. The mean, standard deviation, and relative standard deviation for all of the files at a given level will be listed in the area between the calibration list and the plot of the calibration curve.
9. The area counts for the response for specific compounds from different acquisitions may be de-selected by highlighting the compound in the calibration table on the right side of the program window and then unchecking the box next to the area count for the acquisition to be de-selected.
10. The slope, intercept and r^2 value for the calibration curves are displayed to the right of the plot of the calibration curve. Highlighting specific analyte compounds in the calibration table will cause the calibration curve and statistics for those compounds to be displayed.
11. Additional parameters for the calibration curve may be altered by selecting *Quantitative Parameters…* from the *Method* menu and selecting the *Quantitative* tab.
12. After the calibration curves for each of the analyte compounds have been verified to be correct, select *Save Method File* from the *File* menu and respond *Yes* to the prompt to save data files.
13. Select *Close Method File* from the *File* menu.
14. Select the *Top* arrow icon in the Assistant Bar and then select the *Quantitative* icon.
15. If a data file is loaded on the Quantitative screen close it by selecting *Close Data File* from the *File* menu. Do not save the file.
16. Select the *Top* arrow icon on the Assistant bar and then select the *Batch Processing* icon.
17. Select the tab for batch files (second from the right) at the bottom of the Data Explorer.
18. Load the batch file for the data to be analyzed by double clicking on the file name in the Data Explorer. If the batch file is still loaded in the *GCMS Real Time Analysis* program, respond to the prompt that appears with *Close & Open.* In that case a prompt may appear in the *GCMS Real Time Analysis* program asking if you want to save the current batch file – respond *No.*

19. Select the *Start* icon with the green arrow on the Assistant Bar. If a prompt appears asking what Execution Range to use, select *All Rows* and then *Start.* If a prompt appears asking to save the current batch file, select *OK*.
20. The new calibration will be applied to each of the data files in the batch table.

12.9 Reports

For each data file:

1. Select *Quantitative* from the top level of the Assistant Bar.
2. Load the selected data file.
3. Select *Report* from the Assistant Bar.
4. Select *Open Format File…* from the *File* menu and then load the *Residual Solvents.qgr* file from the *C:/GCMSsolution/Data/Project1/Report* directory.
5. The chromatogram on the report may be scaled by right clicking on the chromatogram and selecting *Properties*, selecting the *Chromato* tab in the *Properties* dialog box and then entering the desired Y scale. Checking the *Auto* box for the Y scale will autoscale the chromatogram, but this will often obscure the baseline.

To label the peaks

1. Select the *Return* arrow on the assistant bar.
2. Select *Peak Integrate for TIC (all Group)* from the *Qualitative* menu.
3. In the *Qualitative Parameters* dialog box select the *Peak Integration* tab and select Auto(Area) and enter 15 for the *# of Peaks* and *0.04* sec for the *Width*. Select *OK* and then *OK* again to register the peaks to the Spectrum Process Table.
4. When the peak integration and spectrum similarity search is complete the compound names should appear on the peaks. If they do not, right click on the chromatogram window and select *Properties* and then select *Name* under *Peak Top Comment* on the *General* tab. Select *OK.*
5. To edit or remove some of the labels select *Show Qualitative Table…* from the *Qualitative* menu. Names may be edited or deleted directly from the Qualitative Table. To view synonyms or alternative library matches double click on the row of the peak in

the Qualitative Table. Names will be updated on the chromatogram as changes are made in the Qualitative Table.

6. Save the data file after any changes have been made.
7. Return to the Report screen and the names will be displayed. Adjust the Y scale of the chromatogram if necessary.
8. Print the report by selecting *Print* from the Assistant Bar.

13.0 REFERENCES

Appendix A

QC Summary

Quality Control Element	Frequency	Acceptance Limits
Limit of Detection (LOD)	Annually or with substantial change to method or instrumentation.	40 CFR Part 136, Appendix B calculations.
Demonstration of Capability (DOC)	Annually for each analyst	Average of replicates must be 80-120% of true value and < 20% RSD.
Initial Calibration Verification (ICV)	Following every initial calibration.	80-120% of true value.
Continuing Calibration Verification (CCV)	At the start of each analytical batch when an initial has not been performed. Note: The CCV concentration is to be varied through the analysis.	80-120% of true value.
Method Blank	1 in every 20 or fewer samples per preparation batch and at least 1 per 12 hour period.	Concentration must be below the MRL.
Laboratory Control Sample (LCS)	1 in every 20 or fewer samples per preparation batch	80-120% of true value.
Matrix Spike (MS)	1 in every 20 or fewer samples per preparation batch. Sample chosen at random.	80-120% of true value. Not required for batch acceptance.
Matrix Spike Duplicate (MSD)	1 in every 20 or fewer samples per preparation batch. Sample chosen at random.	80-120% of true value. Not required for batch acceptance. Relative Percent Difference (RPD) <10%

Appendix A (Continued)

QC Corrective Action Summary

Quality Control Element	Corrective Action
Limit of Detection (LOD)	Determine source of problem, correct, and re-analyze.
Demonstration of Capability (DOC)	Determine source of problem, correct, and re-analyze.
Initial Calibration Verification (ICV)	Re-read ICV. If ICV fails a second time, a new initial calibration is required.
Continuing Calibration Verification (CCV)	Re-read CCV. If CCV fails a second time, a new initial calibration is required.
Method Blank	Determine source of problem, correct, and re-analyze.
Laboratory Control Sample (LCS)	Determine source of problem, correct, and re-analyze.
Matrix Spike (MS)	Check makeup of MS solution. If LCS passes, indicate matrix problem with sample.
Matrix Spike Duplicate (MSD)	Re-analyze. If LCS passes, indicate matrix problem with sample.

Appendix B

SEAL and Method Precision and Accuracy

Analyte	Method LOD (ug/L)	SEAL LOD	Method Accuracy %Rec	SEAL Accuracy* %Rec	Method Precision %RSD	SEAL Precision* %RSD
Cannabis	NA	9.68	***	102.3	***	5.0

*SEAL, accuracy, and precision calculated from LCS and MS/MSD from control charts.
**SEAL LOD was performed 2/12
***See Section 13.0

SEAL Proficiency Testing Results

Analyte	RTC PT (5/11)	% REC	Perf. Eval.	RTC PT (11/11)	% REC	Perf. Eval	RTC PT (5/12)	% REC	Perf. Eval

Cannabis	WP11-2	109	A	WP11-4	106	A	WP12-2	105	A

Revisions

Detection of Pesticides in Cannabis by LCMS

Revision

0

Effective Date

7/19/16

Prepared By

Southeast Alaska Laboratories, LLC

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99801

315-778-1469

APPROVED BY:

_____	_____
Laboratories Director	Date
_____	_____
Quality Assurance Officer	Date

1. SCOPE OF APPLICATION
2. SUMMARY
3. Reagents and Supplies
4. PROCEDURE
5. CALCULATONS
6. REFERENCES

1. SCOPE OF APPLICATION

This procedure is for the chromatographic determination of pesticide residues on *Cannabis* flower. If the method is applied on another matrix, this method will be updated.

2. SUMMARY

Liquid Chromatography (LC) with Mass Spectrometer (MS) will be used to test cannabis flower for the pesticides listed by the State of Alaska.

3. Reagents and Supplies

3.1 Glacial acetic acid
3.2 LC grade water (sample extraction)
3.3 LCMS grade water (mobile phase)
3.4 LCMS grade methanol (mobile phase)
3.5 LC or LCMS grade acetonitrile (sample extration)
3.6 Formic acid
3.7 Ammonium Acetate
3.8 50 mL Falcon tubes
3.91.5 mL eppendorf tubes
3.10 LCMS pesticide residue 10 part mix (Restek)
3.11 Internal standards from Rested
3.12 AOAC Quechers packets (Restek)
3.13 dSPE tubes (Restek)
3.14 LCMS Vials (one-piece high recovery or with inserts)
3.15Ultra AQ C18 column (Restek)
3.16 Pipettes, tips, and other basic lab supplies.

4. PROCEDURE

4.1 Prepare pesticide standard, final concentration 1,000 ng/mL. Pipette 900 uL acetonitrile into a 1.5 mL eppendorf tube. Add 10 uL of each part of the 10 part mix for a total volume of 1 mL. Mix, label and store in the freezer when not in use. (Suggested expiration 90 days, should be validated)

4.2 Pesticide standard, final concentration 5,000 ng/mL. Pipette 500 uL acetonitrile into a 1.5 mL eppendorf tube. Add 50 uL of each part of the 10 part mix for a total volume of 1 mL. Mix, label and store in the freezer when not in use. (Suggested expiration 90 days, should be validated)

4.3 Pesticide internal standard, final concentration 10,000 ng/mL. (Instructions TBD)

4.4 Acidified water for extraction: 1% acetic acid solution may be prepared by adding 10 mL glacial acetic acid to 990 mL LC or LCMS grade water. (Suggested expiration 30 days, should be validated)

4.5 Acidified acetonitrile for extraction: 1% acetic acid in acetonitrile may be prepared by adding 10 mL glacial acetic acid to 990 mL LC or LCMS grade acetonitrile. (Suggested expiration 30 days, should be validated)

4.6 Mobile phase (pump A) 5 mM ammonium formate with 0.1% formic acid. Discard old mobile phase and rinse mobile phase bottle with 100 mL LCMS grade water. Measure 1 L LCMS grade water into bottle. Add 385 mg ammonium acetate and allow to dissolve. Then add 1 mL concentrated formic acid and mix thoroughly. (Suggested expiration 7 days, no validation needed)

4.7 Mobile phase (pump B) 100% LCMS grade methanol may be poured directly into the mobile phase bottle. The methanol may be used indefinitely, however if only a little remains in the bottle, periodically it may be desirable to discard it and rinse the bottle with methanol.

4.8 Sample preparation
Pack not less than 1.5 grams of dried flower into a clean teflon grinding jar, then place the weighted teflon grinding ball inside and screw closed. Shake for 2 min at 30 Hz in the TissueLyzer II grinder. Pour out the material including the ball into a weigh boat and scrape the inside of the jar and the grinding ball with a spatula, recovering as much material as possible. Accurately weigh 1.5 grams +/- 20% of the milled material into a 50 mL Falcon tube and add 15 mL of water containing 1% acetic acid. Vortex and shake on shaker table for 30 minutes. Add 15 mL of acetonitrile containing 1% acetic acid. Any spiked samples should be spiked at this step. Vortex and shake for 30 minutes.

4.9 One calibration sample (500 ppb spike), one QC sample (50 ppb), one matrix blank, and one process blank should be prepared for each batch. The calibration, QC and matrix blank should be prepared using a pesticide-free organic cannabis sample, using a 1.5 gram test portion and prepared identically to an incurred sample, with the appropriate spiking as follows. The 500 ppb calibration sample should be spiked with 150 uL of the 5,000 ng/mL pesticide standard. The QC samples should be spiked with 75 uL of the 1,000 ng/mL pesticide standard. The volume differences between the spiked and unspiked incurred samples are 1% or less and may be ignored. The matrix blank should be prepared with no spiking and the process blank should consist of an empty tube, processed with extraction solvents.

4.10 Add the contents of one AOAC salts QuEChERS packet to each tube, one by one. Each packet should be snapped repeatedly to allow the powder to settle at the bottom, then opened and the contents dumped as quickly and cleanly as possible into the tube. Open each packet only at the moment of use. Packets contain overfill which need not be emptied entirely into the tube. The tube should be closed and shaken vigorously with vortexing as quickly as possible to prevent clumping of powder. Tubes should become warm to the touch. Shake and vortex as much as possible for 2 minutes. Centrifuge the tubes at 3000 rcf for 2 minutes. Remove tubes carefully from the centrifuge to avoid disrupting the acetonitrile (top) and water layers. Transfer 1 mL of the supernatant to a dSPE tube. Vortex each vigorously as before to ensure the entire contents of the tube are dispersed. Then centrifuge the tubes at 3000 rcf for 2 minutes. For the 500 ppb spiked sample and the process blank sample, two 1 mL aliquots should be prepared.

4.11 The following procedure is used to prepare the calibration curve using the left-over supernatant from the quechers extraction. Empty the contents of 5 dSPE tubes into a 15 mL falcon tube. Prepare a second 15 mL falcon tube in a similar way. Then transfer 5 mL acetonitrile supernatant from the matrix blank sample into each falcon tube, vortex and shake for 2 min and centrifuge at 3000 rcf for 2 min. Then remove the supernatants and combine in a fresh 15 mL falcon tube. Save this matrix blank for the calibration curve below.

4.12 From the prepared matrix blank aliquot 285 uL to 7 eppendorf vials labeled levels 2 through 8. To one tube labeled level 1, transfer 285 uL of the supernatant from one of the 500 ppb spiked samples. Transfer 285 uL of the other 500 ppb spiked sample to the level 2 tube. Then mix, then transfer 285 uL from level 2 to level 3 and so on using a 2-fold serial dilution until finished. From the final vial level 8,

discard 285 uL so the volume in each vial is the same. Finally, add 15 uL internal standard to each vial.

4.13 From all other prepared dSPE extracts transfer 285 uL of the supernatant to 1.5 mL eppendorf tubes. To each sample, spike 15 uL of internal standard solution.

In the case of post extraction spiking experiments, the spiking volume is 15 uL however instead of the internal standard solution, the post-extraction spiking solution is used. (TBD)

4.14 The vials should all contain a volume of 300 uL and should be dried down under a stream of nitrogen, or in a speedvac until the volume in each vial is approximately 50 uL. The vials should not be completely evaporated to dryness and the evaporation should be completed in no greater than 6 hours. After drydown, the samples should be closed and vortexed, then centrifuged to remove any particulates. The supernatant should be transferred to a high recovery insert and placed in an autosampler vial (alternatively a one-piece high recovery vial may be used). When transferring the supernatant, transfer only a part of the liquid to avoid transfer of particulates in the vial, about 25-50 uL is appropriate. The samples should be analyzed within 24-36 hours of preparation to avoid any chance of low recovery or sample precipitation in the vial (Should be validated, a longer window may be tested).

4.15 Instrument Setup.

Pump A (5 mM ammonium formate with 0.1% formic acid) and Pump B (methanol) mobile phases should be set up (see above for preparation). The column should be set up as well. Turn on the LC column oven, LC flow, and MS interface to their method settings and allow it to equilibrate for 15 minutes. Monitor the initial pump pressure rise rate at turn-on as well as at plateau to ensure the LC system is performing normally. If the pressure is unusual, investigate the cause and correct. Visually inspect the ESI spray to ensure it is stable. If any sputtering is observed, the ESI source should be inspected and corrected. The method conditions in the master method file should be used as the starting point for the method.

4.16 Batches will be named in accordance with protocols previously established.

4.17 Before running the batch verify the system suitability by running a system suitability batch. The batch consists of two blank runs

followed by duplicate injections of a 100 ng/mL standard in solution of pesticides, and finally two blank injections. This step may be carried out as much as 24 hours in advance, before sample preparation begins, to ensure the system is ready for analysis. The retention time, peak shape, peak width, and ion ratios should be inspected to ensure correct system performance. If the retention times deviate noticeably, update the retention times. A separate written procedure is to be used for this step. If the peak shapes or widths become unacceptable over time, method performance may be reduced and the LC column should be replaced. After replacing the LC column, the new retention times should be checked and adjusted, and the peak shapes, widths, and other quality indicators verified.

4.18 The signal intensity of a subset of analytes should be checked against typical values to ensure system performance as well. Decreased signal intensity is typical over time as the instrument becomes dirty, and a margin of safety is built into method performance requirements to account for this phenomenon. However when the signal intensity decreases beyond a threshold level the desired minimum performance can no longer be achieved and an intervention is required to investigate and possibly clean the MS.

4.19 After passing a system suitability test, the batch may be run. As previously mentioned the batch should be started no more than 24 hours after a successful system suitability test. The batch should begin with at least one blank run, then one replicate of the 50 ppb QC sample, then the calibration curve in ascending order, another 50 ppb QC injection, followed by a blank, then the incurred samples. The 50 ppb QC sample should be run at the end of the batch, and if more than 10 incurred samples are analyzed, the 50 ppb QC sample should be run at least every 10 incurred samples, before and after a blank.

4.20 If the batch does not fully complete, investigate and fix the cause of the fault and run the uncompleted portion of the batch if less than 24 hours have elapsed since the batch was started and if the cause of the fault was due to a simple cause such as a clog or typographical error. If the fault requires intervention such as replacement of the LC column or intervention to the MS, the entire batch should be re-run after system suitability check, keeping in mind that samples should generally be run within 24-36 hours after extraction (to be validated).

4.21 After the batch is completed, the files should be reviewed completely. Load the files into the LabSolutions browser window and integrate the chromatograms with the data processing settings

included in the method. It is recommended to spot-check retention times, peak shapes and widths, signal intensities etc. as in the system suitability test. Next, 100% of chromatograms should be reviewed to verify proper integration and peak identification. This step can be done rapidly but systematically to ensure 100% review. Peak integration should normally be perfomed by software to minimize operator bias, however in some cases the peak integration may be performed manually if there is a justifiable cause. Peak identification is normally performed automatically as well, however if the signal-to-noise ratio, ion ratio, or other peak settings are clearly in or out of range the peak identifications can be manually set or rejected as necessary.

4.22 After peak integration and identification, verify correct appearance of the blank samples, compare the replicates of the 50 ppb QC sample throughout the run and ensure they remain within +\- 15%. The calibration curve should also appear as expected. Any peaks representing positive detections in incurred samples should be carefully checked against authentic standards. If any anomalies are detected in the data review stage, the cause should be investigated. If a simple error (such as a typographical error not affecting the final result) has occured the corrected batch may be accepted. However if the fault could have affected the accuracy of the analysis, the batch should be rejected, the fault corrected, and the samples re-analyzed.

5.0 CALCULATIONS

5.1 Calculations are performed per test method.

6.0 REFERENCES

6.1 Standard Methods 19th Edition, method 1020 B Quality Control, section 1.

Control #_____

xxx.xx

Manual Integration

Revision
1

Effective Date
7/19/16

Prepared by
Southeast Alaska Laboratories, LLC

5450B Jenkins Dr., Juneau AK

99801

315-778-1469

APPROVED BY

_____	_____
Laboratory Director	Date
_____	_____
Quality Assurance Officer	Date

1.0 Scope and Application
2.0 Summary
3.0 Terms and Definitions
4.0 Quality Control
5.0 Procedure
6.0 References
7.0 Appendix
8.0 Revisions

1. SCOPE AND APPLICATION

1.1. This document describes the correct procedures to ensure that manually integrated peaks using chromatographic software are legally defensible. Analysts use these procedures when working with Gas Chromatography (GC), High Performance Liquid Chromatography (HPLC), Gas Chromatography/Mass Spectrophotometer (GC/MS), or Ion Chromatography (IC).

1.2. In an effort to assure data integrity within the laboratory and to limit liability from data that may have been inappropriately manipulated whether intentionally or unintentionally, Southeast Alaska Laboratory has developed a laboratory system to ensure ethical documentation, training, and quality assurance practices are being conducted.

1.3. This SOP is used in addition to any method-specified requirements.

2. SUMMARY

2.1. All data must be integrated consistently in calibration standards, calibration verification standards, samples, and QC samples. Manual integration is only done when justifiable (i.e, integration software misses peak, etc.). The manually integrated peaks must include a technical justification written on the chromatogram. The manually integrated peak must be clearly defined by circling the peak and initialed by the analyst. The original chromatogram and the manually integrated chromatogram are kept in data package for peer and QC review.

3. TERMS AND DEFINITIONS

3.1. Manual Integration – The process by which the automatically generated output of the data handling system of an analytical

instrument (i.e. the calculation of peak area leading to a determination of concentration) is over ridden by an analyst, who adjusts a parameter used in making the determination of peak areas.

4. QUALITY CONTROL

4.1. The Quality Assurance Officer will examine chromatographic data during annual internal audits.

5. PROCEDURE

1. **Proper Manual Integration Practices**: All data must be integrated consistently in calibration standards, calibration verification standards, samples, and QC samples. Integration parameters –both automated and manual-must be technically justifiable and also adhere to valid scientific chromatographic principles. Manual integration is employed to correct improper integration performed by the data system. Manual integrations are performed by visually assessing peak symmetry and comparing the integration performed in the initial calibration with QC and samples. On the occasion that an interfering peak is co-eluting with the peak of interest, the analyst must estimate as accurately as possible where the target peak is likely to end (or begin) and adjust the integration to reflect the correct area of the peak. The analyst must also pay particular attention to integration problems encountered for the analysis of standards or blanks, which, in theory, should be free of contamination and interfering peaks. Unusual baseline characteristics, unidentified peaks, splitting peaks, excessive tailing or other problems may indicate a need for instrument maintenance or other corrective actions. Examples of data system integrations that may need to be manually corrected include: (See Appendix A for examples)

1.1. **Undetected Peaks**: A shift in retention times due to matrix or compound interferences in the sample may cause a data system to exclude (or include) chromatographic peaks. Analysts must review peaks to ensure proper peak identification.

5.1.2 **Incorrect Peak Integration**: Peaks that are very similar in retention times and quantitation ions, or are irregular in shape (i.e. split peaks), can be easily misidentified by the data system. An analyst must carefully review all

peaks, especially peaks that are known to have these characteristics (i.e. benzo (b) and (k) fluoranthene).

5.1.3 **Baseline Corrections**: Highly contaminated samples can cause matrix interference that adversely affects the baseline of target compounds.

1.4. **Peaks Identified Incorrectly**: A data system's method integration parameters may not be fully optimized resulting in the misidentification of chromatographic peaks. Analyst must ensure the data system operates with minimal operator intervention and also demonstrate the methods parameters are set correctly by carefully evaluating the results of calibration standards prior to analyzing samples.

2. **Improper Manual Integration Practices**: Under no circumstances should manual integration be performed solely for the purpose of making failed calibration, surrogates, or internal standards appear to meet quality control (QC) criteria. Practices such as peak shaving, peak enhancement, or manipulations of the baseline to achieve these ends must never occur as the result of an improper integration rather than correcting a data system error. See appendix B for examples. Southeast Alaska Laboratory has adopted a "zero tolerance philosophy" toward any improper, unethical, or illegal actions by any employee. Improper manual integration does fall under Southeast Alaska Laboratory ethics violations. If an employee has intentionally committed improper manual integration, they can be fired, and prosecuted for laboratory fraud.

5.2.1 **Peak Shaving**: Intentionally removing peak areas that are inconsistent with valid scientific chromatographic principles and the integration technique performed on the initial calibration and QC samples is an unacceptable manual integration practice.

1. **Peak Enhancement**: Improperly enhancing peak areas by combining multiple peaks to produce a value for a single compound that is inconsistent with valid scientific chromatographic principles and the integration technique performed on the initial calibration with QC and samples is an unacceptable manual integration practice. While peaks at low concentrations combined with a noisy baseline may result in the appearance of peak

enhancement even when the intent may be unintentional, data must be rejected if the chromatogram is not clear enough that peak enhancement can be conclusively ruled out.

2. **Baseline Manipulation**: Artificially dropping a baseline to increase area amount that is inconsistent with valid scientific chromatographic principles and the integration technique performed on the initial calibration with QC and samples is an unacceptable manual integration practice.

7. **General Peak Identification Practices**: There are some basic peak identification rules that should be followed across all analytical instruments.

   7.1. Peak must be assessed against a signal-to-noise of approximately 3:1.

   7.2. Peak retention time is critical in identifying possible interference and/or degradation.

   7.3. Chemical reasonableness should be used when evaluating the data. This includes historical handling of analytes, known co-elutions, pattern recognition, and analyst's discretion. However, do not force a peak to be selected if the basic response is not met.

8. **Documentation**: Manual integration is employed to correct an improper integration performed by the data system and must include a qualifier clearly stating that a manual integration was performed.

   8.1. Documentation for GC, HPLC, and IC: Both the original and manually integrated chromatogram must be present in the data package for peer and QC review. The manually integrated peak must be clearly defined by circling the peak and initialed by the analyst.

   8.2. Documentation for GC/MS: The "m" footnote next to the compound is initialed and dated on the manually integrated quantitation report. The original chromatogram and the chromatogram of the manually integrated peak is provided in the data package for peer and QC review.

8.3. All manually integrated peaks must include a technical justification noted in the data packet. This can be on each chromatogram or as a general note on the sample run log.

8.4. All manually integrated peaks are identified in the case narrative if required as a data deliverable by the client.

9. REFERENCES

9.1 EPA Region Laboratory SOP 835 CHROMATOGRAPHIC INTEGRATION PROCEDURES summary, www.epa.gov/region09/lab/sops/sop835.html.

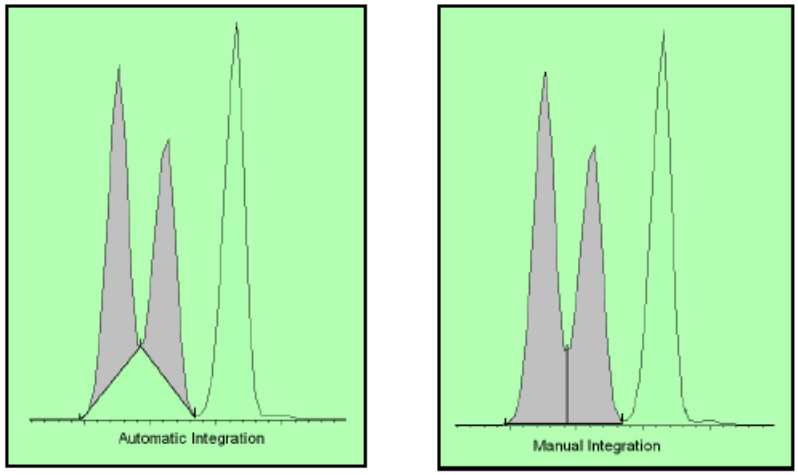
9.2 Battelle Duxbury Operations Standard Operating Procedures for Manual Integration Practices and review.

9.3 DataChem Laboratories, Manual Integration on GC, HPLC, IC, LC/MS, and GC/MS Data Systems.

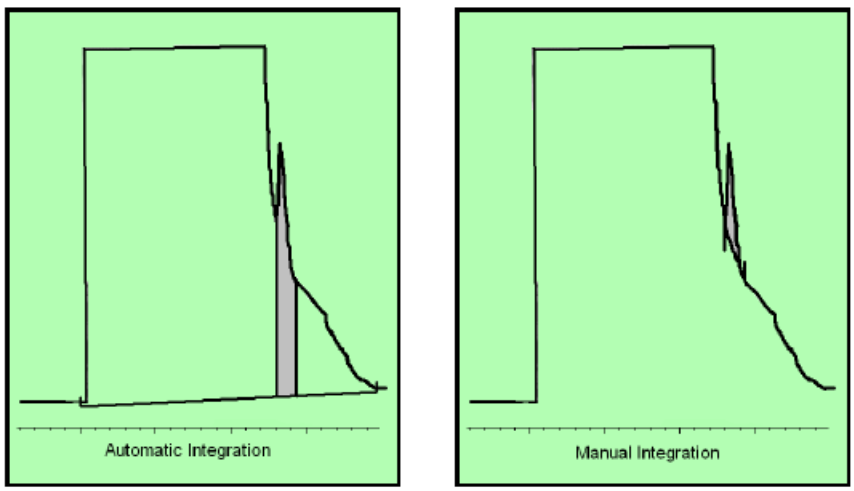
10. APPENDICES

Appendix A

Common Problems



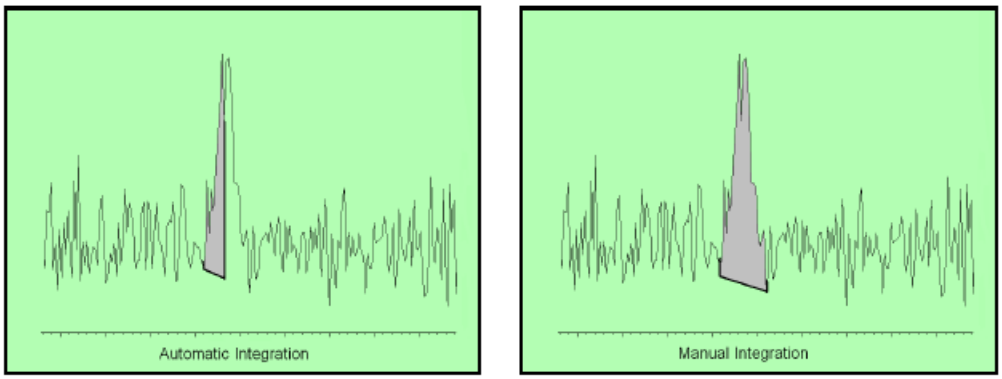
Chromatography Problem 1: Split Peaks



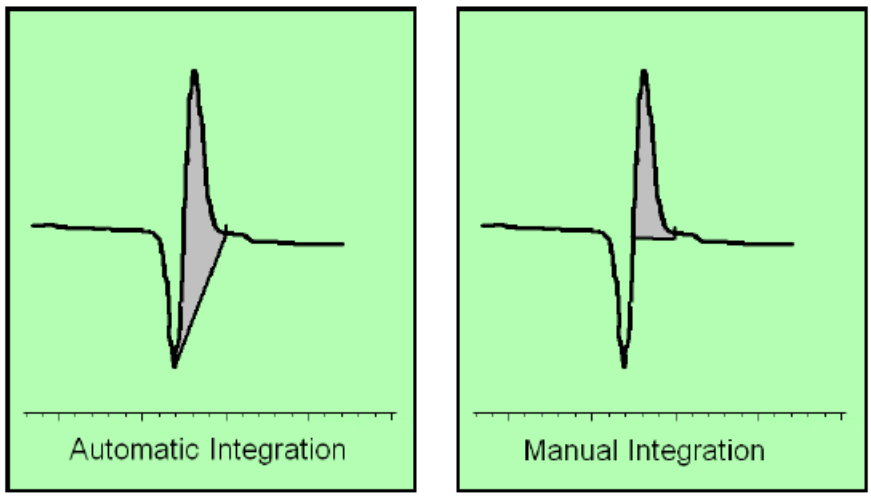
Chromatography Problem 2:
Coelution of Target Compounds/Shoulders

Appendix A Continued

Common Problems



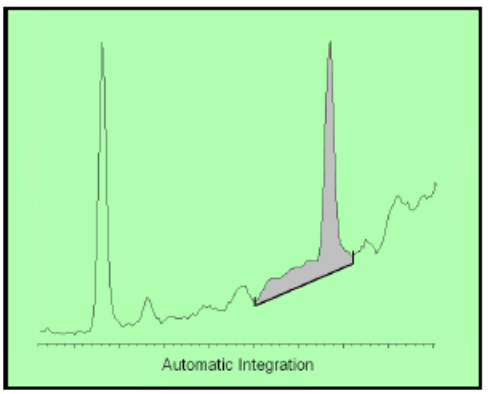
Chromatography Problem 3: Baseline Noise

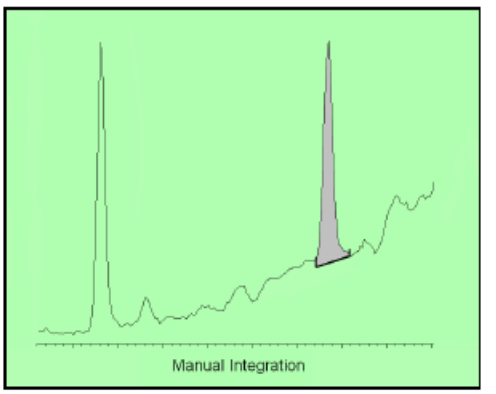


Chromatography Problem 4:
Negative Peaks

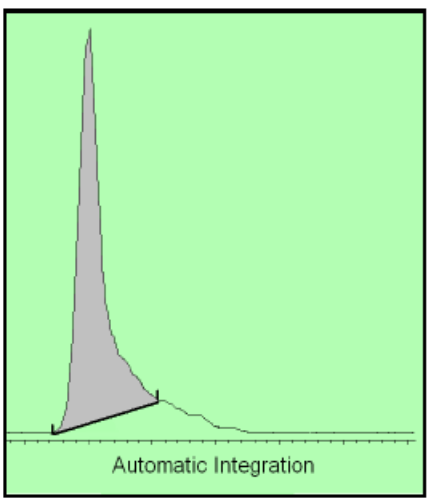
Appendix A Continued

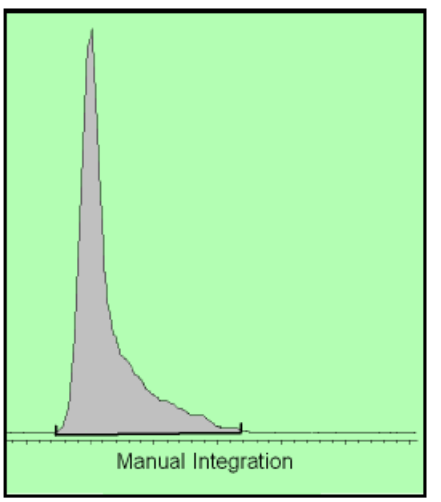
Common Problems





Chromatography Problem 5:
Rising or Falling Baselines

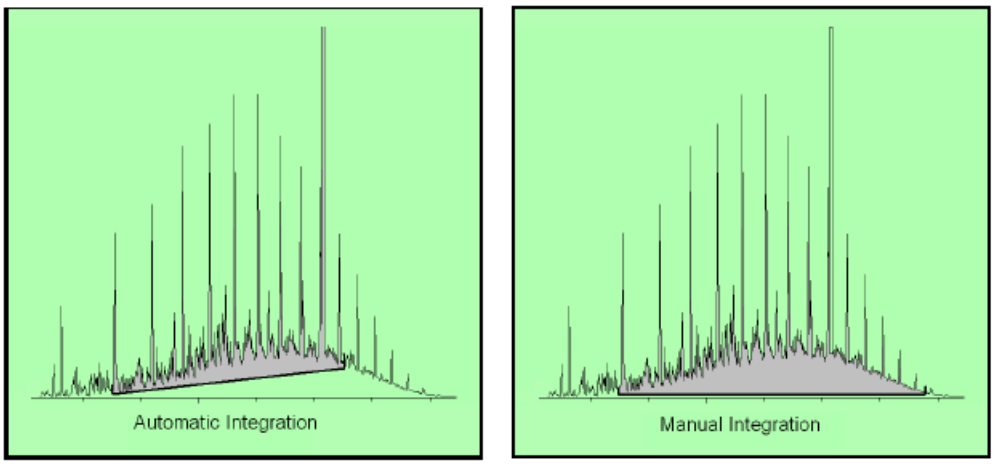




Chromatography Problem 6:
Excessive Peak Tailing

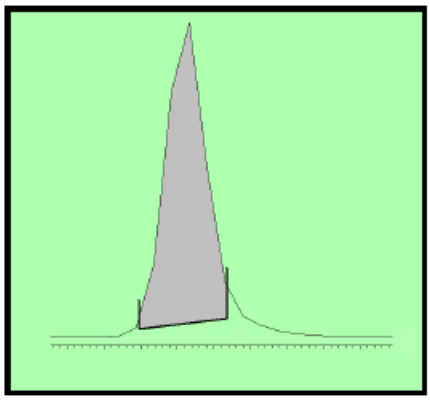
Appendix A Continued

Common Problems

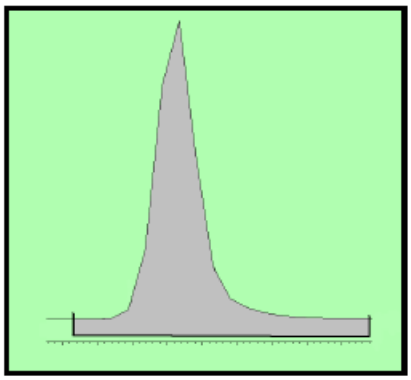


Chromatography Problem 7:
Integration of Hydrocarbons

Appendix B
Incorrect Manual Integration

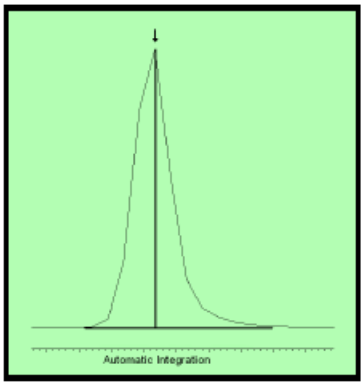


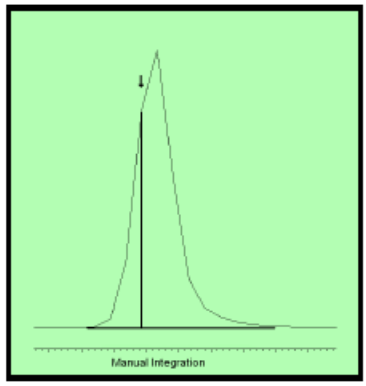
Removing Area from the Peak a.k.a. “Peak Shaving”



Adding Area to the Peak a.k.a. “Peak Enhancing”

Appendix B
Incorrect Manual Integration





Changing Peak Height

Control # _____

OG-0000.00

Determining Retention Time Windows

Revision 0

Effective Date

7/19/16

Prepared by

Southeast Alaska Laboratories, LLC

5450B Jenkins Dr., Juneau AK

99801

315-778-1469

APPROVED BY:

_____

Laboratory Director Date

_____

Quality Assurance Officer Date

1. SCOPE AND APPLICATION
2. SUMMARY
3. PROCEDURE
4. REFERENCES
5. APPENDIX

1. SCOPE AND APPLICATION

1.1. Retention time windows are crucial to the identification of target compounds.

1.2. Absolute retention times are used for compound identification in all GC and HPLC methods that do not use internal standard calibrations.

1.3. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability.

1.4. The width of the retention time window should be careful established to minimize the occurrence of both false positive and false negative results. Tight retention time widows may result in false negative and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.

1.5. The retention time window method is for GC and HPLC procedures using non-MS or FTIR detection. Identification procedures are different for GC/MS (e.g. Methods 8260 and 8270).

2. SUMMARY

2.1. Standards for each compound of interest are analyzed three times in a 72-hour period. The standard deviation is calculated, and the retention time window is determined by multiplying the standard deviation by three.

3. PROCEDURE

3.1. Make sure the chromatographic system is operating reliably and that the system condition have been optimized for the target analytes and surrogates in the sample matrix to be analyzed.

3.2. Make three injections of all single component standard mixtures and multi-component analytes (such as PCB's) over the course of a 72-hour period. Serial injection or injections over a period of less than 72-hours may result in retention time windows that are too tight.

3.3. Record the retention time (RT) for each single component analyte and surrogate to three decimal places (e.g., 0.007). Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, choose three to five major peaks and calculate the mean and standard deviation of those peaks. See calculations below:

$$\text{Mean RT } (x) = \frac{\text{Sum of RT}}{\text{Total Number of RT's}}$$

$$\text{Standard Deviation} = [\sum (x-x_1)/n-1]^{1/2}$$

x = Mean RT
x_1 = RT Result
n = The number of repeated RT's

3.3.1. Use spreadsheet (Appendix A) to calculate retention time windows. N:/Excel/SEAL Quality Assurance/RT Window Calc.

3.4. If the standard deviation of the retention times for a target compound in 0.000 (i.e., no difference between the absolute retention times), then collect data form additional injections of standards, or use a default standard deviation of 0.01 minutes.

3.5. The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute of the mean absolute retention time established during the 72-hour period. If the default standard deviation in section 3.4 is used, the width of the window will be 0.03 minutes.

6. Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

7. Absolute retention time windows must be calculated for each analyte and surrogate on each chromatographic column and instrument. New retention time windows must be established when a new GC column is installed.

8. If the instrument data system is not capable of using compound specific retention time windows, then the widest window is chosen and applied to all compounds.

9. The surrogates are added to each sample, blank, and QC sample and are also contained in each calibration standard. Although the surrogates may be diluted out of certain sample extracts, their retention times in the calibration standards may be useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window, the cause of the problem must be corrected before continuing analyses.

4. REFERENCES

4.1. Test Methods for Evaluating Solid Waste Physical/Chemical (SW-846), Method 8000 section 7.6.

APPENDIX A

RETENTION TIME WINDOW CALCULATION SPREADSHEET

Dates:_____ Method:_____

Analyst:_____ **Instrument:_____**

Column:_____

Compound	**Retention Time #1**	**Retention Time #2**	**Retention Time #3**	**Mean (Minutes)**	**Standard Deviation (SD)**	**Calculated Retention Time Window (SD*3) (Minutes)**

Control #_____
GL-0000.00

BALANCES AND WEIGHTS

Revision

1

Effective Date
7/19/16

Prepared by

Southeast Alaska Laboratories, LLC

5450B Jenkins Dr., Juneau AK

99801

315-778-1469

APPROVED BY:

_____	_____
Laboratory Director	Date
_____	_____

1.0 Scope and Application
2.0 Summary
3.0 Interferences
4.0 Apparatus and Materials
5.0 Quality Control
6.0 Calibration and Standardization
7.0 Procedure
Appendix
Revisions

1. SCOPE AND APPLICATION

1.1 This method describes the procedure followed by Southeast Alaska Laboratory for the maintenance and calibration of balances and weights are calibrated every year by a certified technician against the ASTM Class 1 requirements.

2. SUMMARY

2.1 Analytical and top-loading balances are calibrated annually by a certified technician.

2.2 Each balance is verified daily with class S weights. The tolerance levels are 0.01% for the analytical balances and 1.0% for top-loading balances. If balances or weights are not within tolerance criteria they are not used until a certified technician can repair them.

3. INTERFERENCES

3.1 Drafts caused by heating and air conditioning systems or other means may cause variance in the weight reading. All top-loading balances must be protected from drafts during use. The doors of the analytical balance must be closed during readings.

2. The weights are contained in a case supplied with tweezers and gloves. Both gloves and tweezers must be utilized when handling the weights. DO NOT USE YOUR BARE HANDS TO HANDLE THE WEIGHTS. DO NOT ALLOW THE WEIGHTS TO COME INTO CONTACT WITH ANY SUBSTANCE THAT WILL CLING TO THE

WEIGHT. If necessary, clean the weights with cotton soaked in alcohol and notate in logbook that the weight was cleaned.

4. APPARATUS AND MATERIALS

4.1 Scale Model here.
4.2 Class S-Weights (Reference Weights)-1 mg to 100 g
4.3 Class 1 Weights (Working Weights).

Weight	Serial Number
1 mg	
10 mg	
1 g	
10 g	
20 g	
100 g	

5. QUALITY CONTROL

5.1 All balance surfaces are cleaned prior to use.

5.2 All balances are protected from potential drafts.

5.3 Top-loading and analytical balances will be calibrated and cleaned by a certified technician (Quality Control Services) annually. Labels are attached to each balance indicating the date of last calibration. If the technician during the annual service cannot calibrate a balance, the balance must be labeled to indicate it cannot be used until properly repaired.

4. The accuracy of each balance is checked against Class 1 (working weights) prior to use. The acceptance limits for the weights used at SEAL are found in Table 1. If balances do not meet the criteria, adjustments may be made according to the balance operations manual. If the balance still fails to calibrate then a certified technician is called in to service and/or repair the balance. It must be labeled "Balance not calibrating" with date out-of-service. Contact the Laboratory Director or Quality Assurance Officer immediately if a balance fails to calibrate.

5. The checks are recorded in the specific balance logbook located at each balance. The balance ID is listed on the cover of the logbook and the current weight se in use is listed directly in the logbook.

6. Class S weights are calibrated annually against NIST requirements by a certified technician from Quality Control Services. If the class S weights are out of acceptance limits then the weights must be replaced. The class S weights are only used to check the accuracy of the working weights.

7. The class 1 weights (working weights) are checked for accuracy each month against the class S weights (reference weights). See Table 2 for acceptance limits. If the weights are not within acceptable ranges then they must be replaced.

8. The Quality Assurance Officer routinely checks (during quarterly audit) the balance list to make sure that the assigned weights and balances have not been moved, and that the balance check logbook is current and proper corrective action has been taken. The Quality Assurance Officer will notate on the next available space that the logbook has been reviewed and will initial and date the entry. Also, each balance will be verified against the full range of weights available as appropriate for each balance. The verification will be listed in the logbook and the quarterly audit report.

6. CALIBRATION AND STANDARDIZATION

6.1. Calibrate each balance as needed according to manufacturer's instructions.

7. PROCEDURE

7.1 Analytical Balance: The calibration of the analytical balance is verified every day of use with three weights, 10 mg, 1 g, and 100 g. These weights bracket the actual range of weights recorded during typical use. The acceptance limits for the weights are found in Table 1.

7.1.1 Using a brush or kimwipe, clean off the pan, removing all objects.

7.1.2 Check that the balance is level. If the balance required adjustment, note in the comments of the logbook.

3. With the doors closed, assure that the balance is properly zeroed before checking. If the balance is not zero, the balance has been moved, or the power has been shut off. Re-calibrate the balance according to manufacturer's instruction's, and if the balance zero's, proceed. If it will not zero then certified technician is called, and the balance is not used. See section 6.4.

7.1.4 Place the 10 mg weight on the pan, close the door, allow stabilizing, and record the observed weight.

5. Follow 7.1.4 with both the 1g and 100 g weights.
6. If the weights do not meet the listed acceptance criteria, first clean the weight with cotton soaked in alcohol and re-weigh. If the weight is still not within tolerance, re-calibrate, and re-check weights. If the weight is still outside the acceptable range, note in the comment section, do not use the balance and notify the Laboratory Director or Quality Assurance Officer.

7.2 Top Loading Balance: The top loading balance is verified every day with two weights, 10 g and 100 g. These weights should bracket the actual range of weights recorded during typical use. The acceptance limits for these weights are found in Table 1.

7.2.1 Using a brush or kimwipe, clean off the pan, removing all objects.

7.2.2 Check that the balance is level. If the balance required adjustment, note in the comments of the logbook.

3. With the doors closed, assure that the balance is properly zeroed before checking. If the balance is not zero, the balance has been moved, or the power has been shut off. Re-calibrate the balance according to manufacturer's instructions, and if the balance zero's, proceed. If it will not zero then certified technician is called, and the balance is not used. See section 6.4.

4. Place the 10 g weight on the pan, close the door, allow to stabilize, and record the observed weight.

5. Follow 7.2.4 with the 100 g weights.

6. If the weights do not meet the listed acceptance criteria, first clean the weight with cotton soaked in alcohol and re-weigh. If the weight is still not within tolerance, re-calibrate, and re-check weights. If the weight is still outside the acceptable range, note in the comment section, do not use the balance and notify the Laboratory Director or Quality Assurance Officer.

8. APPENIDIX

TABLE 1

Acceptance Criteria for Balance Verification Using Working Weights

Weight	**Acceptance Range Analytical Balance Tolerance = ±.01%**	**Acceptance Range for Top Loading Balance Tolerance = ±1.0%**
0.010 g	±.0001 g	--
1 g	±0.0001 g	--
10 g	±.001 g	±0.1 g
100 g	± .01g	±1.0 g

TABLE 2

Acceptance Criteria for Working Weight Verification

Working Weights vs. Class S weights

Weight	**Acceptance Range for Working Weights**
0.010 g	±0.0001 g
1 g	±0.0001 g
10 g	±0.001 g
100 g	±0.01 g

Pyrethroid ELISA (Magnetic Particle Format)

Revision
0

Effective Date
7/19/16

Prepared by

Southeast Alaska Laboratories, LLC

5450B Jenkins Dr., Juneau AK

99801

315-778-1469

APPROVED BY:

_____	_____
Laboratory Director	Date
_____	_____
Quality Assurance Officer	Date

1.0 Scope and Application
2.0 Summary
3.0 Interferences
4.0 Procedure
Appendix Table
Revisions

1. SCOPE AND APPLICATION

The assay range is between 1.0 ppb and 15.0 ppb. The assay sensitivity allows the determination of Pyrethroids in a range of environmental samples (water, soil, sediment, fish plasma, etc.).

2. SUMMARY

Pyrethroids are a group of man-made pesticides similar to the natural pesticide pyrethrum, which is produced by chrysanthemum flowers. Pyrethroids are important insecticides used in agriculture, forestry, horticulture, and in household pesticides, pet sprays and shampoos. Some pyrethroids are also used as lice treatments that can be applied directlty to the head, and as mosquito repellents that can be applied to clothes. Pyrethroids usually work as contact insecticides, damaging the nervous system of insects which come into contact with it, leading to their death or "knockdown". Permethrin is bound tightly to soils and it has a half life of 3 to 6 weeks in soils. Permethrin can also be found in surface waters and when applied to control insects concentrations up to 0.8 mg/L have been reported. The WHO has a health- based advisory of 20 ppb for Permethrin in drinking water. This ELISA test kit detects Permethrin and related Pyrethroids in environment samples at the ppb levels.

3. INTERFERENCES

3.1 The antibody binds Permethrin and related Pyrethroids and does not cross-react with other non-related agricultural compounds.

4. PROCEDURE

4.1 Add 250 uL of sample and 500 uL of antibody coupled magnetic particles. Vortex.

4.2 Incubate for 20 minutes.

4.3 Add 250 uL of conjugate. Vortex and incubate for 30 minutes.

7.4 Separate using the magnetic separator, decant and wash.

7.5 Add 500 uL of color solution.

7.6 Incubate 30 minutes.

4.4 Stop the reaction and read color at 450 nm. Quantitate results.

5. APPENIDIX

Cross-reactivity of the Abraxis Pyrethroid ELISA expressed as the least detectable dose (LDD) which is estimated at 90% B/Bo and at the concentration required to displace 50% (50% B/Bo).

LDD 50% B/Bo

Compound (ppb) (ppb)

Permethrin

Lamba (λ) Cyhalothrin

Cypermethrin

Bifenthrin

Resmethrin

Cyfluthrin

Tetramethrin

0.750 4.25

9.2 89.5 4.75 100 13.5 150

200 2,400 220 3,400 >1,000 >10,000 170 1,700

3, PBA _____

The following compounds demonstrated no reactivity in the Prethroid Assay when tested at concentrations up to 1,000 ppb: aldicarb, aldicarb sulfoxide, aldicarb sulfone, alachlor, atrazine benomyl, butachlor, butylate, captan, carbaryl, carbendazim, carbofuran, 2,4-D, 1,3-dichloropropene, dinoseb, MCPA, metolachlor, metribuzin, PCP, picloram, propachlor, terbufos, thiabendazole, thiophanate-methyl.

Organophosphate/Carbamate ELISA

Control #_____
MI-0000.00

Organophosphate/Carbamate ELISA
Revision
0

Effective Date
7/19/16

Prepared by

Southeast Alaska Laboratories, LLC

5450B Jenkins Dr., Juneau AK

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315-778-1469

APPROVED BY:

Laboratory Director	Date
Quality Assurance Officer	Date

1.0 Scope
2.0 Summary
3.0 Interferences
4.0 Reagents
5.0 Assay Procedure
6.0 References

1.0 Scope

1.1 For the detection of a wide range of organophosphates including thiophosphate, and carbamate pesticides in water, (drinking water, ground water, surface water and well water). This plate assay can also be used for testing collected dislodgeable residues from a surface wash, as well as pesticide residues prepared from dried extracts.

2.0 Summary

2.1 The test is a qualitative, colorimetric assay (modification of the Ellman method) for the detection of organophosphates and carbamates, based on a modification of their inhibition of the enzyme Acetyl Cholinesterase (ACh-E). ACh-E hydrolyzes acetylthiocholine (ATC), which reacts with 5,5'-Dithio-bis(2-Nitrobenzoic Acid) [DTNB] to produce a yellow color which is then read at 405 nm. If OP or Carbamate pesticides are present in a sample, they will inhibit ACh-E reducing or eliminating color formation depending on their concentration.

3.0 Interferences

3.1Detection limits of the various OP/C pesticides differ depending on their ability to inhibit the enzyme (refer to Sensitivity table). If it has been

established that only a single OP/C is present, the test can be used in conjunction with appropriate standards for quantitative testing.

4.0 Reagents

4.1 Microtiter plate with removable 8 strips of 12 wells and a stripholder.
4.2 1 vial (blue dot), used as diluent for the substate (ATC), 5 ml.
4.3 1 vial (green dot), used as diluent for the lyophilized ACh-E, 3 ml.
4.4 1 Amber vial with 1 ml of positive pesticide control-5 ppb Diazinon in 50% MEOH
4.5 1 Amber vial (orange fluorescent dot), 1 ml of OXIDIZER .
4.6 Assay Buffer and Oxidizer Diluent (orange fluorescent dot), 10 ml.
4.7 1 vial (red dot), 3 ml.-NEUTRALIZER
4.81 vial (green dot) ACh-E, lyophilized
4.9 1 vial (blue dot), SUBSTRATE, ATC, lyophilized 1 vial (yellow fluorescent dot), CHROMOGEN (DTNB) 3 ml.
4.10 1 vial (purple dot),STOPPING SOLUTION, 3 ml. 1 Amber vial with 1ml of negative pesticide control-50% MEOH.with and equal volume of methanol upon collection and before testing.

(2) Samples may be prepared as dry extracts (solvent evaporated residues) or as residues dislodged from surface washes (see Sample Preparation under Assay Procedure). Other samples may require modifications to the procedure and should be validated.All samples to be analyzed should be in a 50% MEOH matrix.
(3) Samples containing gross particulate matter should be filtered (e.g. 0.2 um AnotopTM 25 Plus, Whatman, Inc.) to remove particles.
Pigmented samples may obscure color and cause some interferences, therefore a negative control should be prepared in a similar matrix.

Reagent Preparation

All reagents must be allowed to come to room temperature.
(1) ACh-E – Add the 3 ml of the ACh-E diluent to the lyophilized enzyme, cap vial and mix by shaking moderately. Allow at least 5 minutes for the ACh-E to go into solution before use in the assay.
(2) Oxidizer –Determine the amount of diluted oxidizer needed for the assay. Dilute the oxidizer (1 part oxidizer to 9 parts oxidizer diluent) and mix by shaking moderately. This diluted oxidizer must be made fresh for each assay.
(3) Substrate (ATC) – Add 3 ml of ATC diluent to the lyophilized ATC, cap the vial and mix by shaking moderately.

Procedural Notes and Precautions

Add reagents directly to the bottom of the well while avoiding contact between the reagents and the pipet tip. This will help assure consistent quantities of reagent in the test mixture.

Avoid cross-contaminations and carryover of reagents by using clean pipet tips for each sample addition and by avoiding contact between reagent droplets on the wells and pipet tips.

5.0 Assay Procedure

1. Add 50 ul of assay buffer (orange fluorescent dot) to microwells to be used in the assay.
2. Add25uLoftheappropriatecontrolorsampleto designated assay wells, swirl plate to mix -15 seconds.

Well
Number Contents of Tube _____
A1,2 Negative Control A3,4 Positive Control A5,6 Sample 1
A7,8 Sample 2
B1,2 Sample 3 B3,4 Sample 4 B5,6 Sample 5
B7,8 Sample 6 _____
1 Microtiter plate with removable 8 strips of 12 wells and a stripholder.
1 vial (blue dot), used as diluent for the substate (ATC), 5 ml.
1 vial (green dot), used as diluent for the lyophilized ACh-E, 3 ml.
1 Amber vial with 1 ml of positive pesticide control-5 ppb Diazinon in 50% MEOH
1 Amber vial (orange fluorescent dot), 1 ml of OXIDIZER .
Assay Buffer and Oxidizer Diluent (orange fluorescent dot), 10 ml.
1 vial (red dot), 3 ml.-NEUTRALIZER
1 vial (green dot) ACh-E, lyophilized
1 vial (blue dot), SUBSTRATE, ATC, lyophilized 1 vial (yellow fluorescent dot), CHROMOGEN (DTNB) 3 ml.
1 vial (purple dot),STOPPING SOLUTION, 3 ml. 1 Amber vial with 1ml of negative pesticide control-50% MEOH.

5.1 Results

The negative control and any sample that has no detectable organophosphate or carbamate will develop a dark yellow color. Any sample with a detectable organophosphate or carbamate residue will have a reduced color development compared to the negative control. A 20% inhibition of color indicates the presence of a organophosphate or carbamate at or above the limit of detection (please refer to sensitivity table).
NOTE: If the negative control does not result in a yellow color, the test is invalid and should be repeated.

5.2 Limit of Detection Pattern (Sensitivity)

Limit of Detection of the Abraxis OP/Carbamate Test is estimated at 20% inhibition (IC 20) of color development Compound in 50% MEOH Organophosphate PPB Azinphos methyl 0.3 Chlorpyrifos methyl 0.4 Chlorpyrifos ethyl 0.5 Diazinon 0.6 Dichlorvos 0.5 Dicrotophos 2.4

Disulfoton 40 Ethion 0.6 Malathion 1.2 Parathion 0.8 Phorate 1.0 Phosmet 1.2 Carbamates PPB Aldicarb 25 Carbaryl 206 Carbafuran 0.9 OP/Carbamate

5.3 Assay Procedure (Part 2)

Add 25 ul of diluted Oxidizer into assay each wells, swirl plate to mix-15 seconds. Incubate for 5 minutes at 70o F +/- 20 degrees
Add 25 ul of Neutralizer (red dot) into each assay well, swirl plate to mix-15 seconds.
Add 25 ul of ACh-E (green dot) into each assay well, swirl to mix plate-15 seconds. Incubate 15 minutes at 70° F +/- 20 degrees.
Add 25 ul of Substrate-ATC (blue dot) into assay each well, swirl plate to mix-15 seconds.
Add 25 ul of Chromogen – DTNB (yellow fluorescent dot) into each assay well, swirl plate to mix-15 seconds. Incubate 30 minutes at 70° F +/- 20 degrees
Add 25 ul of Stopping Solution (purple dot) into each assay well, swirl plate to mix-15 seconds. Read at 405 nm (optimum wavelength) or 450 nm. Be sure no bubbles are viscible in any well. The bubbles cause erroneous readings.

Reagent Storage and Stability
Store all reagents at 2-8°C. Reagents may be used until the expiration date on the box.
Consult state, local and federal regulations for proper disposal of all reagents.

Materials Required but Not Provided
In addition to the reagents listed above, the following items are essential for the performance of the test:
Precision pipets capable of delivering 25-1000 ul and tips Microplate* or strip reader capable of reading absorbance between 405-450 nm
Test tube or vial to use for diluting of Oxidizer
* Please contact Abraxis for supplier information.

Sample Information
(1) This procedure is recommended for use with samples in a matrix of 50% MEOH. Other sample matrices may require modifications to the procedure and should be thoroughly validated (contact Abraxis Technical support for information and guidance). If testing water samples, the samples must be diluted

6.0 References

Precision

Three pools were spiked with an organophosphate pesticide at various levels and then assayed using the Abraxis OP/Carbamate Plate Assay. The following results were obtained when assayed in duplicate and run five times in each of 15 assays.

Between Assay Precision

Pool 1 (ppb)

X 0.51 SD 0.04 CV% 7.86

Within Assay Precision Pool 1

(ppb)

0.53 0.02 3.9

Pool 2 (ppb)

1.06 0.035 3.35

Pool 2 (ppb)

1.07 0.03 3.1

Pool 3 (ppb)

1.54 0.07 4.48

Pool 3 (ppb)

1.53 0.05 3.5

X SD CV%

Control#_____
OG-0000.00

Analysis of terpenes with HS/GC/MS

Revision
0

Effective Date
8/6/16

Prepared By

Southeast Alaska Laboratories, LLC

5450B Jenkins Dr., Juneau AK

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315-778-1469

APPROVED BY:

_____ _____
Laboratory Director Date

_____ _____
Quality Assurance Officer Date

1.0 SCOPE AND APPLICATION
2.0 SUMMARY
3.0 TERMS AND DEFINITIONS
4.0 INTERFERENCES
5.0 HEALTH AND SAFETY
6.0 APPARATUS AND MATERIALS
7.0 REAGENTS
8.0 SAMPLE COLLECTION/ PRESERVATION/ HOLDING TIMES
9.0 QUALITY CONTROL (See QC Summary Tables in Appendix A)
10.0 CALIBRATION AND STANDARDIZATION
11.0 PROCEDURE
12.0 CALCULATIONS
13.0 REFERENCES

1.0 SCOPE AND APPLICATION

This procedure will be used for for flower to determine terpene profile. It will be modified as needed for extracts and concentrates.

2.0 SUMMARY

Determination of the terpene profile for cannabis flower.

3.0 TERMS AND DEFINITIONS

3.1 No new terms in this SOP

4.0 INTERFERENCES

5.0 HEALTH AND SAFETY

5.1 Lab coat, gloves and eye protection should be worn during the preparation of samples and standards for this procedure.

6.0 APPARATUS AND MATERIALS

6.1 GCMS with capability of sample introduction by headspace
6.2 10 mL headspace vials with caps and crimper

6.3 analytical balance readable to 0.1 mg
6.4 micro pipets w/tips
6.5 15 mL graduated centrifuge tubes
6.6 1.8 mL autosampler vials

7.0 REAGENTS

7.1 standard reference materials (will enter Restek #s later):
α-Pinene
Camphene
β-Myrcene
β-Pinene
3-Carene
D-limonene
Linalool
Fenchol
Terpineol
Caryophillene
Aromadendrene
Humulene

8.0 SAMPLE COLLECTION/ PRESERVATION/ HOLDING TIMES

8.1 Samples should be collected and received as described in the collection and chain of custody SOPs.

8.2

9.0 QUALITY CONTROL (See QC Summary Tables in Appendix A)

1. The analysts must make an initial demonstration of capability and generate acceptable accuracy and precision with the method before analyzing any samples. Any changes or modifications to this SOP require the analyst to repeat the demonstration of capability.

1. Demonstration of Capability:

1.1. A quality control sample shall be obtained from an outside source.

1.2. The four quality control samples shall be analyzed according to the test method either concurrently or over a period of days.

1.3. Run the aliquots after analyzing all required calibration standards. Ensure that the reagent blank does not contain any analyte at a concentration greater than the reporting limit (RL).

1.4. Using all of the results, calculate the mean recovery (x) and the Percent Relative Standard Deviation.

1.5. An acceptable DOC has a mean percent recovery between 80% and 120%. The relative percent standard deviation <20%.

2. Limit of Detection (LOD):

9.2.1 LOD's must be determined annually. Also, LOD's must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

9.3 A MB, LCS, DUP and CCV must be run with every batch. A batch includes at most twenty samples.

9.3.1 Method Blanks (MB)-Will be performed at a frequency of one per batch of samples per matrix type per extraction or preparation method. The results of this analysis will be one of the QC measures to be used to assess batch acceptance. The source of contamination must be investigated and measures taken to correct minimize or eliminate the problem if: 1) The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch or 2) The blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

9.3.2 Lab Control Sample (LCS)/Initial Calibration Verification (ICV) - Are performed at a frequency of one per batch of 20 or less of samples per matrix type, and sample preparation. The results of analysis are used to assess batch acceptance. The LCS must have a percent recovery of 80%-120% to be acceptable.

9.3.4 Sample Duplicates (DUP) - Are performed at a minimum of 1 in 20 samples per matrix type per sample preparation. The duplicate sample is chosen at random. The selected samples

are rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. The RPD for water samples is ±10%.

9.4 Continuing Calibration Verification (CCV): After 10 samples and/or at the end of analysis run, prepare a CCV to verify calibration. The concentration must vary to verify different parts of the curve. The CCV must have a recovery of ±20% to be acceptable. If a CCV fails, any sample associated with the CCV must be re-analyzed.

9.5 If the MB or LCS fails, the analysis will be rerun.

6. If the MB and LCS pass but the DUP and/or MS fail, the data is reported with qualifiers.

9.7 All data must be recorded in the Cannabis % Moisture data logbook.

10.0 CALIBRATION AND STANDARDIZATION

Instrumental Method:

Column Rxi®-624Sil MS, 30 m, 0.25 mm ID, 1.40 µm

Injection headspace-loop split (split ratio 50:1)
HS Inj. Time: 0.5 min

HS Transfer Line Temp.: 150 °C

HS Sample Line Temp.: HS incubation: 150 °C
HS Pressurizing gas pressure: 140 °C for 10 min

HS Pressurizing time: 25KPa
HS Sample Equil. Time: 1.0 min

GC Oven Temp.: 30.0 min

GC Carrier Gas He, constant flow
GC Linear Velocity: 33 cm/sec

MS Detector 20-400m/z 2.0 min solvent delay
MS Ion source temp 225C
MS Interface temp: 250°C

FID Detector temp: 320°C

FID gases Air-Hydrogen: 400ml/min-40ml/min

Makeup flow: 20 ml/min

11.0 PROCEDURE

11.1 Prepare a 2000 ug/ml stock solution in Methanol or other appropriate solvent. 20mg of pure standard in 10 ml solvent yields a 2000ug/ml solution.

11.2 Dilute the solution to obtain 5 different concentration between 2000ug/ml and 200ug/ml Put 50 ul of prepared standards solution in 10 ml vial and cap.

11.3 *50ul of a 1000ug/ml solution will correspond to 50 ug of target in the vial.*

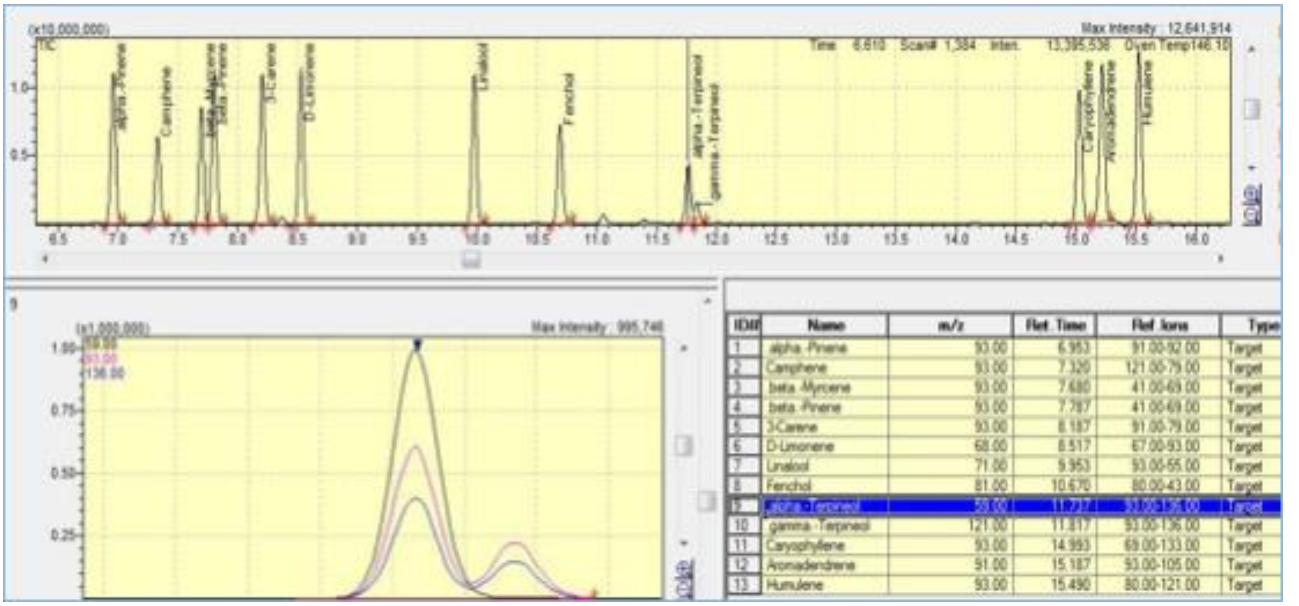
11.4 Do a calibration curve approximately 10-100 ug for each target.

11.5 Sample preparation: Weigh 10-50mg of flower/concentrate. Put in 10 ml vial and cap. Prepare with a methanol-chloroform extraction.

11.6 Result calculation:
The result in ug of terpene will be divided by the sample weight in mg to obtain the result in ug/mg Divide the result in ug/mg by 10 to have value in % w/w w/w%=(ug/mg*1000)*100

12.0 CALCULATIONs

13.0 REFERENCES



ID#	Name	m/z	Ret. Time	Ref. Ions	Type
1	alpha-Pinene	93.00	6.953	91.00-92.00	Target
2	Camphene	93.00	7.320	121.00-79.00	Target
3	beta-Myrcene	93.00	7.680	41.00-69.00	Target
4	beta-Pinene	93.00	7.787	41.00-69.00	Target
5	3-Carene	93.00	8.187	91.00-79.00	Target
6	D-Limonene	68.00	8.517	67.00-93.00	Target
7	Linalool	71.00	9.953	93.00-55.00	Target
8	Fenchol	81.00	10.670	80.00-43.00	Target
9	alpha-Terpineol	59.00	11.737	93.00-136.00	Target
10	gamma-Terpineol	121.00	11.817	93.00-136.00	Target
11	Caryophyllene	93.00	14.993	69.00-133.00	Target
12	Aromadendrene	91.00	15.187	93.00-105.00	Target
13	Humulene	93.00	15.490	80.00-121.00	Target

Appendix A

QC Summary

Quality Control Element	Frequency	Acceptance Limits
Limit of Detection (LOD)	Annually or with substantial change to method or instrumentation.	40 CFR Part 136, Appendix B calculations.
Demonstration of Capability (DOC)	Annually for each analyst	Average of replicates must be 80-120% of true value and < 20% RSD.
Initial Calibration Verification (ICV)	Following every initial calibration.	80-120% of true value.
Continuing Calibration Verification (CCV)	At the start of each analytical batch when an initial has not been performed. Note: The CCV concentration is to be varied through the analysis.	80-120% of true value.
Method Blank	1 in every 20 or fewer samples per preparation batch and at least 1 per 12 hour period.	Concentration must be below the MRL.
Laboratory Control Sample (LCS)	1 in every 20 or fewer samples per preparation batch	80-120% of true value.
Matrix Spike (MS)	1 in every 20 or fewer samples per preparation batch. Sample chosen at random.	80-120% of true value. Not required for batch acceptance.
Matrix Spike Duplicate (MSD)	1 in every 20 or fewer samples per preparation batch. Sample chosen at random.	80-120% of true value. Not required for batch acceptance. Relative Percent Difference (RPD) <10%

Appendix A (Continued)

QC Corrective Action Summary

Quality Control Element	Corrective Action
Limit of Detection (LOD)	Determine source of problem, correct, and re-analyze.
Demonstration of Capability (DOC)	Determine source of problem, correct, and re-analyze.
Initial Calibration Verification (ICV)	Re-read ICV. If ICV fails a second time, a new initial calibration is required.
Continuing Calibration Verification (CCV)	Re-read CCV. If CCV fails a second time, a new initial calibration is required.
Method Blank	Determine source of problem, correct, and re-analyze.
Laboratory Control Sample (LCS)	Determine source of problem, correct, and re-analyze.
Matrix Spike (MS)	Check makeup of MS solution. If LCS passes, indicate matrix problem with sample.
Matrix Spike Duplicate (MSD)	Re-analyze. If LCS passes, indicate matrix problem with sample.

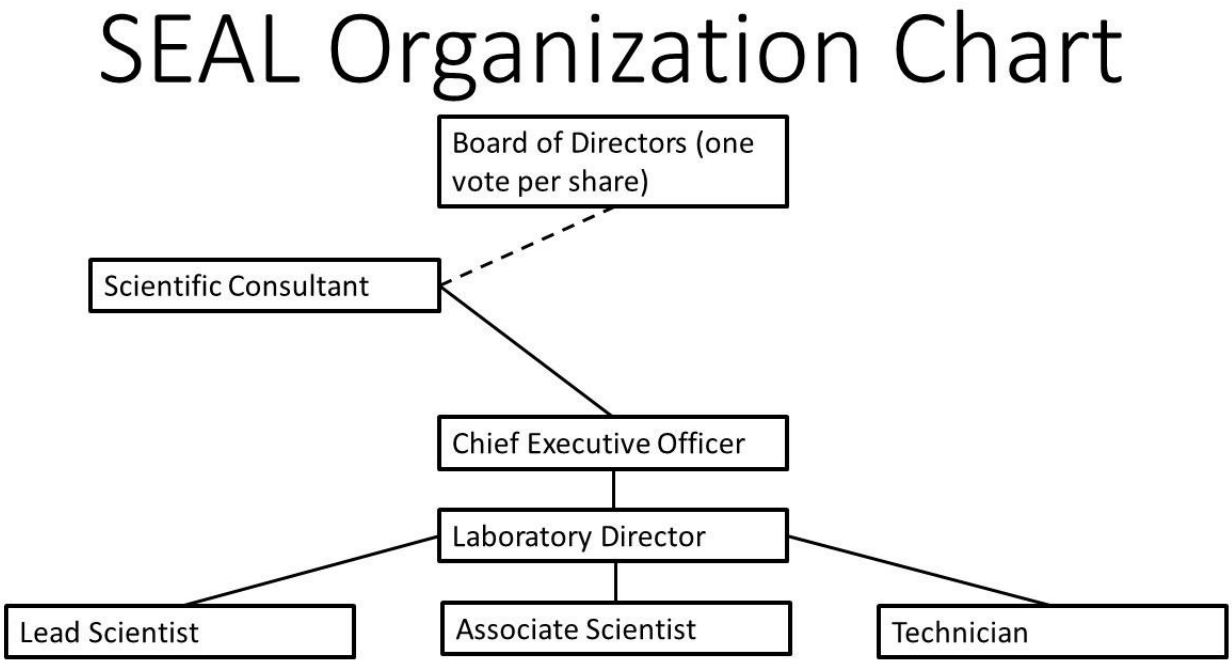
Appendix B
SEAL and Method Precision and Accuracy

Analyte	Method LOD (ug/L)	SEAL LOD	Method Accuracy %Rec	SEAL Accuracy* %Rec	Method Precision %RSD	SEAL Precision* %RSD

*SEAL, accuracy, and precision calculated from LCS and MS/MSD from control charts.
**SEAL LOD was performed
***See Section 13.0

SEAL Proficiency Testing Results

Analyte	RTC PT (5/11)	% REC	Perf. Eval.	RTC PT (11/11)	% REC	Perf. Eval	RTC PT (5/12)	% REC	Perf. Eval

Appendix C

C. SEAL Organizational Chart