# **TestAmerica Ontario**

**Analytical Method Information** 

#### 525.2 Short Hold Time Analytes

## ORGANIC COMPOUNDS BY GC/MS (EPA 525.2)

Water EPA 525.2

Sampling Info:

Preservation: 4 C, Na2SO3, HCL, Dark

Container:1 L AmberAmount Required:2000 mlHold Times:Sampled to Prepared:24.00 hoursPrepared to Analyzed:30 days

Prep Info:

Extraction Method: EPA 525.2 Initial Units: ug/l Final Units: ug/l Final Units: ug/l

Default Initial Amt: 1,000.00 mL Default Final Amt: 1.00 mL Default Dilution: 1

	MDL	Reporting	Surrogate	Dup	Matrix S	Spike	Blank Spik	e (LCS)	RL	CL
Analyte	(ug/l)	Limit (ug/l)	%R	RPD	%R	RPD	%R	RPD	Date	Date
	2.24	0.05		20	<b>5</b> 0 120	20	<b>5</b> 0 120	20	5 10 10 <b>2</b>	C 10 0 10 F
Diazinon	0.24	0.25		30	70 - 130	30	70 - 130	39	5/8/03	6/28/07
Disulfoton	0.46	100		30	70 - 130	30	70 - 130	30	5/8/03	1/1/80

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# **TestAmerica Ontario**

## **Analytical Method Information**

#### 525.2 CA Extended

## ORGANIC COMPOUNDS BY GC/MS (EPA 525.2)

Water EPA 525.2

Sampling Info:

**Preservation:** 4 C, Na2SO3, HCL, Dark

Container: 1 L Amber Amount Required: 2000 ml

Hold Times: Sampled to Prepared: 14 days Prepared to Analyzed: 30 days

Prep Info:

Extraction Method: EPA 525.2 Initial Units: ug/l Final Units: ug/l Final Units: ug/l

	MDL	Reporting	Surrogate	Dup	Matrix	Spike	Blank Spik	e (LCS)	RL CL	CL
Analyte	(ug/l)	Limit (ug/l)	%R	RPD	%R	RPD	%R	RPD	Date	Date
Acenaphthylene	0.76	5.0		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Alachlor	0.078	1.0		30	70 - 130	30	70 - 130	5	5/8/03	4/21/08
Aldrin	0.015	0.075		30	70 - 130	30	70 - 130	10	5/8/03	4/21/08
Ametryn	0.072	1.0		30	70 - 130	30	70 - 130	20	5/8/03	4/21/08
Atrazine	0.073	0.50		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Benz(a)anthracene	0.16	10		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Benzo(a)pyrene	0.019	0.10		30	70 - 130	30	70 - 130	10	5/8/03	4/21/08
Benzo(b)fluoranthene	0.21	10		30	70 - 130	30	70 - 130	10	5/8/03	4/21/08
Benzo(g,h,i)perylene	0.83	10		30	70 - 130	30	70 - 130	10	5/8/03	4/21/08
Benzo(k)fluoranthene	0.25	10		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Butachlor	0.057	0.38		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Butylate	0.080	1.0		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Butyl benzyl phthalate	0.21	10		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Di-n-butyl phthalate	0.35	5.0		30	70 - 130	30	70 - 130	5	5/8/03	4/21/08
2-Chlorobiphenyl	0.062	0.50		30	70 - 130	30	70 - 130	10	5/8/03	11/9/07
Chloroneb	0.068	0.50		30	70 - 130	30	70 - 130	15	5/8/03	11/9/07
Chloropropham	0.17	0.50		30	70 - 130	30	70 - 130	15	5/8/03	4/21/08
Chlorpyrifos	0.10	1.0		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Chrysene	0.19	5.0		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Cycloate	0.079	0.30		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Dibenz(a,h)anthracene	0.70	5.0		30	70 - 130	30	70 - 130	10	5/8/03	4/21/08
2,3-Dichlorobiphenyl	0.066	0.50		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Di(2-ethylhexyl)adipate	0.26	5.0		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Di(2-ethylhexyl)phthalate	0.47	3.0		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
Diethyl phthalate	0.26	5.0		30	70 - 130	30	70 - 130	5	5/8/03	4/21/08
Dimethyl phthalate	0.14	5.0		30	70 - 130	30	70 - 130	10	5/8/03	4/21/08
Diphenamid	0.065	100		30	70 - 130	30	70 - 130	5	5/8/03	4/21/08
Fluorene	0.066	5.0		30	70 - 130	30	70 - 130	5	5/8/03	4/21/08
2,2',3,3',4,4',6-Heptachlorobiphen	yl0.22	1.0		30	70 - 130	30	70 - 130	10	5/8/03	4/21/08
Hexachlorobenzene	0.014	0.50		30	70 - 130	30	70 - 130	10	5/8/03	6/28/07
2,2',4,4',5,6'-Hexachlorobiphenyl	0.29	1.0		30	70 - 130	30	70 - 130	10	5/8/03	4/21/08
Hexachlorocyclopentadiene	0.052	1.0		30	70 - 130	30	70 - 130	15	5/8/03	4/21/08
Indeno(1,2,3-cd)pyrene	0.81	10		30	70 - 130	30	70 - 130	10	5/8/03	4/21/08
Lindane (gamma-BHC)	0.013	0.20		30	70 - 130	30	70 - 130	5	5/8/03	4/21/08

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# **Quality Assurance Manual**

TestAmerica Ontario, CA 1014 E. Cooley Dr., Suites A-F Colton, CA 92324 909-370-4667 909-370-1046

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7	<u>Data Qualifiers</u>	Appendix 7-1	01/31/2008

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# SOPS AND POLICIES REFERRED TO IN THE QA MANUAL

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CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-003	Management of Change Procedure
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-005	Calibration Curves (General)
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-L-P-001	Record Retention
CW-F-P-002	Authorization Matrix
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-004	Controlled Purchases Policy

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#### **SECTION 3**

# INTRODUCTION (NELAC 5.1 - 5.3)

#### 3.1 INTRODUCTION AND COMPLIANCE REFERENCES

**TestAmerica Ontario, CA**'s Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. Each TestAmerica laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (1999). In addition, the policies and procedures outlined in this manual are compliant with the various accreditation and certification programs listed in Appendix 6. The relevant NELAC section is included in the heading of each QAM section.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 815-R-05-004, Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition, EPA, January 2005.
- EPA 600/R-94-173, Technical Notes on Drinking Water Methods, EPA, October 1994.
- EPA 600/4-88/039, Methods for the Determination of Organic Compounds in Drinking Water, EPA, Revised July 1991.
- EPA 600/R-95/131, Methods for the Determination of Organic Compounds in Drinking Water, Supplement III, EPA, August 1995.
- EPA 600/4-79-019, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA, March 1979.
- EPA SW-846, *Test Methods for the Evaluation of Solid Waste, 3<sup>rd</sup> Edition,* September 1986; Update I, July 1992; Update II, September 1994; and Update III, December 1996.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- APHA, Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup> Edition, 19<sup>th</sup>, 20<sup>th</sup> and 21<sup>st</sup> Edition.
- Toxic Substances Control Act (TSCA).

#### 3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by **TestAmerica Ontario**, **CA** conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

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Refer to Appendix 5 for the Glossary/Acronyms.

#### 3.3 SCOPE / FIELDS OF TESTING

**TestAmerica Ontario, CA** analyzes thousands of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested water, air, industrial waste, and soil methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4 as the Quality Control Limits Summary. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by *TestAmerica Ontario*, *CA* shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director (LD) and the Quality Assurance (QA) Manager at a kickoff meeting with the Project Manager (PM), Lab Director, QA Manager, and possibly Marketing. The QAPP and/or DQOs must be available for review and approval at this meeting prior to acceptance of samples. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

#### 3.4 MANAGEMENT OF THE MANUAL

#### 3.4.1 Review Process

The manual is reviewed annually by the QA Manager and laboratory personnel to assure that it reflects current practices and meets the requirements of *TestAmerica Ontario*, *CA*'s clients and regulators. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. The updates will be reviewed by the QA Manager, Laboratory Director, Technical Director(s), relevant operational staff and Corporate Quality Assurance (if a change is made to the Corporate template) and then formally incorporated into the document in periodic updates. The QAM is based on a Corporate QAM Template that is prepared and approved by the Chief Operating Officer (COO) and Corporate Quality Assurance. This template is reviewed annually by the COO, Corporate Quality, and each laboratory. Necessary changes are coordinated by the Executive Director of Quality and Environmental Health & Safety (EHS) and distributed to each laboratory for inclusion in the laboratory specific QA Manuals.

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Policies in the QAM that require immediate attention may be addressed through the use of Corporate QA/QC Policy Memoranda. QA/QC Policy Memoranda are published from time to time to facilitate immediate changes to QA/QC Policy. QA/QC Policy Memoranda supersede the QAM and all other SOPs (refer to Section 5.3). All policy memoranda are dated, archived and distributed by their placement into the front of the QAM between the signature page and Section 2. At a minimum, each policy memorandum is approved by the same authorized signatories as shown on the cover page of the QA Manual. In addition, Corporate QA/QC Policy Memoranda are signed by the COO and VP of Quality and EHS. The QA/QC Policy Memoranda are incorporated into the QAM during the periodic updates. Policy memorandum may also include an expiration date if appropriate. An example format can be found in Figure 3-1. A similar procedure is followed for local laboratory changes.

Laboratory-specific QAM changes are approved and documented through the Management of Change process (Refer to SOP No. CA-Q-S-003, Management of Change Procedure).

#### 3.4.2 Control

This manual is considered confidential within TestAmerica and may not be altered in any manner by other than a duly appointed representative from TestAmerica. If the document has been provided to external users or regulators, it is for the exclusive purpose of reviewing **TestAmerica Ontario**, **CA**'s quality systems and shall not be used in any other way without the written permission of an appointed representative of TestAmerica. The procedure for control of distribution is incorporated by reference to the Document Control SOP.

The order of precedence in the event of a conflict between policies is outlined in Section 5.3 of this Quality Assurance Manual.

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# Figure 3-1

# **Example - Format for a QA/QC Policy Memorandum**

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Corporate (or Laboratory) QA/QC Policy Memorandum #				
Effective Date:	Exp	iration Date	e: When Appropriate QAM Section is	Revised
Corporate: (Only nee	eded for Corporate N	/lemorandun	n – Delete if Laboratory)	
COO - West	Date		Vice-President, QA and EHS	Date
COO - East	Date			
Local:				
Laboratory Director Ap	proval Date	Quality Ass	surance Approval	Date
Technical Director (Che	em) Approval	Date	Technical Director (Micro) Approval	Date
1. <u>Purpose</u>				
2. Procedure				
3. Attachments				
4. References/Cros	s References			

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#### **SECTION 4**

# ORGANIZATION AND MANAGEMENT (NELAC 5.4.1)

#### 4.1 OVERVIEW

**TestAmerica Ontario**, **CA** is part of a national network of laboratories known as TestAmerica. This Quality Assurance Manual (QAM) is applicable to the **TestAmerica Ontario**, **CA** laboratory only.

# TestAmerica Ontario, CA 1014 E. Cooley Dr. Suite A-F, Colton, CA 92324 EPA ID CA01533

The Corporate organization chart can be found in Figure 4-1 and the laboratory's organization chart can be found in Appendix 2. The locations of other TestAmerica labs are as follows:

TestAmerica Anchorage TestAmerica Austin

TestAmerica Buffalo

TestAmerica Buffalo Grove

TestAmerica Burlington

TestAmerica Cedar Falls

TestAmerica Chicago

TestAmerica Connecticut

TestAmerica Corpus Christi

TestAmerica Dayton

TestAmerica Denver

TestAmerica Edison

TestAmerica Honolulu

TestAmerica Houston

TestAmerica Irvine

TestAmerica King of Prussia

TestAmerica Knoxville

TestAmerica Los Angeles

TestAmerica Mobile

TestAmerica Morgan Hill

TestAmerica Nashville

TestAmerica North Canton

TestAmerica Orlando

TestAmerica Pensacola

TestAmerica Phoenix

TestAmerica Pittsburgh

TestAmerica Portland

TestAmerica Richland

TestAmerica San Francisco

TestAmerica Savannah

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TestAmerica Seattle
TestAmerica Spokane
TestAmerica St. Louis
TestAmerica Tacoma
TestAmerica Tallahassee
TestAmerica Tampa
TestAmerica Valparaiso
TestAmerica Watertown
TestAmerica West Sacramento
TestAmerica Westfield

#### 4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions define each role in its relationship to the Quality Assurance Program. More extensive job descriptions are maintained by laboratory management in the Human Resources department at the TestAmerica Irvine Laboratory.

#### 4.2.1 **Quality Assurance Program**

The responsibility for quality lies with every employee of *TestAmerica Ontario*, *CA*. All employees have access to the QAM and are responsible for knowing the content of this manual and upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs.

#### 4.2.2 President/Chief Executive Officer (CEO)

The President/CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. The President/CEO establishes the overall quality standard and data integrity program for the Analytical Division, providing the necessary leadership and resources to assure that the standard and integrity program are met.

#### 4.2.3 Chief Operating Officer (COO)

The COO serves as the ranking executive for all respective analytical laboratory operational functions and reports to the President/CEO of the Analytical Division. The COO is responsible for the daily management of all analytical laboratories, long-term planning and development of technical policies and management plans. The COO ensures the attainment of corporate objectives through the selection, development, motivation, and evaluation of top management personnel. The COO approves all operating budgets and capital expenditures. The COO signoffs on the final QAM template that contains company policies for implementing the Quality Program.

#### 4.2.4 General Manager (GM)

Each GM reports directly to the COO. Each GM has full responsibility for the overall administrative and operational management of their respective laboratories. The GM's

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responsibilities include allocation of personnel and resources, long-term planning, setting goals, and achieving the financial, business, and quality objectives of TestAmerica. The GM ensures timely compliance with corporate management directives, policies, and management systems reviews. The GM is also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

#### 4.2.5 Vice President of Client and Technical Services

The Vice President (VP) of Client and Technical Services reports directly to the President/CEO and is responsible for offerings to clients including quality assurance, environmental health and safety, risk management, technical assistance, legal compliance and contract administration. The VP of Client and Technical Services provides support and direction to the Executive Director and Directors of these areas, and supports the COO in decisions regarding long term planning, resource allocation and capital expenditures.

#### 4.2.6 Executive Director of Quality and Environmental Health and Safety (QA/EHS)

The Executive Director of QA/EHS reports to the VP of Client and Technical Services. With the aid of the Senior Management Team, Laboratory Director/ Managers, Quality Directors, EHS Directors, QA Managers and EHS Coordinators, the Executive Director-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and Environmental, Health and Safety Program within TestAmerica. Additional responsibilities include:

- Review of QA/QC aspects of Corporate SOPs, national projects and expansions or changes in services.
- Coordination/preparation of the Corporate QAM Template that is used by each laboratory to prepare its own laboratory-specific QAM.
- Maintenance of Corporate Policies, Quality Memorandums and SOPs. Maintenance of data investigation records that are reported to Corporate Management.
- Working with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation of a monthly report that includes quality metrics across the Analytical Division and a summary of any quality related initiatives and issues.
- With the assistance of the Corporate Senior Management Teams and the EHS Directors, development and implementation of the TestAmerica Environmental, Health and Safety Program.

#### 4.2.7 **Quality Directors (Corporate)**

The Quality Directors report to the Executive Director-QA/EHS. Together with the Executive Director-QA/EHS, the Quality Directors have the responsibility for the establishment, general overview and maintenance of the Analytical Division's Quality Assurance Program within TestAmerica. The Quality Directors are responsible for:

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- Oversight of the QA/QC programs within each laboratory. This includes a final review of each laboratory-specific QAM and receipt of each laboratory's QA monthly report.
- Working with management to develop a plan of correction when a laboratory's quality system is determined to be inadequate.
- Review of QA/QC aspects of national projects.
- Assistance with certification activities.
- Providing assistance as needed in the selection of Quality Assurance Managers and reviewing their effectiveness.

#### 4.2.8 <u>Ethics and Compliance Officers (ECOs)</u>

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) –VP-Client and Technical Services and the Executive Director-QA/EHS. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the CEO, COO, Laboratory Director/Manager or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

#### 4.2.9 Director of Technical Services

The Director of Technical Services is responsible for establishing, implementing and communicating TestAmerica's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

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#### 4.2.10 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

#### 4.2.11 Environmental Health and Safety Directors (EHSDs) (Corporate)

The EHSDs report directly to the Executive Director-QA/EHS. The EHSDs are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

#### 4.2.12 Laboratory Director

TestAmerica Ontario's Laboratory Director is responsible for the overall quality, financial, technical, human resource and service performance of the whole laboratory and reports to the EVP-Western Division, The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive quality assurance and data integrity program. Specific responsibilities include, but are not limited to:

- Provides one or more technical directors for the appropriate fields of testing. The name(s) of the Technical Director will be included in the national database. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.

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- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as
  requiring such actions by internal and external performance or procedural audits.
   Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be
  temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Technical Directors, and other department managers as direct reports.

# 4.2.13 **Operations Manager**

The Operations Manager manages and directs the analytical production sections of the laboratory. He or she reports directly to the Laboratory Director. He or she assists the Technical Director in determining the most efficient instrument utilization. More specifically, he:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Director and QA Manager and in compliance with regulatory requirements.
- Ensures that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

#### 4.2.14 Quality Assurance Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. Corporate QA may be used as a

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resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective and preventive action systems (Section 13.0).
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 13.
- Monitoring standards of performance in quality control and quality assurance.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a percentage of all final data reports for internal consistency. Review of Chain of Custody, correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.

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#### 4.2.15 <u>Technical Directors</u>

The Technical Directors report directly to the Laboratory Director. They are accountable for all analyses and analysts within their scope of responsibility. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and second- and third-generation instrumentation. Specific responsibilities include, but are not limited to:

- Coordinating, writing, and reviewing preparation of all test methods, i. e., Standard
  Operating Procedures, with regard to quality, integrity, regulatory and optimum and efficient
  production techniques, and subsequent analyst training and interpretation of the SOPs for
  implementation and unusual project samples. They insure that the SOPs are properly
  managed and adhered to at the bench. They develop standard costing of SOPs to include
  supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield)
  utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding his requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved and requested by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This
  activity begins with reviewing and supporting all new business contracts, insuring data
  quality, analyzing internal and external non-conformances to identify root cause issues and
  implementing the resulting corrective and preventive actions, facilitating the data review
  process (training, development, and accountability at the bench), and providing technical
  and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS
  utilization. Capital forecasting and instrument life cycle planning for second generation
  methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department supervisors to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.

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Coordinates audit responses with supervisors and QA Manager.

#### 4.2.16 Hazardous Waste Coordinator

The Hazardous Waste Coordinator reports directly to the Laboratory Director. The duties consist of:

- Staying current with the hazardous waste regulations.
- · Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Chemical Hygiene/Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.

#### 4.2.17 Analytical Group Leaders

Report to the Operations Manager. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. He
  or she performs frequent SOP and QA Manual review to determine if analysts are in
  compliance and if new, modified, and optimized measures are feasible and should be added
  to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 8.1.4), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. He or she evaluates staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.
- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Director, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, nonconformance issues, and the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Ensure that all non-conformance conditions are reported to the QA Manager, Operations Manager, and/or Laboratory Director via the Corrective Action Database.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He or she is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.

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- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

#### 4.2.18 <u>Laboratory Analysts</u>

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on work lists, bench sheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Group Leader and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their Group Leader and the QA Manager. These
  improvements, if approved, will be incorporated. Ideas for the optimum performance of their
  assigned area, for example, through the proper cleaning and maintenance of the assigned
  instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

#### 4.2.19 Laboratory Technicians

- Prepare samples for analysis by weighing, extracting or digesting, filtering, or concentrating samples.
- Prepare method specific QC Samples with each preparation batch. All personnel must adhere to all QC procedures specified in the analytical method and in accordance to procedures or policies and are responsible for the full documentation of these procedures.

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#### 4.2.20 Quality Assurance Scientist

The QA Scientist reports to the facility QA Manager and performs the following functions:

- Reviews data deliverable packages to ensure completeness and accuracy.
- Generates and reviews, in conjunction with the Quality Assurance Manager, Control Charts and Method Detection Limit (MDL) studies.
- Assists the QA Manager and lab staff with internal audits, corrective action review and overall implementation of the QA program and fills in as the "deputy" for QA Manager in their absence.

## 4.2.21 Environmental Health & Safety Coordinator

The Safety Officer reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. The Safety Officer is responsible to:

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.

#### 4.2.22 <u>Sample Control Group Leader</u>

The Sample Control Group Leader reports to the Client Services Manager. The responsibilities are outlined below:

- Direct the logging of incoming samples into the LIMS.
- Ensure the verification of data entry from login.
- Schedule and oversee all sample courier operations.

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- Schedule and oversee all field sampling operations.
- Oversee the processing of bottle orders
- Acts as a liaison between Project Managers and Analysts in respect to handling rush orders and resolving inconsistencies and problems with chain-of-custody forms, and routing of subcontracted analyses.
- Oversees the disposal of samples in accordance with the Waste Disposal SOP, the Hazardous Waste Contingency Plan in the Chemical Hygiene/Safety Manual, and the U. S. Department of Agriculture requirements.

#### 4.2.23 <u>Client Services Manager</u>

The Client Services Manager reports to the Laboratory Director and serves as the interface between the laboratory's technical departments and the laboratory's clients. The staff consists of the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Technical training and growth of the Project Management team.
- Technical liaison for the Project Management team.
- Human resource management of the Project Management team.
- Responsible to ensure that clients receive the proper sampling supplies.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning Chains-of-Custody.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the department managers of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Ensure that all non-conformance conditions are reported to the QA Manager, Operations Manager, and/or Laboratory Director via the Corrective Action Database.

#### 4.2.24 Data Package Coordinator

- Oversee the creation and delivery of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.

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## 4.2.25 **Project Manager**

• The Project Manager (PM) thoroughly coordinating client projects, maintaining clients' satisfaction and reviewing laboratory reports, addresses all project status and technical questions generated by the client. The PM is also responsible for reviewing potential work and incoming work with laboratory department representatives at daily operations meetings.

#### 4.2.26 Project Manager Assistant

 The Project Manager Assistant (PMA) provides clerical support to the project management staff in order to allow them to focus on client service and report review. The PM assistant performs faxing duties, prepares and sends electronic data deliverables (EDD) to clients, generates historical data as a cross reference for the laboratory, retrieves laboratory data, and tracks project reports.

#### 4.3 **DEPUTIES**

The following table defines who assumes the responsibilities of key personnel in their absence:

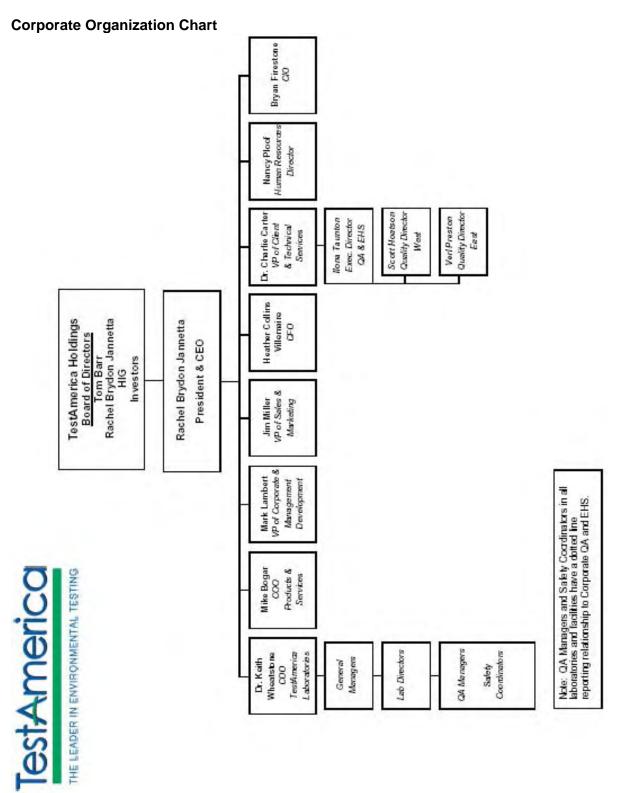
Key Personnel	Deputy	Comment
Laboratory Director	Client Services Manager	
Operations Manager	Client Services Manager	
Client Services Manager	Operations Manager	
QA Manager	Senior QA Scientist	
Chemistry Technical Director	QA Manager	
Micro Technical Director	QA Manager	
EHS Coordinator	Client Services Manager	

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Figure 4-1

Facility Distribution No.



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#### **SECTION 5**

# QUALITY SYSTEM (NELAC 5.4.2)

#### 5.1 QUALITY POLICY STATEMENT

The management of TestAmerica and *TestAmerica Ontario, CA* are committed to providing data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols described in this manual.

In all aspects of the laboratory and business operations, management is dedicated in maintaining the highest ethical standards. The Employee Ethics Statements can be viewed in Appendix 1. Training on ethical and legal responsibilities is provided annually and each employee signs off annually on the policy as a condition of employment.

It is TestAmerica's Policy to continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. The company recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.

**TestAmerica Ontario, CA** strives to provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at **TestAmerica Ontario**, **CA** plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

#### 5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The 7 elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Policy No. CA-L-P-001) and Employee Ethics Statements (Appendix 1).
- An Ethics and Compliance Officer (ECO).
- A training program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Internal Investigation of Potential Data Discrepancies and Determination for Data Recall SOP No. CA-L-S-001)
- Procedures and guidance for recalling data if necessary (Internal Investigation of Potential Data Discrepancies and Determination for Data Recall SOP No. CA-L-S-001).
- An effective external and internal monitoring system that includes procedures for internal audits (Section 16).

As an American Council of Independent Laboratories (ACIL) member, all TestAmerica laboratories adhere to the following ACIL Code of Ethics:

- Produce results, which are accurate and include QA/QC information that meets client predefined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the ethical and quality standards of our industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

#### 5.3 QUALITY SYSTEM SUPPORTING DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents prepared by the laboratory and company management:

- Quality Assurance Manual (QAM) Template
- Quality Assurance Manual Each laboratory has a lab specific quality assurance manual.
- <u>Corporate SOPs and Policies</u> Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- <u>Work Instructions</u> A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms). (controlled documents, may have specific expiration date to help with control)
- Laboratory SOPs General and Technical
- Corporate TestAmerica QA/QC Policy Memorandums (Refer to Section 3.4).
- <u>Laboratory QA/QC Policy Memorandums</u> (Refer to Section 3.4).

#### 5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- TestAmerica QA/QC Policy Memorandum Corporate
- Laboratory QA/QC Policy Memorandum
- Quality Assurance Manual
- Corporate SOPs and Policies

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- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

#### 5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term "analytical quality control". QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

#### 5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually a laboratory control sample duplicate or matrix spike (MS) duplicate samples. The calculation of precision is described in Section 25.

## 5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery. The calculation of accuracy is described in Section 25.

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#### 5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

## 5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories, and by the degree to which approval from the US EPA or other pertinent regulatory agencies is obtained for any procedure for which significant modifications have been made.

#### 5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

#### 5.4.6 <u>Selectivity</u>

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

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#### 5.4.7 <u>Sensitivity</u>

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

#### 5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory prepares a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for analyses performed at TestAmerica Ontario, CA. This summary includes an effective date, is updated each time new limits are generated and is located in the QA files for Control Chart updates. QC parameters that require Control Chart updates are updated on a rotating six month schedule, with different methods being updated each month. A control chart update form is generated for each method including a master list of every analyte that the method is capable of, any default method limits, the new values for non-default limits, and the date range and number of data points used to generate those new values. This information allows the control charts to be regenerated as needed. The effective date of the QC limits is documented in the CL Date field of each test code, and is updated automatically whenever changes are made. A summary of the QC limits for individual test codes can be viewed in Element under QA Admin\Analysis Analyte, or under Print\Analysis Info. A master QC Limit Summary can be gueried from Element when requested. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, TestAmerica Ontario, CA has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in Section 25.

#### 5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)], or when the method does not specify exact limits. *TestAmerica Ontario, CA* routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the *Technical Director* and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory in the Control Chart update files. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the

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sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

#### 5.6.1 <u>Control Charts</u>

As the QC limits are calculated by the QA Staff, Control Charts are generated showing warning and control limits, which are used to update the control limits in the test codes. The QA Staff, Lab Director, Operations Manager, or Group Leaders may also generate and evaluate Control Charts to determine if adjustments need to be made or for corrective actions to methods. All findings resulting in QC limit updates or Corrective Actions are documented and kept on file.

# 5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 17). These metrics are used to drive continuous improvement in the laboratory's Quality System.

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#### **SECTION 6**

# DOCUMENT CONTROL (NELAC 5.4.3)

#### 6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled at each laboratory Facility:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

The Corporate staff posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These are collectively termed "Official Documents" and encompass the Policies and Procedures that all facilities are required to employ. These official documents are only considered controlled when they are read on the company intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving official documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The procedures for control of documents within the laboratory are found in the Document Control SOP.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and Corrective Action Reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports. Discussion on records control is described in Section 15.

The maintenance of purchasing data is discussed in Section 9.

The maintenance of sales and marketing contracts is discussed in Section 7.

#### 6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a control system for each document include a unique name and number, the number of pages of the item, the effective date, revision number and the laboratory's name. The QA Staff is responsible for the maintenance of the system and

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maintains the items as hardcopies in the QA files or electronically in the QA directory on the network.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a Group Leader or Manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain the official document on file. The official document is provided as needed to those using it. Controlled documents shall be available at all locations where the operational activity described in the document is performed (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every year and revised as appropriate. Changes to documents occur when a procedural change warrants a revision of the document.

### 6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

Changes to the QA Manual (QAM) may be made on a section by section basis. The section in question is reviewed by the QA Staff and other appropriate laboratory staff. When the revisions are agreed upon, the changes are made by the QA Staff, including updating the effective date, QAM Filename, and section revision number. The Title Page of the QAM must be must be updated and signed by all appropriate staff. The Title Page, Table of Contents, and the revised section must be replaced in all controlled copies of the QAM. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-ups are stored in the QA files. Electronic copies are stored on the Public server in the QA folder for the applicable revision. Copies of the QAM must be requested and provided by the QA Staff, whether electronic or hardcopies. Copies of the QAM distributed outside of the laboratory, to clients, accrediting authorities, or otherwise, must be marked as uncontrolled.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure (SOP).

Forms, worksheets, work instructions and general information are organized by department in the QA files and on the network under the QA folder. These are updated and distributed as needed by the QA Staff and are not, in most cases, controlled documents.

### 6.3.1 Controlled Document Tracking

When a new or revised Controlled Document has been created and approved, whether corporately or locally, it is entered into the QA database for controlled copy tracking. The information in the database must minimally include the document title, filename (including the method reference and revision number separated by an underscore), the revision number, the method reference, and the effective date. The database can also track the department the SOP is assigned to and whether the Controlled Document is current or not, technical or not, and the last date it was reviewed but no revision was necessary.

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Controlled Document distributions are assigned within the database. Each entry is given a unique, sequential controlled copy number, starting with the original copy as number "0". A Controlled Copy Tracking Form is then printed. Controlled Documents are generally distributed to department SOP binders, and electronically through the Element LIMS. Copies may be distributed to individuals, but this is discouraged due to the lack of control generally demonstrated over these copies. Exceptions to this are Training Copies of SOPs given to specific staff members being trained on a new procedure in order to expedite their training or help revise the SOP, or Edit Copies that are created for reviewing and revising controlled documents. Training and Edit Copies should be marked as such in red ink. Training Copies should be returned to QA when the training is complete and any notes and notations will be evaluated to see if the SOP needs to be revised. Edit Copies must be returned to QA in order to complete the revision.

New controlled copies may be created and distributed after the Controlled Copy Tracking Form has been printed. They may be documented on the form by hand, but they must also be entered into the database. Alternatively, the form may be reprinted after the new copy has been added to the database.

Copies are then made for each hardcopy distribution listed on the Controlled Copy Tracking Form. The Controlled Copy number must be written in the appropriate space on the cover page of the document in red ink, and distributed by the QA Staff to the indicated location or person.

Word documents are considered draft documents. The final electronic copy of the document is a pdf. It is created after the document has been approved by exporting the final version from the Word document directly into a pdf file. The signed cover page is then scanned and placed into the pdf file, replacing the unsigned page. These electronic copies are kept in the Current SOPs folder in the SOP folder of the QA directory on the network, which is accessible only by QA Staff. The electronic copies of specific method SOPs are distributed through the Element LIMS. A copy of the pdf is saved in the SOP folder of the Element directory on the network, with just the method as the filename, and the security set and secured to read only. The lab may access these electronic copies by going to the QA Admin section of Element. On the Analysis page for the method test codes, there is a pdf button that is linked to these electronic copies. The lab may click on the button to open the pdf file and read it, but it cannot be copied or printed.

The original copy is kept on file in the QA SOP files along with the Controlled Copy Tracking Form.

Copies distributed outside of the physical laboratory must be marked as uncontrolled whether they are distributed electronically or by hardcopy. Electronic copies are generally secured as read and/or print only unless prior arrangements have been made and approved.

Controlled documents become obsolete when they are revised, discontinued, or otherwise invalidated. When this occurs, the QA Staff retrieves all copies of the obsolete document according to the Controlled Copy Tracking Form. The date returned is documented on the form for each Controlled Copy, and the copies are destroyed. The original document is archived with the Controlled Copy Tracking Form when all controlled copies have been collected. If, after a good faith effort to locate it, a copy of a controlled document cannot be located, it will be listed on the Controlled Copy Tracking Form as "missing". If a new copy of the document is needed, the new copy will be given a new Controlled Copy number.

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## 6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived as described in Section 15.

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#### **SECTION 7**

#### **REVIEW OF WORK REQUEST**

## 7.1 OVERVIEW

**TestAmerica Ontario, CA** has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is TestAmerica's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and TestAmerica's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the lab's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory 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 complete the work satisfactorily. Any discrepancy between the client's requirements and TestAmerica's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the

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contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The review process is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

## 7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the National Account Director, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- General Manager
- The Laboratory Client Services Manager
- The Laboratory Operations Manager
- Laboratory and/or Corporate Technical Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The National Account Director, Legal Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

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The Legal & Contracts Director maintains copies of all signed contracts. A copy is retained in the Project File in the Client Services Manager's office.

### 7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Regional Account Manager. A copy of the contract and formal quote will be filed with the laboratory PM and the Lab Director/Manager.

Records are maintained of 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. The PMs and all PM Assistants keep a phone log of conversations with the client. Important project information is documented in the Client Notes section of Element.

## 7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, *TestAmerica Ontario, CA* assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the direct client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure that available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during Status Meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

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Such changes are also communicated to the laboratory during Status Meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

TestAmerica strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

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#### **SECTION 8**

## SUBCONTRACTING OF TESTS (NELAC 5.4.5)

### 8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the corporate network. The phrase "work sharing" refers to internal transfers of samples between company laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When we must outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the SOP on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accredited work where required.

Project Managers (PMs), Customer Service Managers (CSM), Regional Account Executives (RAE), or the Drinking Water Product Manager for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

**Note:** In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work. *The Client-Approved Subcontractor Form in Figure 8-1 may be used to document client approval of a subcontractor.* 

#### 8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM, Client Services Manager, or the Drinking Water Product Manager (DWPM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified network laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);

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• Firms listed as pre-qualified and currently under a subcontract with the company (in JD Edwards): A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation for the requested tests prior to sending samples.

- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All intra-company laboratories are pre-qualified for <u>work sharing</u> provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. Refer to SOP No. CA-C-S-001, Work Sharing Process.

When the potential sub-contract laboratory has not been previously approved, Account Executives, PMs, the Client Services Manager, or the Drinking Water Product Manager may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director/Manager. The Laboratory Director/Manager requests that the QA Staff begin the process of approving the subcontract laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

- **8.2.1** The QA Manager must ensure that the Subcontracting Approval Form (Figure 8-2) has been completed and have supporting documentation on file prior to initiation of any work.\_A letter or e-mail is sent to the lab requesting the following information:
- **8.2.1.1** If a lab is NELAC or A2LA accredited,
- **8.2.1.1.1** Copy of necessary certifications verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accredit-able (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.
- **8.2.1.1.2** Insurance Certificate. This is required by TestAmerica's Chief Financial Officer
- 8.2.1.1.3 USDA soil permit if available\*\*
- **8.2.1.2** For Laboratories accredited by other agencies with an auditing program:
- **8.2.1.2.1** Copy of necessary certifications verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accredit-able (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.

- **8.2.1.2.2** Insurance Certificate. This is required by TestAmerica's Chief Financial Officer
- 8.2.1.2.3 USDA soil permit if available\*\*
- **8.2.1.2.4** Description of Ethics and Data Integrity Plan.
- **8.2.1.2.5** The most recent 2 sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action.
- **8.2.1.2.6** State Audit with Corrective Action Response
- **8.2.1.2.7** Example final report to confirm format is compliant and provides the necessary information. (minimally, it must be determined that Batch QC results are included in the laboratory reports and data is appropriately qualified.
- **8.2.1.2.8** A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review (it may need to be sent elsewhere for evaluation). This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously [minimum of 6 months] are grandfathered in.)
- **8.2.1.2.9** DoD work includes additional requirements as described in Section 8.1 above.
- **8.2.1.3** For laboratories performing tests that are unaccredited or accredited by an agency without an audit program:
- **8.2.1.3.1** A copy of their Quality Assurance Manual (controlled if possible). Ensure data quality limits for relevant methods are acceptable and that training procedures are adequate.
- **8.2.1.3.2** Copy of necessary certifications (if available) verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accredit-able (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.
- **8.2.1.3.3** Insurance Certificate. This is required by TestAmerica's Chief Financial Officer.
- 8.2.1.3.4 USDA soil permit if available\*\*
- **8.2.1.3.5** Evidence of a current SOP per method. A copy of the first page and signature page of the SOP is acceptable. A table of contents including effective dates may also be acceptable. The SOP can be examined if an on-site audit is performed.
- **8.2.1.3.6** Description of Ethics and Data Integrity Plan.

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- **8.2.1.3.7** The most recent 2 sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action.
- **8.2.1.3.8** Example final report to confirm format is compliant and provides the necessary information. (minimally, it must be determined that Batch QC results are included in the laboratory reports and data is appropriately qualified.
- **8.2.1.3.9** Statement of Qualification (SOQ) or summary list of Technical Staff and Qualifications position, education and years of experience.
- **8.2.1.3.10** DoD work includes additional requirements as described in Section 8.1 above.
- **8.2.1.3.11** A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review (it may need to be sent elsewhere for evaluation). This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously [minimum of 6 months] are grandfathered in.)
- **8.2.2** Once the information is received by the QA Manager, it is evaluated for acceptability and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site along with the associate documentation and notify the finance group for JD Edwards.
- \*\*USDA permit is required if soils less than three feet deep from New York, North Carolina, South Carolina, Georgia, Florida, Tennessee, Alabama, Mississippi, Louisiana, Arkansas, Texas, Oklahoma, New Mexico, Arizona, California, Hawaii, or outside the continental U. S. are to be analyzed. These samples require special shipping measures; check with the EHS Department. It may be necessary to heat-treat the samples before shipping if the subcontract laboratory does not have a USDA permit; however, some analytes/tests may be irrelevant after heat treatment.
- **8.2.3** The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. The company does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.
- **8.2.4** The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contract Department. Any problems identified will be brought to Corporate QA attention.
- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints must be posted using the Vendor Performance Report (Form No. CW-F-WI-009).

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 Information must be updated on the intranet when new information is received from the subcontracted laboratories.

Subcontractors in good standing will be retained on the intranet listing. The QA Manager will
notify all network laboratories and Corporate QA and Corporate Contracts if any laboratory
requires removal from the intranet site. This notification will be posted on the intranet site
and e-mailed to all Lab Directors/Managers, QA Managers and Sales Directors.

### 8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or RAE or CSM or DWPM) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Figure 8-3) and the form is retained in the project folder. For network laboratories, certifications can be viewed on the company website.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within the network.

The PM will communicate with the subcontracted laboratory to monitor the status of the analyses, facilitate successful execution of the work and ensure the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples, and the subcontracting lab's original report must be included with the final report delivered to the client.

**Note:** The results submitted by a network work sharing laboratory may be transferred electronically and the results reported by the network work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and

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samples. The final report must include a copy of the completed COC for all work sharing reports.

## 8.4 **CONTINGENCY PLANNING**

The Laboratory Director/Manager may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, Corporate QA must be informed, and the QA Manager will be required to verify adequacy of proficiency scores and certifications. The laboratory must also request a copy of the raw data to support the analytical results for the first project submitted to the subcontract laboratory unless the laboratory has NELAC accreditation. -The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. The QA Manager will request full documentation and qualify the subcontractor under the provisions above. The approval process should be completed within 30 calendar days of subcontracting.

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## Figure 8-1

Example - Client-Approved Subcontractor Form	
Client Information:	
Client Name & Account Number:	
Client Contact:	
Client Address:	
Project Information: (Please choose all applicable	•
	 Other
<ul> <li>Required Turn around time (method</li> </ul>	d provisional)
Subcontractor's Information:	
Subcontractor's Name:	
Subcontractor's Contact:	
Subcontractor's Email:	
Subcontractor's Address:	
Subcontractor's Phone Number:  Analytical Test/Compound/Method to be subcont	
Certification Statement:	
I hereby give <b>TestAmerica Ontario, CA</b> permission to use the above no procedures/methods. I realize that the above subcontractor will be held procedures/methods. All subcontractors shall meet the requirements as analytical holding times and turn around times for analytical reports. The liable for liquidated damages for delays in subcontracted analytical reports.	liable for the validity of the above mentioned testing spelled out in project information and will follow all e subcontract laboratory, and not TestAmerica, will be held
Client Signature	Date

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# Figure 8-2 Example - Subcontracting Laboratory Approval Form (Initial / Renewal)

## SUBCONTRACTING LABORATORY APPROVAL

Reference: Section 8 – Quality Assurance Manua	I		
Date:Laboratory:			
•			
Address:			
Contact and e-mail address:			
Phone: Direct			
	Ī	1	
Requested Item <sup>3</sup>	Date Received	Reviewed/ Accepted	Date
1. Copy of State Certification <sup>1</sup>			
2. Insurance Certificate			
3. USDA Soil Permit			
4. Description of Ethics Program <sup>3</sup>			
5. QA Manual <sup>3</sup>			
6. Most Recent (and relevant) 2 Sets of WP/WS Reports with Corrective Action Response <sup>1,3</sup>			
7. State Audit with Corrective Action Response (or NELAC or A2LA Audit) <sup>3</sup>			
8. Sample Report <sup>3</sup>			
9. SOQ or Summary list of Technical Staff and Qualifications <sup>3</sup>			
10. SOPs for Methods to Be Loadshifted <sup>2,3</sup>			
11. For DoD Work: Statement that Lab quality system complies with QSM.			
12. For DoD Work: Approved by specific DoD Component laboratory approval process.			
Required when emergency procedures are implement 2 - Some labs may not submit copies due to internal polis acceptable. This requirement may also be fulfilled by 3 – If the laboratory has NELAC accreditation,			

□ Forwarded to Contract Coordinator, by: \_\_\_\_\_\_ Date:\_\_\_\_\_

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### Figure 8-3

## **Example - Subcontracted Sample Form** Date/Time: **Subcontracted Laboratory Information:** Subcontractor's Name: **Subcontractor Point of Contact:** Subcontractor's Address: Subcontractor's Phone: Analyte/Method: Certified for State of Origin: Yes\_\_\_\_\_No\_\_\_\_ **NELAC Certified:** Yes\_\_\_\_\_ No\_\_\_\_\_ A2LA (or ISO 17025) Certified: CLP-like Required: Yes\_\_\_\_\_ No\_\_\_\_ (Full doc required) Requested Sample Due Date: (Must be put on COC) **Project Manager: Laboratory Sample # Range:** (Only of Subcontracted Samples) **Laboratory Project Number (Billing Control #):** All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files. PM Signature\_\_\_\_\_Date\_\_\_\_

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### **SECTION 9**

## PURCHASING SERVICES AND SUPPLIES (NELAC 5.4.6)

## 9.1 OVERVIEW

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Capital expenditures are made in accordance with the Controlled Purchases Procedure, CW-F-S-004. Only one quote is required where the item being purchased is a sole source product, Examples of sole source capital expenditures are laboratory test equipment, client specified purchases and building leases. A minimum of two quotes is required where the opportunity exists to source from more than one vendor. All documentation related to the purchase of capital items will be maintained in the individual CapEx files located in Corporate Purchasing. Data will be held in accordance with the record retention policy.

TestAmerica will enter into formal contracts with vendors when it is advantageous to do so. Contracts will be signed in accordance with the Authorization Matrix Policy, CW-F-P-002. Examples of items that are purchased through vendor contracts are laboratory instruments, consumables, copiers and office supplies. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

Non-capital expenditure items are purchased through the requisition and approval process in JD Edwards or through other TestAmerica authorized methods (approved web-sites, purchasing cards, etc.). Labs have the ability to select from the approved vendors in JD Edwards.

### 9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

## 9.3 REAGENTS, STANDARDS & SUPPLIES

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents must meet with the requirements of the specific method and testing procedures for which they are

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being purchased. Solvents and acids are pre-tested in accordance with Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

### 9.3.1 Purchasing

The nature of the analytical laboratory demands that all material used in any of the procedures is of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst should use the Supplies Order Form for their area (Figure 9-1) when requesting reagents, standards, or supplies. This form also serves as the master item list of items pre-approved by the Operations Manager.

This list includes the vendor, part number, item description, package size, and the cost. The analyst enters the quantity and date needed for each item, their name and department, prints the form out and submits it for ordering. If an item being ordered is not the exact item requested, it can be added to the bottom of the form, but approval must be obtained from the Operations Manager prior to placing the order. The Ordering Originator (Group Leader, Operations Manager, or EH&S Coordinator) then places the order.

### 9.3.2 Receiving

It is the responsibility of the Ordering Originator to receive the shipment. It is the responsibility of the analyst who ordered the materials to date the material when received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are kept with the EH&S Coordinator or on the laboratory book shelf, and online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

### 9.3.3 **Specifications**

There are many different grades of analytical reagents available to the analyst. All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method.

 An expiration date can not be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.

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• Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

• If the dry chemical is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained with the certificate of analysis.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 200 psig or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a conductivity of less than 1µmho/cm (or resistivity of greater than 1.0 megaohm-cm) at 25°C. The conductivity is checked and recorded daily. If the water's conductivity is less than the specified limit, the Technical Director or Operations Manager must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

## 9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Table 9-1 details specific storage instructions for reagents and chemicals. Section 22 discusses conditions for standard storage.

## 9.4 <u>PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE</u>

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Operations Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which

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piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, it is given a short name, such as HP-20, added to the equipment list described in Section 21 that is maintained by the QA Department and IT must be notified so that can be linked for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (see Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department as specified in the laboratory's procedure for software verification. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained on the laboratory bookshelf.

## 9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 21. The need for service is determined by analysts and/or Group Leaders. The service providers that perform the services are approved by the Operations Manager/Technical Director/Laboratory Director.

## 9.6 **SUPPLIERS**

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). The level of control used in the selection process is dependent on the anticipated spend and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report (CW-F-WI-009).

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

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The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

## 9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (CW-F-WI-007 – refer to Figure 9-2).

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Operations Manager are consulted with vendor and product selection that have an impact on quality.

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Figure 9-1 Example - Materials Request Sheet

Supplies Order Form.
----------------------

Requisitioner:	Dept.:
----------------	--------

#### GCMS Semi Volatiles

	GCMS Semi Volatiles					
Vendor	Part Number	Description	UOM	Cost	Quantity	Date Needed
PHENOMENEX	7HG-G010-17	DB-5MS 15M X 0.25MM X 0.5UM COLUMN	each			
PHENOMENEX	AH0-6053	SPE FEMALE LUER FITTINGS 2/PK	pack	\$25.00		
PHENOMENEX	AG0-4651	4MM ID INJECTOR LINER 5/PACK	pack	\$79.00		
PHENOMENEX	AG0-6029	SPE 12-Position Gaskets	pack	\$25.00		
PHENOMENEX	AH0-6048	SPE 12-Position Stopcocks	pack	\$65.00		
BIORAD	BIOREX 5	BIOREX 5 RESIN	500gram			
SUPELCO	57242	6ML FILTRATION TUBES 30/PACK	pack	\$26.18		
SUPELCO	57059	FLOW CONTROL VALVE LINERS	pack	\$48.40		
SUPELCO	57246-U	STAINLESS STEEL FRITS 60/PACK	pack	\$46.41		
CRESCENT CHEMICAL	CC2496	525.2 TUNING STANDARD	each	\$28.00		
CRESCENT CHEMICAL	CC2458 KIT	CLP BASE/NEUTRAL KIT	each	\$152.00		
CRESCENT CHEMICAL	CC2494	525.2 INTERNAL STANDARDS	each	\$38.00		
CRESCENT CHEMICAL	CC2495	525.2 SURROGATE STANDARD	each	\$28.00		
CRESCENT CHEMICAL	CC2048R	OC PESTICIDES MIX	each	\$45.00		
ULTRA SCIENTIFIC	ATS-161	P-TERPHENYL-D14 4/pk	pack	\$49.28		
ULTRA SCIENTIFIC	PPM-525E	ORGANOCHLORINE PESTICIDES MIX	each	\$101.02		
ULTRA SCIENTIFIC	SVM-525	SEMI-VOLATILES MIX - VWR	each	\$48.61		
ULTRA SCIENTIFIC	ATS-111	ACENAPTHENE-D10 4/pk - VWR	pack	\$34.32		
ULTRA SCIENTIFIC - VWR		ENDOTHALL SOLUTION 4pack	pack	\$28.06		
VICI Gig Harbor Group	LNR-HP4-5	4MM ID INJECTOR LINER 5/PACK	pack	\$181.00		
AGILENT	5182-0717	Blue Screw Caps 100/pk	pack	\$20.61		
AGILENT	5182-0716	blue Screw Caps 100/pk 2mL Vial 100/pk	pack	\$21.88		
		0.4MM FERRULE				
AGILENT	5062-3508		pack	\$66.05		
AGILENT	18740-20885	GOLD SEALS	each	\$35.31		-
AGILENT	5061-5869	Washers for Gold seals 12/pk	pack	\$8.86		<b>——</b>
AGILENT	19091S-133	HP-5MS 30M X 0.25MM X 0.5UM COLUMN	each	\$518.00		<b></b>
AGILENT	5181-3323	0.4MM FERRULE 10/pk	pack	\$58.72		<b></b>
ABSOLUTE	71151	CAFFEINE	each	\$22.00		
ABSOLUTE	79001	ACENAPHTHENE	each	\$22.00		ļ
ABSOLUTE	70363	DIMETHOATE	each	\$22.00		ļ
ABSOLUTE	70222	NAPHTHALENE	each	\$22.00		
ABSOLUTE	70383	PARATHION	each	\$22.00		
ACCUSTANDARD	APP-9-095	DI-N-OCTYL PHTHALATE	each	\$12.00		<u> </u>
ACCUSTANDARD	P-039S-10X	DIMETHOATE	each	\$34.00		
ACCUSTANDARD		OC PESTICIDES MIX	each			
ACCUSTANDARD	M508P-B-R2	PESTICIDES MIX	each	\$45.00		
ACCUSTANDARD	P-085S-10X	SIMAZINE	each	\$24.00		
ACCUSTANDARD	M-502-40-10X	NAPHTHALENE	each	\$14.00		
ACCUSTANDARD	APP-9-001-10X	ACENAPHTHENE	each	\$25.00		
ACCUSTANDARD	M-525-FS-2-PAK	P-TERPHENYL-D14	pack	\$80.00		
ACCUSTANDARD	P-465S-10X	ACETOCHLOR	pack			
ACCUSTANDARD	M-548.1-IS	INTERNAL STANDARD MIX	each	\$30.00		
ACCUSTANDARD	P-183S	ENDOTHALL	each	\$12.00		
ACCUSTANDARD	M-527-BDE	527 PBDE STANDARD	each	\$250.00		
PRAXAIR	AI 0.0HC-T	Air Tank	each	\$53.46		
PRAXAIR	HY 5.0UH-T	Hydrogen Tank	each	\$59.24		
PRAXAIR	NI 5.0UH-T	Nitrogen Tank	each	\$48.16		
PRAXAIR	HE 5.0UH-T	Helium Tank	each	\$103.35		
VWR	55004-080	3M Empore High Performance Extr. Disks	pack	\$191.10		
VWR	JT8055-6	JT BAKER C18 SPEEDISK	box	\$112.39		
RESTEK	20116	Ceramic Scoring Wafer 5/pk	pack	\$27.00		
SIGMA-ALDRICH	T7193-250G	Trizma Pre-Set Crystals pH7	each	\$126.50		
	11.00 2000	New Items		3.00		
				<b>†</b>		
	1					
	+			<b> </b>		
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Table 9-1
Storage of Reagents and Chemicals

Chemical	Storage Requirements
Concentrated Acids and Bases	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Bulk Dry Chemicals	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Working Solutions containing Organic Compounds	Stored as per method recommendation/ requirement. They are generally stored refrigerated at 4°C± 2°C.
Working Solutions containing only Inorganics	Stored at room temperature; refrigeration is optional.
Flammable Solvents	Stored in solvent cabinets at room temperature.
Non-Flammable Solvents	Stored separately from the flammable solvents in cabinets at room temperature.

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## Figure 9-2 Example – JD Edwards Vendor Add Request Form



## JD Edwards Vendor Add Request Form

Vendor name:	Lab location and individual making request:
Vendor address (remit to):	Vendor phone:
Vendor address (remit to):	Vendor fax:
Contact name:	Product / service provided:
Reason for Vendor Addition: Check all rea	sons that apply
☐ Cost Reduction	Estimated Annual Savings \$
☐ Replace Current Vendor	Reason?
	Vendor being Replaced?
☐ New Product / Service	Describe:
☐ ISO Approved (Required for Aerotech / F	P&K only)
Small Business:	
Does this vendor help us to meet our small bu	usiness objectives:
If yes, which category:	-
Personal and Ethical Considerations:	
Is there any personal conflict of interest with a	a TestAmerica employee and the vendor listed above?
Have ethical considerations been taken into a	account in your evaluation of this vendor?
Can this product be sourced from another	TestAmerica facility?
Please complete form and email to NCPurcha	sing@testamericainc.com or fax to (330) 966-9275.
I approve the addition of this vendor:	
Purchasing Manager - Patrick Eckman	n Corporate Controller - Leslie Bowers
Form No. CW-F-WI-007	

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#### **SECTION 10**

## SERVICE TO THE CLIENT (NELAC 5.4.7)

## 10.1 OVERVIEW

**TestAmerica Ontario, CA** cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements discussed in Section 5. The laboratory has procedures to ensure confidentiality to clients (Section 16 and 26).

**Note:** NELAC 2003 states that a laboratory "shall afford clients or their representatives' cooperation to clarify the client's request". This topic is discussed in Section 7.

## 10.2 **SPECIAL SERVICES**

The laboratory's standard procedures for reporting data are described in Section 26. When requested the following special services are provided:

- The laboratory will provide the client or the client's representative reasonable access to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- The laboratory will work with client-specified third party data validators as specified in the client's contract.
- The laboratory will provide the client with all requested information pertaining to the analysis of their samples. An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

## 10.3 CLIENT COMMUNICATION

Project Managers and PM Assistants are an important communication link to the clients. The lab shall inform its clients of any delays in project completion as well as any non-conformances in either sample receipt (refer to Section 24) or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Directors or the QA Manager are available to discuss any technical questions or concerns that the client may have.

## 10.4 REPORTING

The laboratory will work with the client to produce any special communication reports required by the contract.

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## 10.5 <u>CLIENT SURVEYS</u>

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

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#### **SECTION 11**

## COMPLAINTS (NELAC 5.4.8)

## 11.1 OVERVIEW

**TestAmerica Ontario, CA** believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that helps to continually improve processes and improving client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, communications, responsiveness, data, reports, invoicing and other functions expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for dealing with both external and internal complaints.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 13 (Corrective Actions) and is documented following the laboratory's Corrective Actions SOP. It is the laboratory's goal to provide a satisfactory resolution to complaints in a timely and professional manner.

### 11.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process and the documentation of the complaint.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

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The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

## 11.3 <u>INTERNAL COMPLAINTS</u>

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 13. In addition, Corporate Management, Sales and Marketing and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 13.

### 11.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 17)

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### **SECTION 12**

## CONTROL OF NON-CONFORMING WORK (NELAC 5.4.9)

## 12.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative, and it would be considered a Non-Conformance Event. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method, and it would be considered an actual Corrective Action. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 13).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. Any time a staff member encounters a non-conformance event, it is their responsibility to initiate the Corrective Action process, even if they were not associated with the event or it is in another area of the laboratory operations. They may discuss the issue with their Group Leader, the Operations Manager, and/or the QA Manager, but they should never rely on someone else or expect that someone else will initiate the Corrective Action process. This is necessary to insure that all non-conformance events are initially documented and are entered into the corrective action system as described in Section 13.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 20. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Operations Manager and the QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with NELAC (or the analytical method) requirements and the reason. Data being reported to a non-NELAC state would need to note the change made to how the method is normally run.

## 12.2 RESPONSIBILITIES AND AUTHORITIES

SOP No. CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall, outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of the company's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

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Under certain circumstances the Laboratory Director, Operations Manager, or the QA Manager may exceptionally authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc.. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures described in Section 13. This information must be documented in logbooks and/or data review checklists, at the very least by referencing the CAR number and possibly including a copy of the CAR. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility senior laboratory management within 24-hours, including the initiation of a CAR. The Senior Management staff is comprised\_of the Laboratory Director, QA Manager, Operations Manager, Client Services Manager, and the EH&S Coordinator. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures <u>must</u> be conveyed to an Ethics and Compliance Officer (ECO) and Quality Director within 24 hours.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director/Manager, QA Manager, ECOs, COO's – East and West, General Managers and the Quality Directors – East and West have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

## 12.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

SOP No. CA-L-S-001 distinguishes between situations when it would be appropriate for the laboratory QA Manager and Laboratory Director/Manager (or his/her designee) to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting (Section 13) in lieu of the data recall determination form contained in SOP No. CA-L-S-001.

### 12.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system (Section 13).

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On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

## 12.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 12.2, Paragraph 5 above.

Prior to suspension/restriction, confidentiality will be respected, and the problem and the required corrective and preventive action will be stated in writing and presented to the Laboratory Director/Manager.

The Laboratory Director/Manager shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 13 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director/Manager to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (i.e., Project Management, Log-in, etc...). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Operations Manager, QA Manager, Group Leaders) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, the Director of Client Services and Sales and Marketing should be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report as described in Section 13.

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#### **SECTION 13**

## CORRECTIVE ACTION (NELAC 5.4.10)

## 13.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Reports (NCR) and Corrective Action Reports (CAR) in the Element LIMS Corrective Action database (refer to Figure 13-1).

## 13.2 <u>DEFINITIONS</u>

- Corrections: Actions necessary to correct or repair procedure specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions are contained in the method specific SOPs, the Element LIMS Test Codes, and the Custom Analyses within individual Projects when there are project specific criteria. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.
- **Corrective Action**: The action taken is not only a correction made to the immediate event, but a change in process, procedure or behavior that is required to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

### 13.3 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc..

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigation.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify Systematic Problems before they become serious.
- Identify and track Client complaints and provide resolution (see more on client complaints in Section 11).
- **13.3.1 Non-Conformance Report (NCR)** is used to document the following types of corrective actions:
- One time deviations from an established procedure or SOP

- QC outside of limits (non matrix related)
- Isolated Reporting / Calculation Errors
- Client Complaints

## **13.3.2** Corrective Action Report (CAR) - used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of NCRs
- Issues found while reviewing NCRs that warrant further investigation
- Failed or Unacceptable PT results
- Corrective actions that cross multiple departments in the laboratory
- Systematic Reporting / Calculation Errors
- Health and Safety Violations

## 13.4 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a corrective action. In fact, any employee that discovers an issue that qualifies as a NCR/CAR, is obligated to initiate it in the CAR database. It should never be assumed that someone else should or will initiate a NCR/CAR. A NCR/CAR is initiated by choosing Create New in the Corrective Action menu. When filling in the database, the CAR Initiator must include all of the information asked for in the headers of the NCR/CAR including any and all employees or departments involved and the accurate occurrence date. They must also choose the closest Issue from the drop down menu, and then include an accurate description of the issue including as much information as possible such that a person outside of normal laboratory operations can understand the issue. The NCR/CAR initiator must also document all affected samples/batches on the Batch/Workorder Information tab to the extent that the affected samples are known. When all relevant information has been documented, the CAR Initiator clicks the Commit button, and the NCR/CAR is entered into the database. At this point, the PM(s) and Group Leader(s) involved are notified of the NCR/CAR by email, and the NCR/CAR is set at Open status.

There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis (including determining the true issue that occurred), Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up. These components are documented in the Cause/Employee Oversight and Corrective Action sections of the NCR/CAR.

## 13.4.1 Cause Analysis

• Upon discovery of a non-conformance event, the event must be defined and documented by the employee that initially discovers it. A NCR/CAR must be initiated, the employee(s) involved and their Group Leader(s) are assigned to investigate the issue and the event is investigated for cause. Table 13-1 provides some general guidelines on determining responsibility for assessment. During the cause analysis, it may be determined that there is a deeper issue at hand and/or that different employee(s) or department(s) were involved. The NCR/CAR is updated to reflect the more accurate information. The cause determined must accurately match the facts involved.

 The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.

• If the cause is not readily obvious, the Group Leader, Lab Director, Operations Manager, or QA staff member is consulted.

## 13.4.2 <u>Selection and Implementation of Corrective Actions</u>

- Where corrective action is needed, the laboratory shall identify potential corrective actions.
   The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The Corrective Action section of the NCR/CAR is used for this documentation.

### 13.4.3 <u>Monitoring of the Corrective Actions</u>

- The Operations Manager/Group Leader and QA Manager are responsible to ensure that the corrective action taken was effective. The Operations Manager/Group Leader insures that all sections of the NCR/CAR are adequately completed and that all affected samples are documented on the Batch/Workorder Information page. NCRs are differentiated from CARs by checking the NCR box at the top of the form. If the issue resulted from a client complaint, this is also documented by checking the box at the top of the form. When the Operations Manager/Group Leader is satisfied that the NCR/CAR has been filled out completely, they acknowledge the NCR/CAR on the Supervisors page and set the status to Review. The NCR/CAR must always adequately address the questions:
  - What is the final disposition of the data (Was it reported or cancelled)?
  - Why is the data still useable for the purposes to which it is intended?
  - How can this issue be prevented in the future?
- The QA Staff then reviews the NCR/CAR for completeness and accuracy.
- Ineffective actions will be documented and re-evaluated until acceptable resolution is achieved.
   The Operations Manager/Group Leaders are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- A determination is also made whether the client(s) need to be notified of the issue through a Client CAR. If necessary a Client CAR is designated by the check box on the QA page and possibly in the Corrective Action section of the NCR/CAR. The Client CAR information is entered in the Default Report Issue Description, Default Report Issue Cause Description, and Default Report Resolution Description sections on the QA page. These sections are included as a formal letter to the client as a paginated part of the final report, and as such should be written in the third person, past tense, with a passive voice. The Client CAR is, by its nature, intended for clients that may or may not be familiar with laboratory procedures and terminology. The level of detail must be sufficient to this purpose. At a minimum, all acronyms must be defined before usage and common laboratory procedures must be explained when relevant.

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 When the QA Staff member is satisfied that the NCR/CAR has been filled out satisfactorily, and the Client CAR is completed (if necessary), The NCR/CAR is set to Closed or PM Review. At either of these status levels, the sample data may be set to Reviewed and is ready to be sent to the client.

- The PM Review status is used when the PMs may need to review the NCR/CAR, modify the Client CAR for each work order, or when client corrective actions, such as report revisions, need to be documented as follow up. If a report revision is required as part of the corrective action process, the check box on the PM page must be checked.
- A monthly summary of all corrective actions is printed out for review to aid in ensuring that
  the corrective actions have taken effect. Summary reports are generated for CARs, NCRs,
  Client Complaints, Open NCR/CARs, Revised Reports, and a Crosstab Summary.
- The QA Manager reviews monthly NCRs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 17). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the outof-control situation and problems encountered in solving the situation.

## 13.4.4 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as
  possible when the identification of a nonconformance casts doubt on the laboratory's
  compliance with its own policies and procedures, or on its compliance with state or federal
  requirements. (Section 16 includes additional information regarding internal audit
  procedures.)
- These audits often follow the implementation of the corrective actions to verify effectiveness.
   An additional audit would only be necessary when a critical issue or risk to business is discovered.

### 13.5 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 12 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCR or CAR.

Table 13-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs.

Table 13-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, QAM Sections 20 and 21, and SOP CA-L-S-001 (Internal Investigation of Potential Data Discrepancies and Determination for Data Recall). All corrective actions are reviewed at a minimum monthly by the QA Manager and highlights are included in the QA monthly report.

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To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified through the CAR database and it's email notifications, and appropriate corrective action (e.g., reanalysis) is taken and documented.

### 13.6 BASIC CORRECTIONS

When mistakes occur in records, each mistake shall be crossed-out with a single line, and not erased, deleted, made illegible, or otherwise obliterated (e.g. no white-out), and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created. The original and the corrected hardcopies should also be retained and marked as such.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

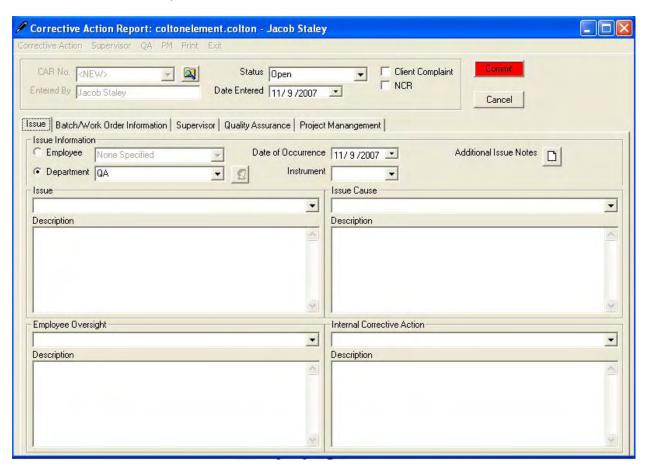
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Figure 13-1 Example - Corrective Action Report

#### CORRECTIVE ACTION REPORT DATABASE

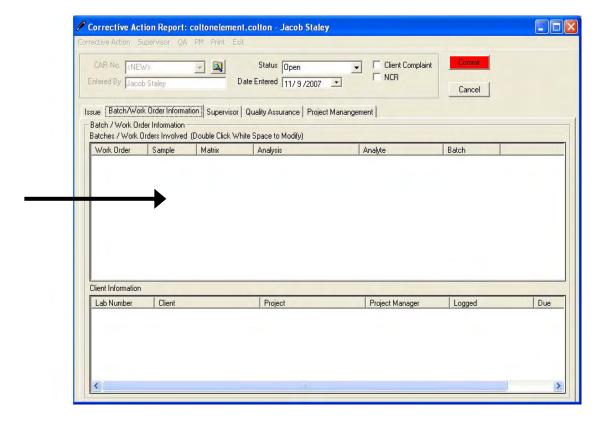
Example Screens:

## CAR Database Main Page

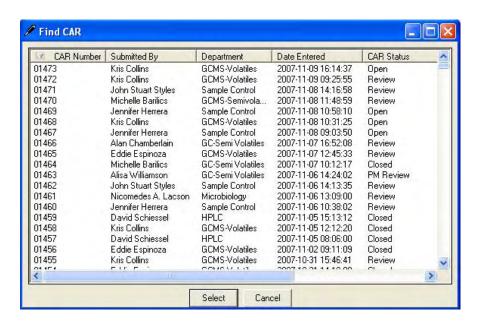


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#### CAR Database Batch/Work Order Information page



#### CAR Database CAR selection window



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Table 13-1
General Corrective Action Procedures

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank (Analyst)	- Instrument response < MDL.	<ul> <li>Prepare another blank.</li> <li>If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc</li> </ul>
Initial Calibration Standards  (Analyst, Supervisor)	- Correlation coefficient > 0.990 or standard concentration value % Recovery within acceptance range See details in Method SOP.	- Reanalyze standards If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) (Analyst, Supervisor)	- % Recovery within control limits.	- Remake and reanalyze standard If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards (Analyst, Data Reviewer)	% Recovery within control limits.	<ul> <li>Reanalyze standard.</li> <li>If still unacceptable, then recalibrate and rerun affected samples.</li> </ul>
Method Blank (MB)  (Analyst, Data Reviewer)	< Reporting Limit <sup>1</sup>	<ul> <li>Reanalyze blank.</li> <li>If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.</li> </ul>
Laboratory Control Sample (LCS)  (Analyst, Data Reviewer)	- % Recovery within limits specified in the Element test codes	- Batch must be re-prepared and re- analyzed.  Note: If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Matrix Spike / Matrix Spike Duplicate (MS/MSD)  (Analyst, Data Reviewer)	- % Recovery within limits documented in the Element test codes	<ul> <li>If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS.</li> <li>If the LCS is within acceptable limits the batch is acceptable.</li> <li>The results of the duplicates, matrix spikes and the LCS are reported with the data set.</li> </ul>
Surrogates (Analyst, Data Reviewer)	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS.

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Microbiology QC - Positive Control  (Analyst, Group Leader)	- Positive Result	- The batch is generally invalidated and the client notified to resample. In some cases, samples with positive results may be reported with qualification. The final determination of sample invalidation for compliance samples will be made by State officials at the
Microbiology QC - Negative Control  (Analyst, Group Leader)	- Negative Result	laboratory's recommendation.  - The batch is generally invalidated and the client notified to resample. In some cases, samples with negative results may be reported with qualification. The final determination of sample invalidation for compliance samples will be made by State officials at the laboratory's recommendation.
Microbiology QC - Air Control (HPC only)  (Analyst, Group Leader)	- < 15 MPN/mL	- All positive results from the preparation batch are invalidated.
Proficiency Testing (PT) Samples  (QA Manager, Department Manager/Supervisor)	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits  (QA Manager, Department Manager/Supervisor, Laboratory Director)	- Defined in Quality System documentation such as SOPs, QAM, etc	- Non-conformances must be investigated through Audit Database and necessary corrections must be made.
Reporting / Calculation Errors  (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager/ Supervisor, QA Manager, Corporate QA, Corporate Management)	- SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CA-L-S-001.

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QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Client Complaints  (Project Managers, Lab Director/Manager, Sales and Marketing)	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 17 for an example) (QA Manager, Lab Director/Manager, Department Supervisors/Managers)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director/Manager, Department Supervisor/Manager)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

#### Note:

1. Except as noted below for certain compounds, the method blank should at least be below the reporting limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit

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#### SECTION 14.0

# PREVENTIVE ACTION (NELAC 5.4.11)

#### 14.1 OVERVIEW

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes **TestAmerica Ontario, CA**'s commitment to its Quality Assurance (QA) program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc..

The monthly Quality Assurance Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's Corrective Action process (Section 13) is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

#### **14.1.1** The following elements are part of a preventive action system:

- <u>Identification</u> of an opportunity for preventive action
- Process for the preventive action
- Define the measurements of the effectiveness of the process once undertaken
- Execution of the preventive action
- Evaluation of the plan using the defined measurements
- Verification of the effectiveness of the preventive action
- <u>Close-Out</u> by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process, and the management review.

**Note:** There may be varying levels of formality and documentation during the preventive action process due to the simplicity/complexity of the action taken.

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**14.1.2** Any Preventive Actions undertaken or attempted shall be taken into account during the Annual Management Review (Section 17). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program will provide management a measure for evaluation.

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#### SECTION 15.0

# CONTROL OF RECORDS (NELAC 5.4.12)

**TestAmerica Ontario, CA** maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

## 15.1 <u>OVERVI</u>EW

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 15-1. Quality records are maintained by the Quality Assurance (QA) Manager in a database, which is backed up as part of the regular network backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the Archiving Coordinator.

Table 15-1 Record Index<sup>1</sup>

Technical				Administrative			
Records	Documents	QA Records	Project Records	Records			
Retention Period							
Retention: 10 Years from analytical report issue, 5 years for the 8000 series methods*	10 Years from document retirement date*	10 Years from archival*  Data Investigation: 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)	12 Years from analytical report issue*	Personnel: 7 Years (HR Records must be maintained as per Policy CW-L-P-001) Finance: See Accounting and Control Procedures Manual			
		Specific Doc	cuments				
Raw Data Logbooks <sup>2</sup>	Quality Assurance Manual (QAM)	Internal and External Audits/ Responses	Sample receipt and COC Documentation	Finance and Accounting			
Standards	Work Instructions	Certifications	Contracts and Amendments	EH&S Manual, Permits, Disposal Records			
Certificates  Analytical	SOPs Manuals	Corrective/Preventive Action	Correspondence	Employee Handbook			
Records  Lab Reports							
		Management Reviews	QAPP	Personnel files,			

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Technical	Official			Administrative
Records	Documents	QA Records	Project Records	Records
		Retention	Period	
Retention: 10 Years from analytical report issue, 5 years for the 8000 series methods*	from document retirement tissue, rs for 000 s from document retirement date*  archival*  Data Investigation years or the life of affected raw data storage whichever greater (beyond 5 years if ongoing		12 Years from analytical report issue*	Personnel: 7 Years (HR Records must be maintained as per Policy CW-L-P-001) Finance: See Accounting and Control Procedures Manual
		Method & Software Validation, Verification data	SAP	Employee Signature & Initials, Administrative Training Records (e.g., Ethics)
		Data Investigation	Telephone Logbooks	Administrative Policies
	Policies		Lab Reports	Technical Training Records

<sup>&</sup>lt;sup>1</sup> Record Types encompass hardcopy and electronic records.

All records are legible and stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. Records are maintained in Long Term Storage on-site at the laboratory for at least 1 year after their generation and archived offsite for the remainder of the required storage time, to the COR-O-VAN record storage facility. The COR-O-VAN storage facility is located at:

COR-O-VAN Orange County 2100 E. Valencia Drive, Suite D Fullerton, CA 92831-4811 714-446-9500

Records are maintained for a minimum of five years unless other wise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 15-2 have lengthier retention requirements and are subject to the requirements in Section 15.1.3. Policy CW-L-P-001 (Record Retention) provides additional information on record retention requirements.

<sup>&</sup>lt;sup>2</sup> Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).

<sup>\*</sup> Exceptions listed in Table 15-2.

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#### 15.1.1 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 15-3 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data. Most data generated by this facility is directly related to or associated with Drinking Water compliance, and as such, the default storage length is 10 years. Exceptions to this are the data for the 8000 series methods still performed here, which will be retained for 5 years, and Administrative records which must only be maintained for 7 years as described in the table above. Also, since some of our projects are in compliance with the Lead & Copper Rule, all project files are retained for 12 years.

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Table 15-2 Special Record Retention Requirements

Program	<sup>1</sup> Retention Requirement
Drinking Water – All States	10 years (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

<sup>&</sup>lt;sup>1</sup>Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

- **15.1.2** All records are held secure and in confidence. Records maintained at the laboratory are located in the Long Term Storage Area and are not considered archived. Records are archived off-site, and are stored in a secure location where a record is maintained of any entry into the storage facility.
- **15.1.3** The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, see section 20.12.1 'Computer and Electronic Data Related Requirements' for more information.
- **15.1.4** The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (Records stored off site should be accessible within 2 days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.
- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the chain of custody is stored with the invoice and the work order sheet generated by the LIMS. The chain of custody would indicate the name of

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the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set according to the laboratory's Record Archiving SOP. Instrument data is stored sequentially by instrument and date of analysis. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks, bench sheets, or entered into the LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20.
   Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "Analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Also refer to Section 20.13.1 'Computer and Electronic Data Related Requirements'.

#### 15.2 TECHNICAL AND ANALYTICAL RECORDS

- **15.2.1** The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement (refer to Section 15.1). The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and checking of results.
- **15.2.2** Observations, data and calculations are recorded at the time they are made and are identifiable to the specific task.
- **15.2.3** Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include (previous discussions relate where most of this information is maintained specifics may be added below):
- laboratory sample ID code;

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- Date of analysis and time of analysis is required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a bench sheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs, with the instrument calibrations, or in the method SOPs.
- analysis type;
- · all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- · test results:
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

#### 15.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a
  description of the specific computational steps used to translate parametric observations into
  a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

## 15.3.1 <u>Sample Handling Records</u>

Sample handling and tracking is discussed in Section 24. Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms;
   and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

#### 15.4 <u>ADMINISTRATIVE RECORDS</u>

The laboratory also maintains the administrative records in either electronic or hard copy form. See Table 15-1.

#### 15.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

**15.5.1** All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available to the accrediting body upon request.

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**15.5.2** All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

- **15.5.3** Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.
- **15.5.4 TestAmerica Ontario, CA** has a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially according to category (Analysis, Maintenance, etc.). No analysis has more than one active notebook at a time, so all data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially in the original data file, or in a separate file when the original data is recorded in a logbook. Standards are maintained in the LIMS no logbooks are used to record that data.
- **15.5.5** Records are considered archived when moved off-site. Access to archived hard-copy information is documented with an access log and in/out records is used in archived boxes to note data that is removed and returned. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to the data is limited to laboratory and company employees.
- **15.5.6** In the event that the laboratory transfers ownership or goes out of business, **TestAmerica Ontario**, **CA** shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

#### 15.5.7 Records Disposal

- 15.5.7.1 Records are removed from the archive and disposed after at least 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration.
- **15.5.7.2** Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.
- **15.5.7.3** If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required. [Refer to Policy No. CW-L-P-001 (Records Retention).]

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#### **SECTION 16**

## AUDITS (NELAC 5.4.13)

#### 16.1 OVERVIEW

Audits measure laboratory performance and insure compliance with accreditation/certification and project requirements. Audits specifically provide management with an on-going assessment of the quality of results produced by the laboratory, including how well the policies and procedures of the QA system and the Ethics and Data Integrity Program are being executed. They are also instrumental in identifying areas where improvement in the QA system will increase the reliability of data. There are two principle types of audits: Internal and External. Internal audits are performed by laboratory or corporate personnel. External audits are conducted by regulators, clients or third-party auditing firms. In either case, the assessment to program requirements is the focus.

Table 16-1 Audit Types and Frequency

Internal Audits	Description	Performed by	Frequency	
	Analyst & Method Compliance	QA Department or Designee	<ul><li>- 100% of all methods over a two year period.</li><li>- 100% of all analysts annually.</li></ul>	
	Instrument	QA Department or Designee	100% of all organic instruments and any inorganic chromatography instruments. Every 2 years.	
	Work Order/ Final Report	QA Department or Designee	- 1 complete report each month.	
	Support Systems	QA Department or Designee	- Annual for entire labs support departments & equipment (e.g., thermometers, balances), can be divided into sub-sections over the course of the year.	
	Performance Audits (Double-Blind PTs)	Corporate QA, Laboratory QA Department or Designee	- As needed.	
	Special	QA Department or Designee	- As Needed	
<b>External Audits</b>	Description	Performed by	Frequency	
	Program / Method Compliance	Regulatory Agencies, Clients, accreditation organizations	- As required by program and/or clients needs	
	Performance Audits	Provided by a third party.	- As required by a client or regulatory agency. Generally provided semi-annually through the analysis of PT samples.	

### 16.2 INTERNAL AUDITS

Annually, the laboratory prepares a schedule of internal audits to be performed throughout the year. As previously stated, these audits verify and monitor that operations continue to comply with the requirements of the laboratory's QA Manual and the Corporate Ethics Program. A

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schedule of the internal audits is maintained by the QA Manager in the *Internal Audit Workbook*. An example can be found in Attachment 1.

It is the responsibility of the QA Manager to plan and organize audits in consideration of the laboratory work load and the department personnel schedules so that all pertinent personnel and operations are thoroughly reviewed. When designees (other than QA department personnel & approved by the QA Manager), perform audits, the QA Manager shall insure that these persons do not audit their own activities except when it can be demonstrated that an effective audit will be carried out. In general, the auditor:

- Is neither the person responsible for the process being audited nor the immediate supervisor of the person responsible for the project/process.
- Is free of any conflicts of interest.
- Is free from bias and influences that could affect objectivity.

Laboratory personnel (e.g., supervisors and analysts) may assist with both method and support system audits as long as the items listed in the above paragraph are observed. These audits are conducted according to defined criteria listed in the checklists of the *Internal Audit Workbook*. These personnel must be approved by the QA Manager; and must complete the audit checklists in their entirety. This process introduces analyst experience and insight into the laboratory's auditing program.

The auditor must review the previous audit report and identify all items for verification of corrective actions. A primary focus will be dedicated to the ability of the laboratory to correct root-cause deficiencies and that the corrective action has been implemented and sustained as documented.

#### 16.2.1 **Systems**

An annual systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems, client and State requirements. This audit is performed in portions throughout the year through method, analyst, instrument, work order/final report and support system audits. Audits are documented and reported to management within 1 week of their performance. Systems audits cover all departments of the facility, both operational and support. The multiple audits are compiled into one systems audit package at the end of the year (*Internal Audit Workbook*).

#### 16.2.1.1 Method, Analyst, Instrument and Work Order/Final Report Audits

Procedures for the method compliance, analyst, instrument and work order/final report audits are incorporated by reference to SOP No. CA-Q-S-004, Method Compliance and Data Authenticity Audits. These audits are not mutually exclusive. For example, the performance of a method audit will also cover multiple analysts and instruments. The laboratory's goal is to review all analysts and instruments as described in SOP No. CA-Q-S-004. The laboratory will also audit all methods within a two year time period and audit a minimum of one Work Order/Final Report from receiving through reporting on a monthly basis.

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#### 16.2.1.2 Support Systems

Support system audits are performed to ensure that all departments & ancillary equipment are operating according to prescribed criteria. Support system audits include the review of both non-analytical and operational departments. Support equipment audits (e.g., metrology items) include the review of balance calibrations, weight calibrations; water quality testing, etc.. Non-analytical may include sample receiving and bottle preparation. These types of support audits ensure that the operations are being performed to support ethical data as well as ensuring the accuracy & precision of the utilized equipment.

These audits can be performed in portions throughout the year or in one scheduled session. However, the audit schedule must document that these aspects are reviewed annually. Many of the metrology systems are considered to be surveillance activities that can be monitored by QA personnel or delegated to specified department personnel. These surveillance activities are performed on a semi-annual basis unless issues warrant a greater frequency or previous audits continually showing no deficiencies allow the frequency to be reduced to once a year.

An example audit checklist can be found in Attachment 2. Instructions for reporting findings are included in the *Internal Audit Workbook*. In general, findings are reported to management within 1 week of the audit and a response is due from management within 30 days.

#### 16.2.2 <u>Performance Audits</u>

Corporate QA may arrange for double blind PT studies to be performed in the laboratories. Results are given to Management and Corrective actions of any findings are coordinated at each facility by the QA Managers and Laboratory Directors/Managers. These studies are performed on an as needed basis. They may be performed when concerns are raised regarding the performance of a particular method in specific laboratories, periodically to evaluate methods that may not normally be covered in the external PT program or may be used in the process of developing best practices. The local QA Manager may also arrange for PT studies on an as needed basis. (Refer to Section 16.3.2 for additional information on Performance Audits.)

#### 16.2.3 **Special Audits**

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

### 16.3 **EXTERNAL AUDITS**

TestAmerica facilities are routinely audited by clients and external regulatory authorities. External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. The Operations Manager and Group Leaders are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in

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the time allotted by the client or agency performing the audit. This time frame is generally 30 days.

Be aware that NELAC requires that the audit response report be acceptable to the primary accrediting authority after the second submittal. The lab shall have accreditation revoked for all or any portion of its scope of a accreditation for any or all fields of testing, a method, or analyte within a field of testing if it is not corrected.

**TestAmerica Ontario, CA** cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

## 16.3.1 <u>Confidential Business Information (CBI) Considerations</u>

During on-site audits, on-site auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

#### 16.3.2 Performance Audits

The laboratory is involved in performance audits conducted semi-annually through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Water Supply (WS) for Drinking Water compliance and Water Pollution (WP) for Wastewater compliance. The SW-846 methods that the laboratory still performs are done so for specific short lists of analytes. There are no PTs available for these analytes.

- It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Further, where PT samples present special or unique problems in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.
- PTs generally do not have holding times associated with them. In the absence of any
  holding time requirement, it is recommended that the holding time begin when the PT
  sample is prepared according to the manufacturers instructions. Holding times should apply
  to full volume PT samples only if the provider gives a meaningful "sampling date". If this is

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not provided, it is recommended that the date/time of opening of the full volume sample be considered the beginning of holding time.

- Login will obtain the COC information from the documentation provided with the PTs with review by QA or other designated staff.
- Vials will be prepared as required in the instruction set provided with the samples. After preparation to full volume the sample may be spiked, digested, concentrated, etc., as would be done for any normal sample requiring similar analysis.
- PT samples will not undergo multiple preps, multiple runs, multiple methods (unless being used to evaluate multiple methods), multiple dilutions, UNLESS this is what would be done to a normal client sample (e.g. if a client requests, as PT clients do, that we split VOA coeluters, then dual analysis IS normal practice).
- The type, composition, concentration and frequency of quality control samples analyzed with the PT samples shall be the same as with routine environmental samples.
- Instructions may be included in the laboratory's SOPs for how low level samples are analyzed, including concentration of the sample or adjustment of the normality of titrant.
   When a PT sample falls below the range of the routine analytical method, the low-level procedure may be used
- No special reviews shall be performed by operation and QA, UNLESS this is what would be
  done to a normal client sample. To the degree that special report forms or login procedures
  are required by the PT supplier, it is reasonable that the laboratory WOULD apply special
  review procedures, as would be done for any client requesting unusual reporting or login
  processes.
- Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

#### 16.4 AUDIT FINDINGS

Internal or External Audit findings should be documented using the Audit database (refer to Section 13). The audit findings are entered into the database, and Corrective Action forms are generated. This facilitates the tracking and follow-up on audit findings, as well as the generation of audit response reports. The corrective action process is the same as that used for the Corrective Action database. The laboratory is expected to prepare a response to audit findings within 30 days of receipt of an audit report unless the report specifies a different time frame. The response may include action plans that could not be completed within the 30 day timeframe. In these instances, a completion date must set and agreed to by operations management and the QA Manager.

Responsibility for developing and implementing corrective actions to findings is the responsibility of the Operations Manager or Group Leader where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

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If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

The procedures must be in accordance to SOP No. CA-L-S-001, Internal Investigations of Data Discrepancies and Determination of Data Recall.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

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## Figure 16-1

## **Example - Internal Audit Workbook**



### TestAmerica <Name>

Last Updated: 9/10/2007

Workbook Instruction No. CA-Q-WI-011

#### Internal Audit Workbook Summary Page

Note: Click on the (Summary Page) to located on each audit sheet to return to this page.

\* The lab may choose to audit these areas with each method/analyst/instrument audit. The auditor must document on the checklist that this item is audited as part of the <defined> audit.

Area Audited	Audit Type	Audit Cycle	Scheduled	Date Audited	<b>Date Closed</b>	Comments
(Click on the Area to get to that Spreadshe	eet)			/		
Balances	System	6 mo				
Temperature Logs/Thermometers	System	6 mo				
Sample Storage and Disposal	System	1 yr				
Maintenance Logs *	System	6 mo				
Volatile Storage Blanks	System	6 mo				
Lab Water Quality Testing	System	6 mo				
Sample Log In	System	1 yr				
Shipping Procedures	System	1 yr				
Computer Operations (LIMS)	System	1 yr		Per	nding Corp. IT	Policies
SOP & Document Distribution System	System	1 yr				
Archiving Electronic & Paper Records	System	1 yr		Pei	nding Corp. IT	Policies
Statistical Process Control	System	1 yr				
Data Review System	System	1 yr				
Final Report Generation	System	1 yr				
Standards/Reagents *	System	6 mo				
Manual Integration *	System	1 yr				
Corrective Action System	System	1 yr				
Training Records	System	6 mo				
MDLs	System	1 yr				
SOPs - Prep/Review/Update Process	System	1 yr				
Purchasing/Procurement	System	1 yr				
Eppendorf/Diluter/Dispenser Calibration Check	System	6 mo				
Subcontract Lab Approval	System	1 yr				
Customer Complaint System	System	1 yr		6		
Methods	Method	2 yr				
	****	***				
· · · · ·	4444	3443		12-		
100		****				

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Figure 16-2

## **Example – Internal Audit System Checklist: Corrective Actions**

To	act A morica					TestAmerica <location></location>	
	estAmerica					INTERNAL AUDIT - Corrective Actions	
THE L	EADER IN ENVIRONMENTAL TESTING					[ Printed Name(s) or Date(s) ]	
	(Summary Page) Area Audited:					[Fillited Name(s) of Date(s)]	
	Auditor:						
	Additor:						
	Pers	ons Contacted	During				
	Date Repo	rted to Departn	nent M Repo				
	Date Repor	ted to Lab Dire	ctor/M	anac	ıer:		
			Repo				
		Date R	espons	se Du	ıe:		
	Response Received ar	nd Accepted by	QA M	lanaç	jer:		
	Associated Correcti	ive Action Repo	ort Nur	nber	(s):		
		Schedu	led Fo	llow-	up:		
Itom	Requirement	Ref.	٧.	M	NA	Evidence/Comments	Follow
пеш	Requirement	Ker.	Υ	N	NΑ	Evidence/Comments	ОР
1	Does the laboratory have a corrective action program in place?	5.4.10.1					
2	Does the laboratory have a current corrective action SOP or is this	5.4.10.1					
3	information in the QA Manual?	5.4.10.1			_		
3	Do all laboratory personnel have documented training and access to initiate corrective actions?	5.4.10.1					
4	Are causes clearly identified by department, staff name, scope of	5.4.10.6					
	issue (how many reports affected)?						
5 6	Is a root cause for the issue identified?	5.4.10.2		H	_		
7	Is a corrective action (plan) clearly described? Was the corrective action fully implemented?						
8	Is documentation (if applicable) completed as specifed by the			H	_		
	corrective action (training, revised SOP, etc)						
9	Has a follow-up assessment been conducted to verify the corrective						
40	action was successful?	5 4 40 0 - 5		Ш			
10	Are corrective actions reviewed on a regular basis by management?	5.4.10.6a 5					
11	Is there a defined distribution flow for corrective action notification,	5.4.10.6a					
40	review, closure, and follow-up?						
12	Are non-conformances reviewed on a regular basis and used, if necessary, to initiate root cause corrective actions?						
13	Does the lab have a documented procedure for QC corrective action (i.e.,	4.10.1					
	documented within each method / parameter SOP or in the QA Manual)?						
14	Verify Corrective Actions from previous systems audits. L	ist Items:					
15							
16							
17							
	Auditor Signature:  Primary Reference(s):	_ Corporate SOF				cceptable Manual Integration Practices	
						/ersion 3, January 2006	
1		EPΔ Manual fo	r the Co	ertific	ation	of Laboratories Analyzing Drinking Water	

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#### **SECTION 17**

# MANAGEMENT REVIEWS (NELAC 5.4.14)

#### 17.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director for review and comments. The final report shall be submitted to the Technical Directors and Operation Manager as well as the appropriate Quality Director and General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. At a minimum, the report content will contain the items listed below. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

The TestAmerica QA Report template is comprised of a discussion of three key QA issues facing the laboratory and ten specific sections (Figure 17-1):

- **Metrics**: Describe actions or improvement activities underway to address any outlying quality metrics that have been reported in the monthly Quality System Metrics Table.
- SOPs: Report SOPs that have been finalized and report status of any outstanding SOP reviews.
- Corrective Actions: Describe highlights and the most frequent cause for report revisions and corrective/preventive action measures underway. Include a discussion of any recalls handled at the lab level as per Section 6.2.2 in the Investigation/Recall SOP (SOP: CA-L-S-001). Include a section for client feedback and complaints. Include both positive and negative feedback. Describe the most serious client complaints and resolutions in progress.
- MDLs and Control Limits: Report which MDLs/ MDL verifications are due. Report the same for Control Limits.
- Audits: Report Internal and External Audits that were conducted. Include all relevant information such as which methods, by whom, corrective actions needed by when and discuss unresolved audit findings.
- **Performance Testing (PT) Samples:** Report the PT tests that are currently being tested with their due dates, report recent PT results by study, acceptable, total reported and the month and year.
- **Certifications:** Report on any certification programs being worked on by due date, packages completed. Describe any issues, lapses, or potential revocations.
- Regulatory Updates: Include information on new state or federal regulations that may impact the laboratory. Report new methods that require new instrumentation, deletion of methods, changes in sampling requirements and frequencies etc...
- Miscellaneous: Include any issues that may impact quality within the laboratory.
- Next Month: Report on plans for the upcoming month.
- Lab Director Comments Section: This section gives the Laboratory Director the opportunity to comment on issues discussed in the report and to document plans to resolve

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these issues. Unresolved issues that reappear in subsequent monthly reports must be commented on by the Laboratory Director.

• Quality System Metrics Table: The report also includes statistical results that are used to assess the effectiveness of the quality system. Effective quality systems are the responsibility of the entire laboratory staff. Each laboratory provides their results in a template provided by Corporate QA (Figure 17-2).

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Executive Director-QA/EHS prepares a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Analytical Division Senior Management Team and General Managers.

## 17.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Technical Directors, Operations Manager, and QA Manager) conducts an annual review of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This review uses information generated during the preceding year to assess the "big picture" by ensuring that routine quality actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review (refer to Section 17.1) should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review
- Prior Monthly QA Reports issues
- Laboratory QA Metrics
- Review of report reissue requests
- Review of client feedback and complaints
- Issues arising from any prior management or staff meetings
- Minutes from prior Senior Management team meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources
  - Adequacy of policies and procedures
  - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),

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• Compliance with the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

The annual review includes the previous 12 months. Based on the annual review, a report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants
- A reference to the existing data quality related documents and topics that were reviewed
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

The QA Manual is also reviewed at this time and revised to reflect any significant changes made to the quality systems.

## 17.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The Corporate Data Investigation/Recall SOP shall be followed (SOP No. CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

The Chairman/CEO, President/CEO, COO and Quality Directors receive a monthly report from the VP of Quality and EHS summarizing any current data integrity or data recall investigations as described in SOP No. CA-L-S-001. The General Manager's are also made aware of progress on these issues for their specific labs.

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#### Figure 17-1

#### **Example - QA Monthly Report to Management**

LABORATORY: x

PERIOD COVERED: Month/Year

PREPARED BY: x DATE: Month Day, Year

DISTRIBUTED TO: xx (Include LD, GM, QA Director, etc...)

#### THREE KEY ISSUES FOR MONTH:

Include a discussion of three key issues that were focused in on this month.

1. x

2. x

3. x

#### 1. METRICS

Describe actions or improvement activities underway to address any outlying quality metrics.

#### 2. SOPs

See Tab for SOP specifics.

The following SOPs were finalized (or reviewed for accuracy): (See Tab)

The following SOPs are due to QA: xx

In QA to complete: xx

#### 3. CORRECTIVE ACTION

Highlights: xx

#### **Revised Reports:**

Describe the most frequent cause for report revisions and corrective/preventive action measures underway.

Data Investigations/Recalls (Corporate Data Investigation/Recall SOP): Include a discussion of any recalls handled at the lab level as Corp SOP.

#### Client Feedback and Complaints:

Include both positive and negative feedback.

Describe the most serious client complaints) and resolutions in progress.

#### 4. MDLs AND CONTROL LIMITS

MDLs Due:

Control Limits Due:

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LAB DIRECTOR REVIEW:

INTERNAL AUDITS	
Discuss Any Outstanding Issues (or Attach Summary):	
EXTERNAL AUDITS Discuss Any Outstanding Issues (or Attach Summary):	
6. PT SAMPLES	
The following PT samples are now in house (Due Dates):	
7. CERTIFICATIONS	
Certification Packages Being Worked On (Include Due Date):	
Describe any issues, lapses, or potential revocations.	
8. REGULATORY UPDATE Include information on new state or federal regulations that may impact the laboratory – new methods that require new instrumentation, deletion of methods, changes in sampling requirements or frequencies,	
9. MISCELLANEOUS Include any issues that may impact quality within the laboratory.	
10. NEXT MONTH Items planned for next month.	
LAB DIRECTOR COMMENTS AND PLANNED CORRECTIVE ACTIONS:	

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## **Figure 17-2.**

## **Example - Laboratory Metrics Categories**

# Reports for month
# Reports revised due to lab error
% Revised Reports
# of Data Recall Investigations
# of Reports Actually Recalled
# Corrective Action Reports
# Corrective Action Reports still open
Total Number of Unresolved Open Corrective Action Reports
% of Unresolved Open Corrective Action Reports
# Reports independent QA reviewed
% QA Data Review: Reports
# Technical staff (Analysts/technicians, including Temps)
# of Analyst work product reviewed year-to-date
# of Analytical instruments w/electronic data file storage capability
# of Analytical instruments reviewed for data authenticity year-to-date
% Analyst/Instrument Data Authenticity Audits
# Client Complaints
# Client Compliments
# of planned internal audits
# of planned internal method audits performed year-to-date
% Annual Internal Audits Complete
# of Open Internal Audit Findings Past Due
Total Number of External Audit Findings
# of Open External Audit Findings Past Due
% External Audit Findings Past Due
# of PT analytes participated and received scores
# of PT analytes not acceptable
% PT Cumulative Score
# PT Repeat Analyte Failures Cumulative (analyte failed more than once in 4 consecutive studies by PT Type) (only applies to failed analytes)
# SOPs

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# SOPs Reviewed/revised within 24 months

# Methods or Administrative procedures without approved SOPs

**SOP Status** 

Method certification Losses due to performance/audit issues

**Hold Time Violations due to lab error** 

**Date of Last Comprehensive Ethics Training Session** 

# Staff that haven't Received Comprehensive Ethics Training (>30 Days From Employment Date)

MDL Status (Good, Fair, or Poor) >90%, >70%, <70%

**Training Documentation Records (Good, Fair, or Poor)** 

**LQM** Revision/review Date

**QAM Updated to New Integrated Template** 

Last Annual Internal Audit Date (Opened, Closed)

**Last Management QS Review Date** 

#SOPs required for 12 month review cycle (DOD or drinking water)

#SOPs for 12 month cycle/revised within 12 months (Includes QS and Methods Listed in QSM)

12 month % SOP Status (Includes QS and Methods Listed in QSM)

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#### **SECTION 18**

PERSONNEL (NELAC 5.5.2)

### 18.1 OVERVIEW

TestAmerica's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Appendix 2.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

# 18.2 <u>EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL</u>

TestAmerica makes every effort to hire analytical staff that posses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. There are competent analysts and technicians in the industry who have not earned a college degree. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are

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located in the employees personnel files in the Human Resources office in the Irvine facility (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc. are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), Titrimetric and Gravimetric Analyses, or Microbiology methods	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Directors/Department Managers – General	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry  An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Director – <u>Wet Chem</u> only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

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Specialty	Education	Experience
Technical Director - Microbiology	Bachelors degree in applied science with at least 16 semester hours in general microbiology and biology	And 2 years of relevant experience
	An advanced (MS, PhD.) degree may substitute for one year of experience	

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Group Leader, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

#### 18.3 TRAINING

TestAmerica is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame*	Employee Type
Environmental Health & Safety	Refer to EH&S	All
	Manual	
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive	Annually	All
Refresher		
Initial Demonstration of	Prior to unsupervised	Technical
Capability (DOC)	method performance	

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 20.

The training of technical staff is kept up to date by:

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• Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.

- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in the laboratory's Training SOP.

## 18.4 <u>DATA INTEGRITY AND ETHICS TRAINING PROGRAM</u>

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established an Ethics Policy No. CA-L-P-001 and an Ethics Statement/Agreement (Appendix 1). All initial and annual training is documented by signature on the signed Ethics Policy and Code of Ethical Conduct demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy (Appendix 1)
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.

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• Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)

- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

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#### **SECTION 19**

# ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS (NELAC 5.5.3)

#### 19.1 OVERVIEW

**TestAmerica Ontario, CA** is a 7,500 ft<sup>2</sup> secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc.. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic and inorganic sample analysis, microbiological sample analysis, and administrative functions.

#### 19.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include temperature levels in the laboratory.

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When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels (refer to Section 12).

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

#### 19.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Microbiological culture handling and sample incubation areas
- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory
- Sample receipt areas
- Sample storage areas
- Chemical and waste storage areas
- Data handling and storage areas
- Sample processing areas
- Sample analysis areas

Refer to Standard Methods, 20<sup>th</sup> Ed., 9020B, Section 2 for specific requirements for microbiological laboratory facility requirements.

#### 19.4 FLOOR PLAN

A floor plan can be found in Appendix 3.

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# 19.5 **BUILDING SECURITY**

Building keys and alarm codes are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of *TestAmerica Ontario, CA*. In addition to signing into the laboratory, the *Environmental, Health and Safety Manual* contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

Signs are posted in the laboratory designating employee only areas - "Authorized Personnel Only" or "Employees Only".

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#### **SECTION 20.0**

# TEST METHODS AND METHOD VALIDATION (NELAC 5.5.4)

#### 20.1 OVERVIEW

**TestAmerica Ontario, CA** uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

#### 20.2 STANDARD OPERATING PROCEDURES (SOPs)

**TestAmerica Ontario, CA** maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory (refer to Section 6 on Document Control):

- All SOPs contain a revision number, effective date, and appropriate approval signatures.
   Controlled copies are available to all staff.
- Procedures for preparation, review, revision and control are incorporated by reference to SOPs: CW-Q-S-002 (Writing a Standard Operating Procedure (SOP) and the laboratory's Document Control SOP.
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

# 20.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP. Refer to the corporate SOP CW-Q-S-002 "Writing a Standard Operating Procedure" for content and requirements of technical and non-technical SOPs.

**Note:** If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from

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the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

# 20.4 <u>SELECTION OF METHODS</u>

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc..), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

### 20.4.1 **Sources of Methods**

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

In general, TestAmerica Ontario, CA follows procedures from the referenced methods shown below in 20.3.1.4.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

**20.4.1.1** The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- <u>Prescribed Procedures for Measurement of Radioactivity in Drinking Water</u>, EPA-600/4-80-032, August 1980.
- Analytical Method for Determination of Asbestos Fibers in Water, EPA-600/4-83, September 1983.
- <u>Determination of Asbestos Structures Over 10-mm in Length in Drinking Water</u>, EPA-600/R-94-134, June 1994.
- Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.

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- Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039,
  December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II,
  EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 August 1995 (EPA 500 Series)
  (EPA 500 Series methods)
- Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994
- NIOSH Manual of Analytical Methods, 4<sup>th</sup> ed., August 1994.
- <u>Standard Methods for the Examination of Water and Wastewater</u>, 18<sup>th</sup>/19<sup>th</sup> /20<sup>th</sup> edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- <u>Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)</u>, Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.
- Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)
- Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261
- <u>Determination of 1,2,3-Trichloropropane in Drinking Water by Purge and Trap Gas Chromatography/Mass Spectrometry (CA DHS DDWEM SRL, February 2002)</u>

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

# 20.4.2 **Demonstration of Capability**

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

- **20.4.2.1** A demonstration of capability is performed whenever there is a change in instrument type, method or personnel.
- 20.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures (refer to Section 15, Control of Records).

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20.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

**Note:** In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method).
- The reporting limit is set at or above the first standard of the curve for the analyte.
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*
- Refer to Section 12 (Control of Non-Conforming Work).

# 20.4.3 <u>Initial Demonstration of Capability (IDOC) Procedures</u>

Refer to the laboratory's Training SOP for specific training procedures.

A certification statement (see Figure 20-1 as an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

# 20.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP/Methods Manual (Section 20.2) and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method. The information included in the checklist below (Figure 20-2) is needed before samples are accepted for analysis by a new method.

#### 20.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled. (From 2003 NELAC Standard)

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to

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meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

#### 20.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

#### 20.6.1.1 <u>Determination of Method Selectivity</u>

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

#### 20.6.1.2 <u>Determination of Method Sensitivity</u>

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. The laboratory determinations of MDLs are described in Section 20.6.

# 20.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

#### 20.6.1.4 <u>Determination of Interferences</u>

A determination that the method is free from interferences in a blank matrix is performed.

# 20.6.1.5 <u>Determination of Range</u>

Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of

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quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

#### 20.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

#### 20.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

#### 20.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

# 20.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 20.7.10). The analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

- 20.7.1 MDL's are initially performed for each individual instrument and non-microbiological method analysis. Unless there are requirements to the contrary, the laboratory will use the highest calculated MDL for all instruments used for a given method as the MDL for reporting purposes. This MDL is not required for methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report values to the MDL. Titration and gravimetric methods where there is no additional preparation involved, the MDL is based on the lowest discernable unit of measure that can be observed.
- 20.7.2 MDL's must be run against acceptable instrument QC, including ICV's and Tunes. This is to insure that the instrument is in proper working condition and falsely high or low MDL's are not calculated.

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- 20.7.3 Use only clean matrix which is free of target analytes (e.g.: Laboratory reagent water, Ottawa Sand) unless a project specific MDL is required in a field sample matrix.
- 20.7.4 The Reporting Limit (also may be referred to as Limit of Quantitation or LOQ) should generally be between 2 and 5 times the MDL. If the MDL is being performed during method development, use this guideline to determine the Reporting Limit for the analysis. If a sample is diluted, the reported MDL is adjusted according to the dilution factor.
- 20.7.5 The calculated MDL cannot be greater than the spike amount.
- 20.7.6 If the most recent calculated MDL does not permit qualitative identification of the analyte then the laboratory may use technical judgment for establishing the MDL (e.g., calculate what level would give a qualitative ID, compare with IDL (20.7), spike at a level where qualitative ID is determined and assign that value as MDL, minimum sensitivity requirements, Standard deviation of method blanks over time, etc.). These alternate verification procedures must be documented in the MDL section of the specific method SOP.
- 20.7.7 Each of the 7 or 8 spikes must be qualitatively identifiable (e.g., appear in both columns for dual column methods, characteristic ions for GCMS mass spectra, etc). Manual integrations to force the baseline for detection are not allowed.
- 20.7.8 The initial MDL is calculated as follows:

 $MDL = t_{(n-1, 1-a=0.99)} x$  (Standard Deviation of replicates)

where  $t_{(n-1, 1-a=0.99)} = 3.143$  for seven replicates. or where  $t_{(n-1, 1-a=0.99)} = 2.998$  for eight replicates.

- 20.7.9 Subsequent to the initial MDL determination, periodic MDL verification, confirmation or determinations may be performed by the procedure in <u>40 CFR Part 136</u>, <u>Appendix B</u> or alternatively by other technically acceptable practices (e.g., method blanks over time, single standard spikes that have been subjected to applicable sample prep processes, etc.). The procedures utilized are documented in the laboratory's MDL SOP.
- 20.7.10 Because of the inherent variability in results outside of the calibration range, TestAmerica does not recommend the reporting of results below the lowest calibration point in a curve; however, it is recognized that some projects and agencies require the reporting of results below the RL. Any result that falls between the MDL and the Reporting limit, when reported, will be qualified as an estimated value.
- 20.7.11 Detections reported down to the MDL must be qualitatively identified.
- 20.7.12 MDLs and Reporting limits are adjusted in LIMs based on moisture content and sample aliquot size.

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# 20.8 <u>INSTRUMENT DETECTION LIMITS (IDL)</u>

20.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

- 20.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.
- 20.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

# 20.9 <u>VERIFICATION OF DETECTION AND REPORTING LIMITS</u>

- 20.9.1 Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and 1-4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified or see section 20.6.7 for other options. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established (See 20.7.9). MDLs must be verified at least annually.
- 20.9.2 When a Reporting limit is established, it must be initially verified by the analysis of a low level standard or QC sample (LCS at 1-2 the reporting limit) and annually thereafter. Unless there are requirements to the contrary the acceptance criteria is  $\pm$  50%. The annual requirement is waved for methods that have an annually verified MDL.

For Drinking Water compliance methods, the minimum reporting limit (MRL) must be verified each analysis day. This verification involves analyzing an LCS spiked at the MRL. The MRL must meet any state or Federal regulations for Drinking Water reporting limits and the MRL must be below the Maximum Contamination Limit (MCL).

# 20.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method for GC, HPLC, and IC methods or as specified by the method for GCMS methods. These records are kept with the files associated with an instrument for later quantitation of the analytes.

For GC, HPLC and IC methods, there must be sufficient separation between analyte peaks so as to not misidentify analytes. In the mid-level standard, the distance between the valley and peak height cannot be any less than 25% of the sum of the peak heights of the analytes. This also applies to GCMS in the case where the two compounds share the same quantitation ion.

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**Note:** Some analytes do not separate sufficiently to be able to identify or quantitate them as separate analytes (e.g. m-xylene and p-xylene) and are quantitated and reported as a single analyte (e.g. m,p-xylenes).

Once the analyst has determined that the instrument is in optimum working condition through calibration and calibration verification procedures, they use a mid-range calibration or calibration verification standard to establish the retention times for each of the individual analytes in a method. The analyst makes three injections of the same standard over a 72-hour (24 hour period for 300.0) period, tabulating the retention times for each analyte for each of the three injections. The width of retention time window is normally the average absolute retention time  $\pm$  3 Standard Deviations. A peak outside the retention time window will not be identified by the computer as a positive match of the analyte of interest.

It is possible for the statistically calculated RT window to be too tight and need to be adjusted based on analyst experience. In these instances method default retention time windows may be used (e.g., for 8000 series methods a default of 0.03 minutes may be used, and EPA CLP 0.05 minutes is used). The same concept is applied when any peak outside of that window will not be identified by the computer as a positive match.

The calibration verification standard at the beginning of a run may be used to adjust the RT for an analyte. This is essentially re-centering the window but the size of the window remains the same. The RTs are verified when all analytes are within their RT windows and are properly identified.

#### 20.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, chromatography retention time windows, sample blanks, spectrochemical, fluorescence profiles, and specific electrode response factors.

#### 20.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

- 20.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.
- 20.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable,

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assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

- 20.12.3 The uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.
- 20.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 +/- 0.5 mg/l.
- 20.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g. 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

# 20.13 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

#### 20.13.1 Computer and Electronic Data Related Requirements

The three basic objectives of the laboratory's computer security procedures and policies are shown below. A more detailed outline can be found in the laboratory's Computer Security SOP. The laboratory is currently running the Element LIMS which is a 3<sup>rd</sup> party LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes an SQL Database which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

- **20.13.1.1** Maintain the Database Integrity: Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.
  - LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
  - Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

**Note:** "Commercial off-the-shelf software in use within the designed application range is considered to be sufficiently validated." From NELAC 2003 Standard.

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However, laboratory specific configurations or modifications are validated prior to use.

- In order to assure accuracy, all data entered or transferred into the LIMS data system goes through a minimum of two levels of review.
- The QA department performs random data audits to ensure the correct information has been reported.
- Changes to reports are documented in the Corrective Action database.
- Analytical data file security is provided through three policies.
  - The first policy forbids unauthorized personnel from using laboratory data acquisition computers.
  - The second policy is the implementation of network passwords and login names that restrict directory access.
  - The third layer is maintained through the LIMS and includes the use of username/password combinations to gain access to the LIMS system, the fact that all data in the LIMS is associated with the user to added/reviewed the data, and the restriction of review authority of data.
- All software installations will be in accordance with any relevant copyright licensing regulations.
- All software installed on any computer within the laboratory must be approved by the Information Technology Department regional support technician assigned to the laboratory Shrink-wrapped or otherwise sealed OEM software that is directly related to instrument usage does not need approval but the Information Technology department must be notified of the installation.
- Anti-virus software shall be installed on all servers and workstations. The anti-virus software shall be configured to check for virus signature file and program updates on a daily basis and these updates will be pushed to all servers and workstations. The anti-virus software will be configured to clean any virus-infected file if possible, otherwise the file will be deleted. Disks and CDs brought from any outside source that are not OEM software must be scanned for viruses before being accessed.

# Interlab LIMS Permissions Policy

- PURPOSE The purpose of this policy is to provide a mechanism for maintaining the integrity of information contained in each laboratory's LIMS while providing the necessary access for information sharing to staff at other laboratory facilities.
- <u>DEFINITIONS</u> Host Laboratory: The laboratory facility that 'owns' the LIMS system or 'hosts' a project/job.

#### POLICIES

- (a) All permissions for the laboratory's LIMS system must only be granted by a representative of that laboratory.
  - If someone outside of the host lab needs permissions for Project
    Management or other uses, they must go through the Lab Director or his/her
    designated representative.
  - Permissions must never be granted without the knowledge of the host laboratory.
  - (b) Only laboratory analytical or QA staff from the home laboratory may have edit permissions for laboratory analysis data.
  - (c) Any changes made in laboratory's LIMS system:
  - Must be documented and traceable.

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- If made by staff of an affiliate lab, written permission from the home lab to make the changes (email approval is sufficient) is required.
- No corrections may be made in another laboratories system without their knowledge.
- (d) Data qualifiers in laboratory reports must only be corrected, edited, etc. by the staff at the host laboratory.
- (e) Full analytical data "View" only permissions may be granted to outside Project Management and Sales staff. Query Search permissions may also be granted so status may be checked.
- (f) All qualifiers must be approved by QA staff before adding them to the Element Static Tables and must be approved by Corp QA.
- (g) Please contact Corporate QA or IT staff if you have any questions regarding implementation or interpretation of this policy.
- **20.13.1.2** Ensure Information Availability: Protection against loss of information or service through scheduled back-ups, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.
  - Insured by timely backup procedures on reliable backup media, stable file server network architecture, and UPS protection
  - UPS Protection:
    - Each fileserver is protected by an appropriate power protection/backup unit. In the
      event of a power outage, there is approximately 15-30 minutes of up-time for the
      servers prior to shutdown. This allows for proper shutdown procedures to be
      followed with the fileservers.
  - File Server Architecture
    - All files are maintained on multiple Windows 2000 or newer servers which are secured physically in the Information Technology office. Access to these servers is limited to members of the laboratory Senior Management staff.
    - All supporting software is maintained for at least 10 years from the last raw data generated using that software.
  - System Back-up Overview and Procedures
    - Data from both servers and instrument attached PC's are backed up and purged in compliance with the corporate back-up policy.
    - A Maintenance Plan has been defined to create a daily archive of all data within the LIMS database to a backup location. This backup is initiated automatically by either the database or back-up system.
    - Backup tapes will be stored in compliance with the corporate Data Backup Policy.
       Backup verifications are carried out in accordance with the corporate Data Backup Policy.
    - Instrument data back-ups are verified on a periodic basis by the QA department when performing electronic data audits. The audit takes place on data that has been moved to a back-up location ensuring that it has been moved.

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**20.13.1.3** <u>Maintain Confidentiality:</u> Ensure data confidentiality through physical access controls, and encryption of when electronically transmitting data.

- All servers are located in a secure area of the IT department offices. Access to the servers is limited to IT staff members, lab Senior Management staff, the President and Vice President of Operations.
- The company website contains SSL (Secure Socket Layer) encryption for secure website sessions and data transfers.
- The reporting portion of the LIMS system requires a project manager to enter their unique password anytime they create a report that displays a signature on it (.PDF).
- Electronic documents such as PDF files and electronic data deliverables will be made available to clients via the secure web site. The logon page for this web site contains an agreement that the customer must accept before they will be logged on which states that the customer agrees not to alter any electronic data made available to them.
- If electronic documents are made available outside of the web site, the customer must sign an agreement in advance that states they will not alter the data in any way.

# 20.13.2 **Data Reduction**

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry or Microbiology, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices*, and the laboratory's Manual Integration SOP

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

**20.13.2.1** All raw data must be retained in the daily analysis folder, computer file (if appropriate), and/or run log. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/<u>year</u>). It must be easily identifiable who performed which tasks if multiple people were involved.

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**20.13.2.2** In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter ( $\mu$ g/l) for liquids and milligrams per kilogram ( $\mu$ g/kg) or micrograms per kilogram ( $\mu$ g/kg) for solids. The units "mg/l" and "mg/kg" are the same as "parts per million (ppm)". The units " $\mu$ g/l" and " $\mu$ g/kg" are the same as "parts per billion (ppb)." For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%.

- Several environmental methods, such as color, turbidity, conductivity, use very specific, non-concentration units to report results (e.g., NTU, umhos/cm etc).
- Occasionally, the client requests that results be reported in units which take into account the measured flow of water or air during the collection of the sample.
   When they provide this information, the calculations can be performed and reported.
- 20.13.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report, with three for QC results to accommodate percent recoveries equal to or greater than 100%.
- 20.13.2.4 For those methods or data that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results, dilution factors, sample volumes, etc., are documented in a handwritten logbook, bench sheet, or log sheet. These sets of data are then entered directly into LIMS by the analyst, where the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.
- 20.13.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file.
- 20.13.2.6 The electronic data files are stored in a monthly folder on the instrument computer or directly to the network Data folder. Files that are stored on the local computer hard drives are transferred to the network Data folder 6 nights a week. Data is deleted from the local hard drives after 100 days. The network Data folder is backed up to a Network Attached Storage (NAS) device, keeping 6 months worth of data on the Data folder. These backups are completed automatically through the use of a program created by the IT department.

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#### 20.13.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 13.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"d out, signed and dated.
- Worksheets are created with the approval of the Operations Manager and/or QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

#### 20.13.4 Review / Verification Procedures

Review procedures are out lined in the individual method SOPs, the General Data Review SOP, and the Data Qualifiers SOP to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data. The general review concepts are discussed below, more specific information can be found in the SOPs.

- **20.13.4.1** The data review process at *TestAmerica Ontario, CA* starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. Sample Control Staff peer review the transaction of the chain-of-custody forms and the inputted information. The Project Management Assistants perform final review of the chain-of-custody forms and inputted information.
- **20.13.4.2** The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add data qualifiers if applicable (see Appendix 7 for list of common data qualifiers).
- 20.13.4.3 To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Approximately 15% of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. Manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies for the GCMS instruments. Issues that deem further review include the following:

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- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range
- **20.13.4.4** Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Manager, Operations Manager, or Group Leader for further investigation. Corrective action is initiated whenever necessary.
- **20.13.4.5** A hard copy (or .pdf) final report is then printed for the client.
- 20.13.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters or narratives are present, flags are appropriate, and project specific requirements are met. The following are some examples of chemical relationships that are reviewed (if data is available):
  - Total Results are ≥ Dissolved results (e.g. metals)
  - Total Solids (TS) ≥ TDS or TSS
  - TKN ≥ Ammonia
  - Total Phosphorus ≥ Orthophosphate
  - COD ≥ TOC
  - Total cyanide ≥ Amenable Cyanide
  - TDS ≥ individual anions
- **20.13.4.7** The Project Manager then signs the final report. (*Also see section 26 on Reporting Results*). The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is delivered to the client.
- **20.13.4.8** A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 20-3.

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# 20.13.5 **Manual Integrations**

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines and the laboratory's Manual Integration/Data Integrity SOP.

- 20.13.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.
- 20.13.5.2 Analysts shall not increase or decrease peak areas to for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.
- **20.13.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- 20.13.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

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# Figure 20-1 Demonstration of Capability Documentation

		TRATION OF CAP	ABILITY	7		
	CERTIF	FICATION STATE	MENT			
	ory Name: <b>TestAmerica - C</b> ory Address <b>1014 E. Cooley</b>	Ontario Dr., Suite A, Coltoi	n, CA 923	324		
Matrix: SOP # a Paramete Analyte Comme	Group:	Section:	Rev.	Rev. Date (	)	
We, the und	ersigned, CERTIFY that:					
1.	The analysts identified abouse at this facility for the a Environmental Laboratory Demonstration of Capabili	nalyses of samples u Accreditation Progr ty.	inder the N am, have i	National met the		
2.	The test method(s) was performed by the analyst(s) identified on this certification.					
3.	A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.					
4.	The data associated with the demonstration capability are true, accurate, complete, and self explanatory. 1					
5.	All raw data (including a c reconstruct and validate the and that the associated info review by authorized asses	ese analyses have be ormation is well orga	en retaine	d at the facility,		
Technical Director's Name and Title		Signature			Date	
Quality Assurance Manager		Signature			Date	
Accura Compl	Consistent with supporting data.  te: Based on good laboratory practete: Includes the results of all suppopulanatory: Date properly labeled a ation.	orting performance testing	ng.		ional	
ing ID#:				Issued:		

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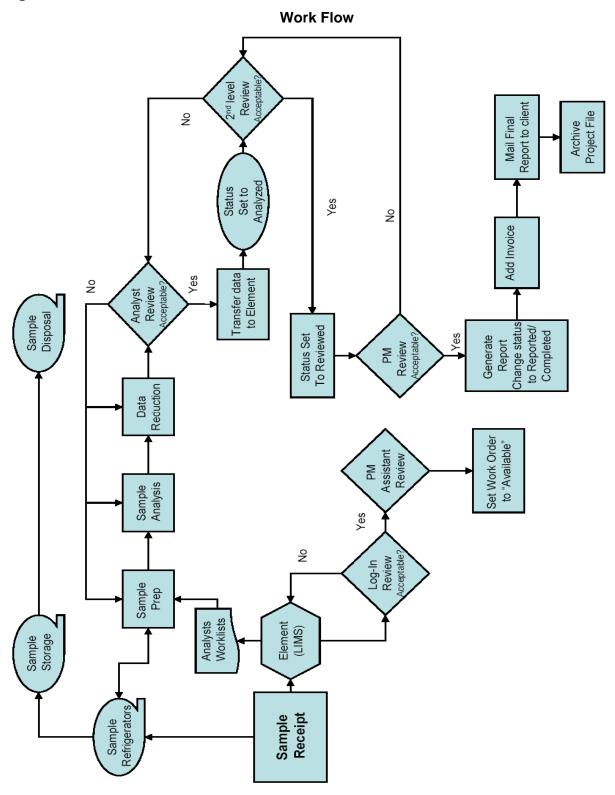
# Figure 20-2

# **New Method / Additional Analyte Checklist**

# **New Method / Additional Analyte Checklist**

not ap			or QC samples are analyzed (includes run
log).	New Method	Adde	ed Analytes
	Analysis SOF Preparation S SOP for any Pages from a	ytes, a <b>ROMD</b> can be used to ado SOP other relevant process ny applicable logbooks (instrume	•
2		pplicable: e.g. Retention Time V s, spectral or fluorescence profile	Vindow Study, second column confirmation, es, etc.
3	Initial Calibration Curve (Include	de Tune verification or similar (e.ç	g. degradation checks) if applicable)
4	Method Detection Limit (MDL) Water Soil Other	Study (summary and raw data)	
5		methods /-846 methods (if applying for soi be used in lieu of tap water if it is	
6			ntire method. MDL study should be able to be overy( <del>y</del> ies)
7	<ul> <li>4 LCS for each matrix –</li> <li>DOC criteria are met.</li> <li>Non-Standard methods -</li> </ul>	·	ne methods. The MDL study may be used if , 75% of the calibration range + Blank)
8	Acceptable PT sample(s) if av	ailable	
	Notes: PT sample required PT sample required	for all new methods for all new analytes under NELA	Р
Subm	itted by	Date	
9	Certification/Approval from Re	gulatory Agency where available	<b>).</b>
QA R	eview / Acceptance	Da	te

Figure 20-3



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#### **SECTION 21**

# EQUIPMENT (AND CALIBRATIONS) (NELAC 5.5.5)

# 21.1 OVERVIEW

TestAmerica purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory method specific SOPs and in the Instrument Calibration SOP. A list of laboratory equipment and instrumentation is presented in Table 21-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

# 21.2 PREVENTIVE MAINTENANCE

- **21.2.1 TestAmerica Ontario, CA** follows a well-defined program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.
- **21.2.2** Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.
- **21.2.2.1** Calibrations, routine maintenance, and adjustments are part of the analysts', Group Leaders', and the Operations Manager's responsibilities. Service contracts may be in place for some instruments to cover any major repairs.
- **21.2.2.2** High purity gases, reagents, and spare parts are kept on hand to minimize repair time and optimize instrument performance.
- **21.2.3** Table 21-2 summarizes the schedule for routine maintenance. It is the responsibility of each Group Leader and the Operations Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log.

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**21.2.4** Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- **21.2.4.1** Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- 21.2.4.2 Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.).
- 21.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be referred to on the paginated page of the logbook.
- **21.2.5** In addition, the maintenance records contain:
- The identification of the instrument/equipment (instrument's Serial Number and Model Number)
- The date the instrument/equipment was put into use.
- If available, the condition when the instrument was received (e.g. new, used, reconditioned).
- Any routine maintenance procedures and their required frequency.
- **21.2.6** If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses (refer to Sections 12 and 13).
- 21.2.7 In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted using the procedures outlined in Section 8.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

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# 21.3 <u>SUPPORT EQUIPMENT</u>

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

# 21.3.1 Weights and Balances

Balances and weight sets are calibrated and maintained according to the laboratory's Balance Calibration Verification and Documentation SOP. The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

# 21.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to  $\pm$  0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

#### 21.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers are calibrated quarterly.

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The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) at the levels that it is normally used by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer has increments of 0.2 °C, and has a range applicable to all method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks or certificates from outside calibration services. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the Thermometer Calibration / Temperature Monitoring and Documentation SOP.

# 21.3.4 <u>Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators</u>

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0°C and ≤ 6 °C.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

# 21.3.5 <u>Autopipettors, Dilutors, and Syringes</u>

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are checked for accuracy at least quarterly. Glass micro-syringes are considered the same as Class A glassware.

The laboratory maintains a sufficient inventory of autopipettors, and dilutors of differing capacities that fulfill all method requirements.

These devices are given unique identification numbers, and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements. Refer to the laboratory's Pipette Calibration SOP.

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Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

#### 21.3.6 Autoclaves

Autoclaves used in the laboratory meet all specified temperature tolerances. At a minimum, the date, contents, maximum temperature, pressure, sterilization time, total run time, and the analyst's initials are documented in an Autoclave Logbook. A maximum registering thermometer is used with each cycle to document that the appropriate sterilization temperature was achieved, and temperature sensitive tape is used with each batch run to indicate that the contents have been processed.

Annual maintenance is performed by an outside contractor including a pressure check and calibration of the internal temperature device. The autoclave timing device is verified against a stopwatch and the actual time elapsed is documentation. Documentation of these maintenance procedures is maintained in the autoclave Maintenance Logbook according to the procedures described in Section 15 and previously in this section.

# 21.4 <u>INSTRUMENT CALIBRATIONS</u>

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg. RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 13).

**Note:** Instruments are calibrated initially and as needed after that and at least annually.

# 21.4.1 CALIBRATION STANDARDS

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. However, the general procedures are described below.

**21.4.1.1** For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical

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methods. If a reference or mandated method does not specify the number of calibration standards, the minimum number is three, not including blanks or a zero standard. All of the standard solutions are prepared using Class A volumetric glassware, calibrated pipettes, and/or microsyringes and appropriate laboratory quality solvents and stock standards.

- 21.4.1.2 Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to NIST whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. A standard log in the Element LIMS is maintained for each department, containing concentration, date of receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.
- **21.4.1.3** The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).
- 21.4.1.4 The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The lowest calibration standard must be at or below the reporting limit.
- 21.4.1.5 Given the number of target compounds addressed by some of the organic methods, it may be necessary to prepare several sets of calibration standards, each set consisting of the appropriate number of solutions at different concentrations. The initial calibration will then involve the analysis of each of these sets of the appropriate number of standards.
- **21.4.1.6** All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

#### 21.4.2 CALIBRATION FOR ORGANIC METHODS (GC, HPLC, GC/MS)

- 21.4.2.1 Many of the organic analytical methods utilize an internal standard calibration (GCMS and some GC). Because of the complex nature of the multipeak chromatograms produced by the method, some instruments necessitate the use of external standard calibration (most GC and HPLC). Surrogate compounds are included in the calibration processes for all appropriate organic analyses. For more details on the calibration types listed below, refer to SOP No. CA-Q-S-005, Calibration Curves.
- **21.4.2.2** Once the operating parameters have been established according to the method, each instrument is calibrated for the appropriate method. The analyst prepares five or more

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standard solutions at various concentrations containing all of the analytes of interest, internal standards, and surrogates that are appropriate for the method. Note: There are a several EPA methods that have different requirements and are exceptions (e.g. EPA 547) where a minimum of 3 calibration standards are prepared and analyzed.

- 21.4.2.3 The standard solutions are introduced into the instrument in the same manner as samples are; whether it be by direct injection, by headspace analysis, or by purge and trap. The calibration factor (CF) for methods that use external standards, and the response factor (RF) for methods that use internal standards are calculated for the five standards.
  - External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the response to the amount of analyte in the calibration standard is defined as the Calibration factor (CF).
  - Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. The use of specific internal standards is available in the method SOP.

Whichever internal standards are employed, the analyst needs to demonstrate that the measurement of the internal standard is not affected by method analytes and surrogates or by matrix interferences. In general, internal standard calibration is not as useful for GC and HPLC methods with non-MS detectors because of the inability to chromatographically resolve many internal standards from the target compounds. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

When preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes will vary. The internal standard solution will contain one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution (e.g., not all internal standards need to be at the same concentration in this solution). The mass of each internal standard added to each sample extract immediately prior to injection into the instrument or to each sample prior to purging must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts should be such that minimal dilution of the extract

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occurs (e.g., 10 uL of solution added to a 1 mL final extract results in only a negligible 1% change in the final extract volume which can be ignored in the calculations).

An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard should produce an instrument response (e.g., area counts) that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This should result in a minimum response factor of approximately 0.01 for the least responsive target compound. Refer to SOP No. CA-Q-S-005, Calibration Curves, for specific calculations.

- **21.4.2.4** Policies regarding the use of calibration standard results for creating the calibration curve are as follows:
  - A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting level must be elevated to be the lowest calibration standard used for calibration.
  - The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.
  - Mid-calibration standards may not be excluded unless an obvious reason is found, i.e., cracked vial, incorrectly made, etc. The failed standard should be re-run immediately (generally within the same analytical work shift, but possibly up to 24 to 48 hours, and before anything else is run. Refer to the Calibration Curves (General) SOP, CA-Q-S-005) and inserted into the initial calibration. If not useful, or if anything else is run on the instrument, recalibration is required.

#### 21.4.2.5 Percent RSD Corrective Action

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

- 21.4.2.5.1 The first step is generally to check the instrument operating conditions. This option will apply in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance, changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.
- 21.4.2.5.2 If the RSD for any analyte is greater than the applicable acceptance criteria in the applicable analytical method, the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

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21.4.2.5.3 A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

**Note:** When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the method quantitation limit is at least as low as the regulatory limit or action level.

- 21.4.2.6 Alternatively, the least squares regression may be used to determine linearity. A five point line must result in a correlation coefficient (r) of 0.990 or better using the least squares method to be considered acceptable. \*\*\*\* In many cases it may be preferred that the curves be forced through zero (not to be confused with including the origin as an additional data point, which is not allowed). Note: EPA method 8000B does not allow forcing through zero however the agency has revaluated this position and has since changed this stance to allow forcing through zero. In addition, from EPA Method 8000C: "However, the use of a linear regression or forcing the regression through zero may NOT be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards.").
- 21.4.2.7 Instead of a linear curve model (either Average RF or least squares regression), a second order curve (Quadratic) may be used (and preferred) as long as it contains at least six data points. As a rule of thumb, if there is a consistent trend in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (COD or r²) for the quadratic curve must be at least 0.990 for it to be considered acceptable. For more details on the calculations see Calibration Curve SOP CA-Q-S-005. Some limitations on the use of Quadratic Curve fits:
- **21.4.2.7.1** Care MUST be exercised to assure that the results from this equation are real, positive, and fit the range of the initial calibration.
- **21.4.2.7.2** They **may not** be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).
- **21.4.2.7.3** They **may not** be used to compensate for detector saturation. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve and try a 1<sup>st</sup> order fit or average RF.

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# 21.4.3 <u>Calibration for Inorganic Analyses</u>

EPA Method 7000 from EPA SW-846 is a general introduction to the quality control requirements for metals analysis. For inorganic methods, quality control measures set out in the individual methods and in the *Standard Methods for the Examination of Water and Wastewater* (20th Edition or other edition as required) may also be included. Standard Operating Procedures for the analysis and the quality control documentation measures are kept in the analysts' reference in their work area.

In general, inorganic instrumentation is calibrated with external standards. Some exceptions would be Inductively Coupled Plasma (ICP), Inductively Coupled Plasma Mass Spec (ICPMS), and Ion Chromatography Mass Spec (ICMS). These analyses may use an internal standard to compensate for viscosity or other matrix effects. While the calibration procedures are much the same for inorganics as they are for organics, CF's or RF's are not used. The calibration model in 21.4.2.6 is generally used for most methods, however in some instances the model from section 21.4.2.7 may be used. A correlation coefficient (r) of 0.995 or greater must be used to accept a calibration curve generated for an inorganic procedure. Correlation coefficients are determined by hand-held scientific calculators or by computer programs and documented as part of the calibration raw data. Coefficients of calibration curves used for quantitation must be documented as part of the raw data. Curves are not allowed to be stored in calculator memories and must be written on the raw data for the purposes of data validation.

- **21.4.3.1** "Calibrations" for titrimetric analyses are performed by standardizing the titrants against a primary standard solution. See specific methods in *Standard Methods for the Examination of Water and Wastewater* (20th Edition) for more information.
- **21.4.3.2** Spreadsheets that are used for general chemistry calculations must have all cells containing calculations locked to prevent accidental changes to the calculations.

# 21.4.4 Calibration Verification

The calibration relationship established during the initial calibration must be verified at periodic intervals as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

**Note:** The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration, and is not appropriate nor permitted in SW-846 chromatographic procedures for trace environmental analyses.

21.4.4.1 Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.

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- 21.4.4.2 A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples.
- 21.4.4.3 The acceptance limits for calibration verifications can be found in each method SOP. As a rule of thumb: GCMS ± 20%, GC and HPLC ± 15%, Inorganics: ± 10 or 15%. Actual methods may have wider or tighter limits; see the method SOPs for specifics.
- 21.4.4.4 If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF, RF or % drift values from the initial calibration to quantitate sample results.
- 21.4.4.5 If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new initial instrument calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:
  - **21.4.4.5.1** When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
  - **21.4.4.5.2** When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

#### 21.4.4.6 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

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The Percent Difference is calculated as follows:

% Difference = 
$$(CF(v) \text{ or } RF(v)) - (Avg. CF \text{ or } RF)$$
 X 100 (Avg. CF or RF)

Where: CF(v) or RF(v) = CF or RF from verification standard

Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

The Percent Recovery is calculated as follows:

# 21.4.4.7 <u>Verification of a Non-Linear Calibration</u>

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations described in 21.4.4.6 above.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

**Note:** If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

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# 21.5 POLICY ON TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it will not be reported as a TIC. If the compound is reported on the same form as true TICs, it must be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

- **21.5.1** Use the following guidelines for making tentative identifications
- **21.5.1.1** Sample spectra are compared to the NIST library of reference spectra. These reference spectra have ranges that are wider than the spectra range that our mass spectrometer reads. The sample spectra are only compared to the reference spectra within the range that our mass spectrometer reads.
- **21.5.1.2** Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion within the scanning range of the mass spectrometer) should be present in the sample spectrum.
- 21.5.1.3 The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
- **21.5.1.4** Molecular ions present in the reference spectrum within the scanning range of the mass spectrometer should be present in the sample spectrum.
- 21.5.1.5 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- 21.5.1.6 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

The concentration of any non-target analytes identified in the sample (see above) should be estimated. The same formulae as calibrated analytes should be used with the following modifications: The areas  $A_x$  and  $A_{is}$  should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

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The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

Note: The above guidelines above are from EPA SW846 III edition, method 8260B.

For general reporting if TICs are requested, the ten (10), largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard will be termed "Tentatively Identified Compounds" (TICs). More or fewer TICs may be identified based on client requirements.

TICs that cannot be completely identified to a known reference spectrum will be identified as an unknown analyte class (e.g. Unknown Hydrocarbon, Unknown Siloxane, etc.).

# 21.5.2 TIC Reporting Limits

In general Reporting limits cannot be specified because of the unknown nature of the TIC. Any reporting limit that is reported can only be evaluated as an estimate as the quantitation is based on the assumption that the TIC responds exactly as the IS responds which is most likely not the case. In general, it is not recommended to set a Reporting limit at too low of a concentration as it gives a false impression.

TICs that meet the above identification criteria (Section 21.5.1) at 10% area of the IS: The RL would be 10% of the concentration of the internal standard used for quantitation (e.g. 10% of 5.0 ug/L for an RL of 0.5 ug/L for EPA Method 524.2). In general, if the 10% area criterion is not met, the TIC result is not reported.

If a compound meets the TIC criteria, the reporting limit will reflect the ratio between the TIC and the IS.

For compounds that are not completely identified and are classified as Unknowns, the RL is set at 5x the reporting level of the poorest performer in the analysis.

# 21.6 POLICY ON GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

**21.6.1** The concentration of the BFB or DFTPP must be at or below the concentrations that are referenced in the analytical methods. Part of the purpose of the tune is to demonstrate sensitivity and analyzing solutions at higher concentrations does not support this purpose. Tune failures may be due to saturation and a lower BFB/DFTPP concentration may be warranted.

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**21.6.2** Tune evaluations usually utilize the "Autofind" function and are set up to look at the apex +/- 1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction cannot include any part of the target peak.

- 21.6.3 Other Options or if Auto Tune Fails:
- 21.6.3.1 Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. In this case, manually identify and average the apex peak +/- 1 scan and background correct as in 21.6.4 above. This is consistent with EPA 8260 and 8270.
- **21.6.3.2** Or the scan across the peak at one half peak height may be averaged and background corrected. This is consistent with Standard Methods 6200, EPA 624 and EPA 625.
- 21.6.3.3 Adjustments such as adjustments to the repeller and ion focus lenses, adjusting the EM Voltage, etc. may be made prior to tune verification as long as <u>all</u> of the subsequent injections in the 12 hour tune cycle are analyzed under the same MS tune settings and it is documented in the run sequence log and/or maintenance log that an adjustment was made. Excessive adjusting (more than 2 tries) without clear documentation is not allowed. Necessary maintenance is performed and documented in instrument log.
- 21.6.3.4 A single scan at the Apex (only) may also be used for the evaluation of the tune. For SW 846 and EPA 600 series methods, background correction is still required.
- 21.6.3.5 Cleaning the source or other maintenance may be performed and then follow steps for tune evaluation above. Note: If significant maintenance was performed, see methods 8000B or 8000C then the instrument may require recalibration prior to proceeding.
- **21.6.4** Tune evaluation printouts must include the chromatogram and spectra as well as the Tune evaluation information. In addition, the verifications must be sent directly to the printer or pdf file (no screen prints for DFTPP or BFB tunes). This ability should be built into the instrument software.
- **21.6.5** Since the limits are expressed in whole percentages, the results may be rounded to whole percentage before comparing to criteria when assessing the tune verification against the tune requirements. However, the comparison to the criteria is usually done automatically by the software and if the printout says "Fail" then there would have to be documentation of the hand calculation on the raw data and comparison to the criteria if the lab intends to still accept the tune. In most cases the analyst is better off performing an adjustment and rerunning the tune standard.
- **21.6.6** All MS tune settings must remain constant between running the tune check and all other samples. It is recommended that a separate tune method not be used, however a separate method may be used as long as the MS conditions between the methods are the same as the sample analysis method and tracked so any changes that are made to the analysis method are also made to the tune method.

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Table 21-1

Laboratory Equipment and Instrumentation

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Volatiles	Manufacturer	Wiodel Nulliber	Serial Nulliber	Service	Received
Gas Chromatograph #9	Agilent	6850 Series II	US00002015	2002	New <sup>2</sup>
Mass Spectrometer #9		5973		2002	New <sup>2</sup>
	Agilent	5975	US10440578	2002	inew
Purge & Trap Concentrator #9	O.I. Amalutical	4560	114460212	2002	No. 42
Auto Sampler #9	O.I. Analytical Varian	4500 Archon	I14460213 13520	2002	New <sup>2</sup>
Auto Sampier #9	Variari	Archon	13520	2002	new
Gas Chromatograph #39	Hewlett Packard	6890N	CN10521030	2004	New <sup>2</sup>
Mass Spectrometer #39	Hewlett Packard	5973	4540620627	2004	New <sup>2</sup>
Purge & Trap					
Concentrator #39	O.I. Analytical	4560	N126460727	2001	New <sup>2</sup>
Auto Sampler #39	O.I. Analytical	4552	14420	2004	New <sup>2</sup>
Gas Chromatograph	A ''	C050 C : "	11000001000	2002	N 2
#40	Agilent	6850 Series II	US00001682	2003	New <sup>2</sup>
Mass Spectrometer #40	Agilent	5973	US92522712	2003	New
Purge & Trap	07.4 1.7	45.00	14010460700	2002	2
Concentrator #40	O.I. Analytical	4560	M012460798	2003	New <sup>2</sup>
Auto Sampler #40	Varian	Archon	13389	2003	New <sup>2</sup>
Semi-Volatiles					
Gas Chromatograph #6					
(FID)	Hewlett Packard	5890	2623A08469	1995	1
Auto Sampler #6	CTC	A200S	146389	2001	New <sup>2</sup>
Gas Chromatograph #10 (ECD)	Agilont	6900	LIC10212004	2002	No.v <sup>2</sup>
Auto Sampler #10	Agilent Agilent	6890 7683	US10212094 US93108476	2003	New <sup>2</sup>
Auto Sampier #10	Agilent	/003	0393108476	2003	New
Gas Chromatograph					
#14 (ECD)	Agilent	6890	US10244151	2003	New <sup>2</sup>
Auto Sampler #14	Agilent	7683	CN23927539	2003	New <sup>2</sup>
Gas Chromatograph	A ''	6000	11010044150	2002	N 2
#15 (ECD)	Agilent	6890	US10244152	2003	New <sup>2</sup>
Auto Sampler #15	Agilent	7683	CN23927541	2003	New <sup>2</sup>
Gas Chromatograph					
#16 (ECD)	Agilent	6890	US10402034	2005	New <sup>2</sup>
Auto Sampler #16	Agilent	7683	CN32030890	2005	New <sup>2</sup>
Gas Chromatograph				1	1
#17 (ECD)	Hewlett Packard	5890	3336A56851		
Auto Sampler #17	Hewlett Packard	7673	3443A40622	1	1
Gas Chromatograph					
#37/Mass Spectrometer					
#37	Agilent	6890	US10243060	2003	New <sup>2</sup>
" ) 1	Ayiletit	0030	0310243000	2003	INCM

Equipment/	Manadaataaa	Madal Namehan	Ossis I November	Year Put into	Condition When
Instrument	Manufacturer	Model Number	Serial Number	Service	Received
Auto Sampler #37	Agilent	7683	CN22425747	2003	New <sup>2</sup>
Gas Chromatograph					
#38/Mass Spectrometer					
#38	Agilent	6890	US10243060	2003	New <sup>2</sup>
Auto Sampler #38	Agilent	7683	CN23927536	2003	New <sup>2</sup>
7 tato bampioi = 50	, tg	7 000	0.123327330	2005	
HPLC #1 (UV)	Hewlett Packard	HP 1100/1050	28037G00138	1	1
Autosampler#1	Hewlett Packard	HP 1050/18596M	2813G00326	1	1
HPLC #2					_
(Fluorescence)	Hewlett Packard	HP 1100	US54000680	1997	New <sup>2</sup>
Autosampler#2	Hewlett Packard	HP 1100/1313A	US54000680	1997	New <sup>2</sup>
Post Column					3
Derivatizer	Pickering	PCX 5200	1102202	2003	New <sup>2</sup>
LIDI C #2 (D): 1 A `	Hambar D. J. J.	LID 1100	DE22020127	2002	N1 2
HPLC #3 (Diode Array)	Hewlett Packard	HP 1100	DE23920127 DE23920127	2003 2003	New <sup>2</sup>
Autosampler#3	Hewlett Packard	HP 1100	DE23920127	2003	ivew
Inorganics					
pH Meter	Orion	EA 940	TZ22A	1995	1
p. r meter	<u> </u>	27.13.10		2333	
Conductivity Meter	YSI	35	Q022545	2004	New
•					
Turbidity Meter	VWR	66120-200	TUR 800 1842	2004	New
Extractions					
Solid Phase Extractor					
Controller 1	Horizon	SPC-100	02-0355	2003	New
Solid Phase Extractor	110112011	51 € 100	02 0333	2003	11011
Controller 2	Horizon	SPC-100	03-0407	2004	New
Solid Phase Extractor 1	Horizon	4790	02-0340	2003	New
Solid Phase Extractor 2	Horizon	4790	02-0341	2003	New
Solid Phase Extractor 3	Horizon	4790	03-0379	2004	New
Solid Phase Extractor 4	Horizon	4790	03-0380	2004	New
Evaporator #1	Labconco	Rapidvap	990391288C	1	1
Evaporator #2	Labconco	Rapidvap	266894	1997	New
NA (()   F	TI I	62700	1075000330333	2001	N
Muffle Furnace	Thermolyne	62700	1075990330323	2001	New
Orbital Shaker 1	Lab Line	3506	0695-0113	1997	1
Orbital Shaker 2	Lab Line	4329	0199-0153	2000	New
Orbital Shaker 2	Lab Line	7323	0133 0133	2000	INCW
Centrifuge	Fisher	225	21100412	1997	1
Misychiolos					
<b>Microbiology</b> Quebec Colony				+	
Counter	Leica Inc.	3327	000243804YX0004	2004	New
Counter	LCICA IIIC.	3321	000273004170004	2004	14644
Quanti Tray sealer	Idexx	98-10894-02	4025	2004	New
Quanti Tray sealer	Idexx	2020	141122	2004	New
C :			_ : <b>-</b>		

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Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
44.5 deg Circulating					
Water Bath	Precision	51221039	60211184	2004	New
44.5 deg Circulating Water Bath	Precision	2886	202462-195	2007	New
35 deg. Dry Coliform Incubator	VWR	1915	800902	2004	New
35 deg. Dry Coliform Incubator	VWR	1915	1102003	2005	New
58 deg. Dry Spore Incubator	Barnstead	DB104115	1.04102E+12	2004	New
Isotemp Oven (41 deg. Incubator)	Fisher Scientific	7256	40300021	1	1
Autoclave	Tuttnaur/Brinkman	2540EK	210929	2004	New
Autoclave	Sanyo	MLS-3780	3Y0521	2004	New
Vacuum Manifold (6 port)	Pall/Gelman	15403	NA	2004	New
Optical Microscope (10x100)	VWR	BB-P/TB-P	V167531	2004	New
Stereo Microscope w/Fluorescence source	VWR	HF-745	V167531	2004	New
UV Viewing Cabinet	SpectroLine	CM-10	NA	2004	New
UV Lamp	SpectroLine	EA 160	1417722	2004	New
Stirrer/Hotplate	VWR	DY19-DUAL	NA	1	1
Hotplate	Corning	PC 500	NA	2004	New
Repeater Pipette (2)	Eppendorf	Repeater Plus	NA	2004	New
Pipet-Aid Pipettor	Drummond	Pipet-Aid XP	68640	2004	New

<sup>&</sup>lt;sup>1</sup>Although equipment is operational and calibration maintained (if required), this information is not available. <sup>2</sup>The exact condition of this equipment when it was received was either New or Refurbished.

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Table 21-2 Schedule of Routine Maintenance
Preventive Maintenance Procedures For Laboratory Equipment

Instrument/ Equipment Type	Maintenance	Frequency <sup>†</sup>	
71.	Replace Gas line dryers and filters	As needed*	
	Replace Gas cylinders	As needed	
	Check or adjust column gas flow and/or detector make-up flow	As needed*	
	Replace Injection port Septa	As needed	
	Replace Injection port liners/re-silonize liners	GC, As needed; GC/MS, As needed	
	Replace injection port liner o-ring	GC, As needed; GC/MS, As needed	
Gas Chromatograph	Replace inlet seal and ring	GC, As needed, GC/MS, As needed	
Cas Omomatograph	Replace column ferrules	GC, As needed; *	
	Trim column or guard column (injector and detector end)	As needed*	
	Replace syringes on autosamplers	As needed*	
	Replace heated-zones heaters and sensors	As needed*	
	Replace inlet assembly	As needed*	
	Empty solvent rinse and solvent rinse-waste vials (on autosampler tower)	Daily or as needed	
	Replace column	As needed*	
	Bake out detector	As needed	
Electron Capture	Clean detector	As needed	
Detector (ECD) or Micro ECD	Check for negative peaks and elevated baseline. Send out for cleaning and refoiling	As needed	
	Wipe test	Semi-Annually	
Flores lowing the	Clean/replace jet	As needed*	
Flame Ionization Detector (FID)	Clean collector	As needed*	
	Check and/or adjust gas flows	As needed*	
	Clean source, replace source parts, replace filaments	As needed*	
	Clean analyzer	As needed*	
	Replace electron multiplier	As needed*	
	Clean or replace glass jet separator, replace transfer line from jet separator to MS	As needed*	
Mass Spectrometer	Check differential pump oil	After each source cleaning	
(MS)	Change differential pump oil	Annually	
	Check rough pump oil	After each source cleaning	
	Change rough pump oil	Annually	
	Refill calibration compound (PFTBA) vial	As needed	
	Ion Gauge Degassing?	As needed	
	Adjust resolution	As needed	
Purge and Trap	Refill rinse water supply/Empty rinse water waste	Weekly or as needed	

Instrument/ Equipment Type	Maintenance	Frequency <sup>†</sup>
<b>Equipment</b> CI	lean or replace 6-port valve	As needed*
	eplace Transfer lines (from Autosampler to LSC nd from LSC to GC)	As needed*
Ac	djust gas flows and pressures	As needed
Pe	erform leak check	As needed
W	Vipe Archon rods with alcohol	Monthly
Ca	alibrate Archon robotics	Monthly
Ve	erify purge flow	Monthly
Re	econdition trap	As needed
Ins	nspect probes for hardness buildup	Monthly
Re	eplace pre-column filter	As needed*
Re	efill Solvent reservoirs	Daily or as needed
Re	everse column and rinse with solvents	Daily or as needed*
Re	eplace column	As needed*
Re	eplace guard column	As needed*
Ch	hange lamps	As needed*
High Pressure Liquid Chromatography	heck/Change pump seals	As needed*
	eplace tubing	As needed
Ch	hange fuses in power supply	As needed*
CI	lean solvent reservoir filters	As needed*
	eplace ball-valve cartridges on high pressure ump	As needed*
Re	eplace DAD flow cell windows	As needed*
Ch	heck system solvent pressure	Daily
Cl	lean pan and platform	After each use
Balance	heck Level bubble	Daily
Ve	erify calibration	Daily
CI	leaning and calibration by authorized service	Annually
CI	lean gasket and chamber	As needed
Ch	heck temperature	After each use
Autoclave	alibrate temperature gauge and check pressure	Annually
Dr	rain water	As needed
Cł	heck Conductivity	Daily
M	Ionitor for contamination	Daily
Nanopure water Sy	ystem cleaning	As needed
Re	eplace cartridges	As needed
Ch	theck Conductivity, Residual Chlorine, and HPC	Monthly
Microbiology Reagent Ch	heck Metals content (Cd, Cr, Cu, Ni, Pb, Zn)	Annually
	Vater Use test	Quarterly
CI	lean the system	As needed

Instrument/ Equipment Type	Maintenance	Frequency <sup>†</sup>	
	Replace cartridges	As needed	
nH Metero	Clean or replace electrode	As needed	
pH Meters	Refill electrode electrolyte	As needed	
Turbidity Meter	Clean exterior of meter	As needed	
Turbidity Weter	Check Bulb	Daily	
Conductivity Meter	Clean probe	As needed	
Burettes and Pipets	Clean and check calibration	Quarterly*	
Thermometers	Check calibration	Annually, Quarterly for Digitals and IR Thermometer*	
	Check temperature, record on log sheet	Twice Daily at least 4 hours apart	
Incubators	Adjust temperature	As needed	
	Clean door gasket and shelves	As needed	
Ovens	Check temperature, record on log sheet	Daily	
Overis	Adjust temperature	As needed	
Defrimenatore and	Check temperature, record on log sheet	Daily	
Refrigerators and Freezers	Adjust temperature	As needed	
. 1002010	Clean and/or Defrost freezers	As needed	
Colony Counter	Clean view mirror glass and bulb	As needed	
UV Light	Clean view mirror and bulb	As needed	
OV Light	Check UV light intensity	Quarterly	
Quanti-Tray Sealer	Clean exterior	As needed	
Qualiti-Ilay Sealel	Perform sealer check	Monthly	
Water Baths	Monitor Temperature	Twice Daily at least 4 hours apart	
Waler Dallis	Clean and replace water	Monthly or as needed	

\*Date and maintenance performed are recorded in Maintenance Log of the instrument/equipment †Daily indicates each day of use or each day lab staff are present

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#### **SECTION 22**

# MEASUREMENT TRACEABILITY (NELAC 5.5.6)

# 22.1 OVERVIEW

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. The following definitions are provided by the American Association for Laboratory Accreditation (A2LA):

"Traceability is the property of a measurement result whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, each step in the chain having stated uncertainties." There are six essential elements:

- An unbroken chain of comparison
- A calculated measurement uncertainty for each step in the chain to allow for an overall uncertainty calculation
- Documentation of each step in each calibration report
- All steps in the chain are performed by individuals with evidence of technical competence and accredited by a recognized accreditation body
- Reference to International Standard (SI) units
- Recalibration at appropriate intervals to preserve traceability

Calibration is defined as "determining and documenting the deviation of the indication of a measuring instrument (or the stated value of a material measure) from the conventional 'true' value of the measurand."

Uncertainty is defined as "a parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measurand." Measurement of Uncertainty is discussed is Section 20 of this QA Manual.

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# 22.2 <u>NIST-TRACEABLE WEIGHTS AND THERMOMETERS</u>

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

The calibration report or certificate submitted to *TestAmerica Ontario*, *CA* must contain, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses when applicable, and a clearly identified record of the quantities and functional test results before and after re-calibration. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

## 22.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. (Refer to Section 9 for additional information on purchasing). The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number (from Element) and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification

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(ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to Table 9-1 in Section 9 for general storage requirements and Table 22-1 for additional storage information. Refer to the laboratory's Reagent and Standard Preparation, Control, and Documentation SOP. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

# 22.4 <u>DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS</u>

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to SOP No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained within each department. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. Refer to the laboratory's Reagent and Standard Preparation, Control, and Documentation SOP.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

- **22.4.1** All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system by the analyst, are assigned a unique identification number, and are at least peer reviewed. The following information is typically recorded in the electronic database within the LIMS.
- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date

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- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date, and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

**22.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date
- Standard ID from Element
- Special Health/Safety warnings if applicable

# **22.4.3** In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods; and 3) according to Table 22-1.

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Table 22-1
Standard Sources and Preparation

Instrument	Source	How Received	Stock Storage**	Preparation	Intermediate & Working Standard Storage	Frequency
Volatile Organics	*	Ampules/ Solutions	Freezer (-10°C)	Working standards from stock	Freezer	6 months; Monthly; Gases, biweekly
Semi- Volatile Organics	*	Ampules/ Solutions	Freezer, refrigerator, or Room Temperature	Working standards from stock	Freezer or refrigerator	6 months
Inorganics (pH buffers)	VWR; Fisher	Solution	Room Temperature	Ready to use	Room Temperature	Vendor's expiration date
Inorganics (Turbidity)	GFS Chem.	Solutions	Room Temperature	Ready to use	Room Temperature	Vendor's expiration date
Microbiology	B.D.; Gibson Labs	Swabs	Refrigerate (2 – 8 °C)	Control Culture plates from Control Organism Swabs	Refrigerate	Transfer Control Organisms to new plates weekly up to 4 weeks

<sup>\*</sup> Standards are purchased from a variety of sources which may be modified at the analyst's, Group Leader's, and/or Operation Manager's discretion. Standard sources include but are not limited to Absolute, AccuStandard, Crescent Chemicals, Restek, Ultra Scientific, and O2SI.

<sup>\*\*</sup>Stock standards are stored according to the manufacturer's recommendations and may be in the freezer, refrigerator, or at room temperature.

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#### **SECTION 23.0**

# SAMPLING (NELAC 5.5.7)

# 23.1 OVERVIEW

**TestAmerica Ontario, CA** provides sampling services. Sampling procedures are described in the following SOPs:

- Field Sampling SOP
- Drinking Water and Microbiological Sampling Supplement to the Field Sampling SOP

# 23.2 <u>SAMPLING CONTAINERS</u>

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory. Some containers for used to collect samples for General Physical tests such as Color, Odor, and Turbidity are reused when the original samples are tested and are ND for each parameter tested. This is, in effect, a certification of the cleanliness of each container by the laboratory. Sample containers with samples that show some signs of Color, Odor, or Turbidity are disposed of.

# 23.2.1 <u>Preservatives</u>

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid Reagent ACS (Certified VOA Free) or equivalent
- Methanol Purge and Trap grade
- Nitric Acid Instra-Analyzed or equivalent
- Sodium Bisulfate ACS Grade or equivalent
- Sodium Hydroxide Instra-Analyzed or equivalent
- Sulfuric Acid Instra-Analyzed or equivalent
- Sodium Thiosulfate ACS Grade or equivalent
- Sodium Sulfite ACS Grade or equivalent
- Ascorbic Acid ACS Grade or equivalent
- Monochloroacetic Acid (MCAA) ACS Grade or equivalent
- Ammonium Chloride ACS Grade or equivalent
- Zinc Acetate ACS Grade or equivalent

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# 23.2.2 <u>Preparing Container Orders</u>

When new containers arrive at the laboratory, the date of receipt is recorded on the packing list received with them for retained documentation. Periodically, containers are evaluated for cleanliness based upon their intended parameter sample analysis. Upon request, the containers are then sent to clients for use in collecting samples.

Shipping personnel insure that container stock is rotated so that "first in" is "first out." When a client requests containers, a client services representative creates a container request in LIMS; it is then stored permanently in LIMS with a unique container order number. Copies of the container request are printed for the shipping department. The shipping date, type and number of containers are maintained on file by the lab. One copy goes to the client with the containers; one copy is filed in the shipping department.

If containers are provided directly to the client from the manufacturer or from other sources, the laboratory will not be responsible for any of the above records.

## 23.3 FIELD QUALITY CONTROL (QC)

Common field quality control samples are defined in the following paragraphs. The frequency of field quality control samples should be specified in the site specific Quality Assurance Project Plan (QAPP) or by the client. TestAmerica provides trip blanks for VOC analysis with the sample containers for all volatile organic analyses. Blanks generated in the field will be analyzed along with the field samples (exception soil samples where the blank is aqueous).

- 23.3.1 Equipment Blank / Rinsate Blank The equipment blank, sometimes referred to as a rinsate blank, is a sample of the water used to decontaminate sampling equipment. The source water should be as free of target analytes as possible. An aliquot of this water is poured over or through the sample collection device after decontamination, collected in a sample container, preserved with appropriate reagents, and returned to the laboratory. This serves as a check on sampling device cleanliness, and will also be affected by the site and sample handling conditions evaluated by the other types of blanks. The sampling time for the equipment blank should begin when the equipment is rinsed and the water is collected.
- **23.3.2** Field Blank The field blank is water that is as free of target analytes as possible and from the same source as the equipment blank. The water is poured into a sampling container at the sampling site, preserved with the appropriate reagents, and returned to the laboratory. This serves as a check on reagent and environmental contamination. The sampling time for the field blank should be when the blank is prepared in the field.
- 23.3.3 Trip Blank The trip blank pertains to volatile analysis only. This serves as a check on sample contamination originating from sample transport, sample container contamination, shipping and storage, or from certain site conditions. Trip blanks are often referred to as travel blanks. They are prepared using pre-cleaned sample containers. They are filled with organic-free water (the source of the organic free water is the same source of water used to prepare volatile standards, method blanks, LCS and sample dilutions), sealed and taken into the field with the empty containers which will be used for sampling. The recommended frequency is one trip blank per cooler (in duplicate or triplicate), per volatiles method. Unless otherwise specified, the sampling time for the trip blank is the time of receipt at the laboratory (When the "Trip" ends).

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**23.3.4** <u>Field Duplicates</u> - Field duplicates are replicate samples collected from the same sampling point or location during a field collection event. This control sample is used to demonstrate the ability of both the sampling and analytical process to generate data of acceptable precision.

## 23.4 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g. 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g. 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis.

- 23.4.1 <u>Semi-Volatile</u> Holding times for sample preparation for semi-volatile organics are measured from the sampling date (and time where applicable) until the day (and time where applicable) solvent contacts the sample. If a sample is to be extracted on the day of expiration, the actual time of extraction must be recorded on the sample preparation worksheet. Holding times for analysis are measured from the date (and time where applicable) of initiation of extraction to the time of injection into the gas chromatograph.
- <u>Volatiles</u> Holding times for volatile organics are measured from the date (and time where applicable) of sampling to the date and time of injection into the gas chromatograph. The time that the sample is prepared and loaded onto the autosampler is considered the injection time as long as the sample is analyzed within the same workshift. The preparation time documented on the bench sheet for the analysis is the date and time that the analyst began preparing the samples for the batch. If a sample is so near expiration that the exact preparation time is needed to document the integrity of the sample; the analyst must document this in the bench sheet.
- **23.4.3** <u>Inorganics</u> For inorganic and metals analysis, the preparation/digestion/distillation must be started within the maximum holding time as measured from the sampling date (and time where applicable).
- **23.4.4** Microbiology Holding times for microbiology samples are measured from the sampling date and time to the date and time that the samples are placed in the incubator or waterbath for incubation. For Colilert 18 samples, it is the date and time that the samples are placed in the incubator, not when they are placed in the water bath prior to incubation.

## 23.5 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times (refer to Tables 23-1 to 23-7) or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

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# 23.6 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis. Refer to the laboratory's Subsampling SOP.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

- **23.6.1** For water samples, before taking each aliquot for analysis, invert the sample container end-over-end three times and immediately pour off the aliquot. Especially when suspended solids are present, adequate mixing of the sample is extremely important.
- **23.6.2** All microbiology samples should be homogenized by shaking vigorously 25 times prior to removing any aliquots or adding media.
- **23.6.3** For solid samples, when volatile organics are not requested, if the solid can be mixed, stir before removing the aliquot. Mix more than is needed for the analysis to be performed (e.g. if 30 g are needed, mix 50-100 g, if 1 g is needed, mix 20 g, etc...).
- If the solid cannot be easily mixed: After thoroughly mixing the sample within the sample container or, for non-organic methods, the sample can be transferred to a wip bag (or other suitable plastic bag) for manual mixing, a sub-sample from various quadrants and depths of the sample are taken to acquire the required sample weight.
- For soil samples, avoid debris in the subsample aliquot as much as possible (e.g. gravel, sticks, roots and grass); note this information in the sample comments on the bench sheet.
- If the solid is extremely heterogeneous, and the client has given no instructions, utilize the following technique: separate the like materials into groups on a clean surface and take portions of masses from each group, proportional to their contribution to the original sample, to make a composite. Record in detail exactly how the composite was created. For very unusual samples, consult with the QA department, Group Leader, or Operations Manager.
- **23.6.4** For multiphasic samples, the client should instruct the laboratory as to the intent of the testing and how to handle the sample. If the entire sample is to be accounted for, and the phases do not mix easily with inversion/stirring, such that a representative aliquot can be taken, the analyst should record the percent by volume of each phase. The analysis must be conducted on each phase separately; the final results are combined mathematically, weighting the individual phase results by volume. One exception to this procedure is the situation addressed in the TCLP and SPLP methods for wastes containing free liquids. However, if the leachate and final filtrate are not miscible, it is necessary to combine mathematically the concentrations of the two (or more) solutions by volume.

Tables 23-1 to 23-7 detail holding times, preservation and container requirements, and sample volumes for SDWA and NPDES methods. The sample volumes are intended to be a minimal amount to perform the method, the containers that are used may be of larger size. *Please note*:

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the holding times are program specific and different programs may have different holding times for equivalent methods (e.g., there are difference in Holding times for many Organic analytes between SDWA and NPDES. RCRA methods may also be different.)

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Table 23-1 Holding Times, Preservation and Container Requirements: Drinking Water (SDWA)

PARAMETER	CONTAINER	PRES Temp. <sup>2</sup>	SERVATION <sup>1,2</sup> 3 Chemical	HOLDING TIME <sup>3</sup>	SAMPLE VOLUME
Asbestos	Plastic/Glass	4°C	None	48 hours <sup>5</sup>	1 L
Coliforms (Total and Fecal)	Plastic/Glass <sup>20</sup>	10°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	30 hours <sup>21</sup>	120 mL
Heterotrophic Plate Count	Plastic/Glass <sup>20</sup>	10°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	8 hours (24 hours <sup>22</sup> )	120 mL
Color	Glass <sup>8</sup>	4ºC	None	48 hours	500 mL
Odor	Glass <sup>8</sup>	4ºC	None	24 hours <sup>25</sup>	500 mL
Turbidity	Glass <sup>8</sup>	4°C	None	48 hours	500 mL
Cyanide	Plastic/Glass	4°C	NaOH to pH >12 <sup>9</sup> Ascorbic acid <sup>9</sup> or Sodium arsenite <sup>9</sup>	14 days	500 mL
Fluoride	Plastic/Glass	None	None	None	250 mL
Mercury	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	28 days	250 mL
Metals <sup>4</sup>	Plastic/Glass	None	HNO <sub>3</sub> to pH<2 <sup>4</sup>	6 months	250 mL
Lead & Copper Rule	Plastic	None	HNO₃ to pH<2	6 months	1 L "first draw sample"
Nitrate	Plastic/Glass	4°C	None	48 hours <sup>6</sup>	250 mL
Nitrate-Nitrite	Plastic/Glass	None	H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	250 mL
Nitrite	Plastic/Glass	4°C	None	48 hours	250 mL
TOC	Glass	4°C	H <sub>3</sub> PO <sub>4</sub>	28 days	250 mL
Perchlorate	Plastic	4°C	None	28 days	500 mL
THMs Only	Glass <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days	3 X 40 mL
Volatile Organic Compounds	Glass <sup>8</sup>	4°C	Ascorbic acid <sup>9</sup> ; HCl to pH <2	14 days	3 X 40 mL
EDB, DBCP (EPA 504.1)	Glass <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days	3 X 40 mL
Organochlorine Pesticides/PCBs (EPA 505)	Glass <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days <sup>10,11</sup>	3 X 40 mL

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PARAMETER	CONTAINER	PRES Temp. <sup>2</sup>	SERVATION <sup>1,2</sup> 3 Chemical	HOLDING TIME <sup>3</sup>	SAMPLE VOLUME
Nitrogen and Phos. Pesticides (EPA 507)	Glass-Amber <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days <sup>12</sup>	1 L
Total PCBs (EPA 508A)	Glass-Amber <sup>8</sup>	4°C	None	14 days <sup>13</sup>	1 L
Pesticides and PCBs (EPA 508.1) <sup>14</sup>	Glass-Amber <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>9</sup> ; HCl to pH <2	14 days <sup>13</sup>	1 L
Chlorinated Acids (EPA 515.3)	Glass-Amber <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days <sup>12</sup>	3 x 60 mL
Chlorinated Acids (EPA 515.4)	Glass-Amber <sup>8</sup>	4°C	Na <sub>2</sub> SO <sub>3</sub>	14 days <sup>12</sup>	3 x 60 mL or 1 x 250 mL
Semivolatiles (EPA 525.2)	Glass-Amber <sup>8</sup>	4°C	HCl to pH <2 Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>9</sup>	14 days <sup>13</sup>	1 L
N-Methylcarbamoyloxamines and N-Methcarbamates (EPA 531.1)	Glass <sup>8</sup>	4ºC	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , Monochloroacetic Acid buffer to pH<3	28 days	3 X 60 mL
Glyphosate (EPA 547)	Glass <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	14 days	3 X 40 mL
Endothall (EPA 548)	Glass-Amber <sup>8</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	7 days <sup>15</sup>	250 mL
Diquat/Paraquat (EPA 549.2)	Amber PVC	4°C	$H_2SO_4$ to PH <2 $Na_2S_2O_3^9$	7 days <sup>16</sup>	1 L
Chlorinated Disinfection Byproducts, Chlorinated Solvents, and Halogenated Pesticides/Herbicides (EPA 551)	Glass <sup>8</sup>	4ºC	Phosphate Buffer and Ammonium Chloride <sup>19</sup>	14 days <sup>17</sup>	3 X 60 mL
Haloacetic Acids (EPA 552.2)	Glass-Amber <sup>8</sup>	4°C	Ammonium Chloride	28 days <sup>18</sup>	3 X 60 mL or 250 mL
Radiologicals (Gross Alpha, Gross Beta, Radium 226 & 228, Strontium)	Plastic	None	HNO <sub>3</sub> to pH<2 <sup>26</sup>	365 days	1 L
Uranium (200.8)	Plastic	4°C	HNO <sub>3</sub>	180 days	500 mL
Tritium	Glass <sup>8</sup>	None	None	365 days	250 mL
Dioxin (2,3,7,8-TCDD)	Glass-Amber <sup>8</sup>	4°C	None	365 days	2 x 1 L

# **Key to Table**

<sup>1.</sup> Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler

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#### **Key to Table**

makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at  $4^{\circ}$ C until compositing and sample splitting is completed.

- 2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
- 3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
- 4. All metals except Hg.
- 5. Instructions for containers, preservation procedures and holding times as specified in Method 100.2 must be adhered to for all compliance analysis including those conducted with Method 100.1.
- 6. If the sample is chlorinated, the holding time for an un-acidified sample kept at 4°C is extended to 14 days.
- 7. Nitrate-Nitrite refers to a measurement of total nitrite.
- 8. With Teflon lined septum.
- 9. If chlorinated add reagent prior to acidification, if not, the dechlorinating reagent may be omitted (for Cyanide, add before NaOH).
- 10. Heptachlor has a 7 day hold time
- 11. 14 days until extraction. 24 hours after extraction.
- 12. 14 days until extraction. 28 days after extraction.
- 13. 14 days until extraction. 30 days after extraction.
- 14. For cyanazine, cool to 4°C only.
- 15. 7 days until derivatization. 1 day after derivatization.
- 16. 7 days until extraction. 21 days after extraction.
- 17. 14 days until extraction. 14 days after extraction.
- 18. 28 days until extraction. 48 hours after extraction.
- 19. Sodium Sulfite may be used as a dechlorinating agent in some instances. Verify with laboratory prior to sampling.
- 20. Sterilized. Plastic must be Polypropylene.
- 21. 40 CFR part 141.74 regulations to avoid filtration or disinfection state 8 hours (DW compliance testing). Most facilities are using either disinfection or filtration so the 8 would not apply in most cases.
- 22. 40 CFR part 141.74 regulations for Disinfection By-Product rule state 8 hours (DW compliance testing) where SM 9215 allows up to 24 hours if sample is stored between > 0 and  $\le 4^{\circ}$  C
- 23. For samples with a temperature requirement of  $4^{\circ}$ C, a sample temperature of just above the water freezing temperature to  $\leq 6^{\circ}$ C is acceptable.
- Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.
- 25. A six hour hold time is recommended, but there is no regulatory limit. The laboratory has set the limit at 24 hours.
- 26. The samples may be collected without preservation and then preserved by the lab within 5 days of sampling. The samples must be preserved and held in the original container for 16 hours before analysis or subsampling.

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Table 23-2
Holding Times, Preservation and Container Requirements: NPDES – Bacteria, Protozoa, Toxicity Tests

PARAMETER	CONTAINER <sup>1</sup>	PRES Temp.	SERVATION <sup>2,3</sup> Chemical	HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
Total, Fecal, and E.coli Coliforms	Plastic/Glass	10°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>6</sup>	6 hours	100 mL
Fecal Streptococci	Plastic/Glass	10°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>6</sup>	6 hours	100 mL
Enterococci	Plastic/Glass	10°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>6</sup>	6 hours	100 mL
Cryptosporidium	LPDE Plastic	0-8°C	None	96 Hours	500 mL
Giardia	LPDE Plastic	0-8°C	None	96 Hours	500 mL
Toxicity – Acute/Chronic	Plastic/Glass	≤ 6°C <sup>5</sup>	None	36 Hours	2 L

#### **Key to Table**

- 1. Plastic should be Polypropylene or other sterilizable plastic.
- 2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
- 3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
- 4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
- 5. Samples must not be frozen. Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present, when samples arrive, it is necessary to measure the temperature of the samples and confirm that the  $\leq$  6°C temperature has not been exceeded.
- 6. Should only be used in the presence of residual chlorine.

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Table 23-3
Holding Times, Preservation and Container Requirements: NPDES - Inorganic

PARAMETER	CONTAINER <sup>1</sup>	PRE Temp <sup>14</sup>	SERVATION <sup>2,3</sup> . Chemical	HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
Acidity	Plastic/Glass	≤ 6°C	None	14 days	100 mL
Alkalinity	Plastic/Glass	≤ 6°C	None	14 days	100 mL
Ammonia	Plastic/Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	400 mL
BOD 5 Day	Plastic/Glass	≤ 6°C	None	48 hours	1000 mL
Boron	Plastic <sup>5</sup>	None	HNO <sub>3</sub> to pH<2	6 months	200 mL
Bromide	Plastic/Glass	None	None	28 days	100 mL
CBOD 5 Day	Plastic/Glass	≤ 6°C	None	48 hours	1000 mL
COD	Plastic/Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	100 mL
Chloride	Plastic/Glass	None	None	28 days	50 mL
Chlorine, Residual	Plastic/Glass	None	None	15 min. <sup>6</sup>	200 mL
Color	Plastic/Glass	≤ 6°C	None	48 hours	50 mL
Cyanide –Total <sup>16, 17</sup>	Plastic/Glass	≤ 6°C	NaOH to pH >12,	14 days	100 mL
Cyanide – Total	1 183110/01833	<u> </u>	0.6 g ascorbic Acid <sup>7</sup>		
Cyanide – Amenable <sup>16, 17</sup>	Plastic/Glass	≤ 6°C	NaOH to pH >12,	14 days	100 mL
Amenable 10, 17	. 146116/ 6/1466		0.6 g ascorbic Acid <sup>7</sup>	dayo	
Fluoride	Plastic	None	None	28 days	300 mL
Hardness	Plastic/Glass	None	HNO <sub>3</sub> to pH<2 <sup>8</sup>	6 months	100 mL
Hexavalent Chromium	Plastic/Glass	≤ 6°C	Ammonium sulfate buffer pH = 9.3 - 9.7	28 days / 24 hrs <sup>15</sup>	200 mL
Hydrogen Ion (pH)	Plastic/Glass	None	None	15 min. <sup>6</sup>	200 mL
Kjeldahl and organic Nitrogen	Plastic/Glass	≤ 6°C	H₂SO₄ to pH <2	28 days	500 mL
Mercury <sup>11</sup>	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	28 days	200 mL
Metals <sup>9,10</sup>	Plastic/Glass	None	HNO <sub>3</sub> to pH<2 <sup>18</sup>	6 months	200 mL
Nitrate	Plastic/Glass	≤ 6°C	None	48 hours	100 mL
Nitrate-Nitrite	Plastic/Glass	≤ 6°C	H₂SO₄ to pH <2	28 days	100 mL
Nitrite	Plastic/Glass	≤ 6°C	None	48 hours	100 mL
Oil and Grease	Glass	≤ 6°C	H₂SO₄ or HCl to pH <2	28 days	1 L

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PARAMETER	CONTAINER <sup>1</sup>	PRE Temp <sup>14</sup> .	SERVATION <sup>2,3</sup> . Chemical	HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
Organic Carbon (TOC)	Plastic/Glass	<u>&lt;</u> 6°C	H <sub>2</sub> SO <sub>4</sub> or HCl to pH <2 <sup>12</sup>	28 days	250 mL
Orthophosphate	Plastic/Glass	≤ 6°C	Filter within 15 min.	48 hours	250 mL
Oxygen, Dissolved Probe	Glass <sup>13</sup>	None	None	15 min. <sup>6</sup>	200 mL
Oxygen, Winkler	Glass <sup>13</sup>	None	Fix on site and store in dark.	8 hours	300 mL
Phenols	Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days	500 mL
Phosphorus, Elemental	Glass	≤ 6°C	None	48 hours	250 mL
Phosphorus, Total	Plastic/Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days	250 mL
Residue, Total	Plastic/Glass	≤ 6°C	None	7 days	1 L
Residue, Filterable	Plastic/Glass	<u>&lt;</u> 6°C	None	7 days	1 L
Residue, Non- Filterable	Plastic/Glass	<u>&lt;</u> 6°C	None	7 days	1 L
Residue, Settleable	Plastic/Glass	≤ 6°C	None	48 hours	1 L
Residue, Volatile	Plastic/Glass	≤ 6°C	None	7 days	1 L
Silica	Plastic <sup>5</sup>	≤ 6°C	None	28 days	250 mL
Specific Conductance	Plastic/Glass	≤ 6°C	None	28 days	250 mL
Sulfate	Plastic/Glass	≤ 6°C	None	28 days	250 mL
Sulfide	Plastic/Glass	≤ 6°C	Zinc acetate plus NaOH to pH>9	7 days	500 mL
Sulfite	Plastic/Glass	None	None	15 min. <sup>6</sup>	200 mL
Surfactants	Plastic/Glass	≤ 6°C	None	48 hours	1 L
Temperature	Plastic/Glass	None	None	N/A	100 mL
Turbidity	Plastic/Glass	≤ 6°C	None	48 hours	1 L

# **Key to Table**

- 1. Plastic should be Polyethylene.
- 2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at < 6°C until compositing and sample splitting is completed.

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#### **Key to Table**

3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

- 4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
- 5. May also be collected in quartz or PFTE Plastic.
- 6. For compliance testing, the analysis must be performed in the field at the time of analysis. If transported to the laboratory for analysis, the analysis will be performed as soon as practical and reported qualified.
- 7. Should only be used in the presence of residual chlorine. (Alternatively, sodium arsenite may be used)
- 8.  $H_2SO_4$  to a pH <2 is also acceptable.
- 9. Except Mercury and Hexavalent Chromium.
- 10. For dissolved metals, samples must be filtered on site before adding HNO<sub>3</sub> preservative (or before shipping to laboratory).
- 11. Samples collected for determination of trace level mercury (100 ng/L) using EPA 1631 must be collected in tightly capped fluoropolymer or glad bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipping, samples should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. Samples that been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.
- 12. Phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) may also be used.
- 13. Should have glass lid or top.
- 14. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of "≤ °C" is used in place of the "4 °C" and "<4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).
- 15. Holding time is 24 hours if pH adjustment is not performed.
- In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH. If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered (with sulfide treatment by laboratory) and qualify the results in the final report.
- 17 It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.
- Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.

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Table 23-4 Holding Times, Preservation and Container Requirements: NPDES - Organic

PARAMETER	CONTAINER	PRES Temp. <sup>15</sup>	SERVATION <sup>1,2</sup> Chemical	HOLDING TIME <sup>3</sup>	SAMPLE VOLUME
Purgeable Halocarbons	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	14 days	40 mL
Purgeable Aromatic Hydrocarbons	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , HCl to pH<2	14 days <sup>6</sup>	40 mL
Acrolein and Acrylonitrile	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , adjust pH to 4-5 <sup>7</sup>	14 days	40 mL
Phenols <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days <sup>8</sup>	1 L
Benzidines <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days <sup>8, 11</sup>	1 L
Phthalate esters <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	None	7 days <sup>8</sup>	1 L
Nitrosamines <sup>9,12</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5,13</sup>	7 days <sup>8</sup>	1 L
PCBs <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	None	1 year <sup>8</sup>	1 L
Nitroaromatics and Isophorone <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5,13</sup>	7 days <sup>8</sup>	1 L
Polynuclear Aromatic Hydrocarbons <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5,13</sup>	7 days <sup>8</sup>	1 L
Haloethers <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days <sup>8</sup>	1 L
Chlorinated Hydrocarbons <sup>9</sup>	Glass <sup>4</sup>	≤ 6°C	None	7 days <sup>8</sup>	1 L
CDD/CDFs <sup>9</sup> – Aqueous: Field/Lab Preservation	Glass	<u>&lt;</u> 6°C	pH <9, 0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	1 year	1 L
CDD/CDFs <sup>9</sup> – Solids/Mixed Phase/ - Field Preservation	Glass	≤ 6°C	None	7 days	1 L
CDD/CDFs <sup>9 -</sup> Tissue – Field Preservation	Glass	≤ 6°C	None	24 hours	
CDD/CDFs <sup>9</sup> – Solids/Mixed Phase/Tissue - Lab Preservation	Glass	< -10°C	None	1 year	1 L
Pesticides <sup>9</sup>	Glass	≤ 6°C	pH 5-9 <sup>14</sup>	7 days <sup>8</sup>	1 L

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#### Key to Table

Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at ≤ 6°C until compositing and sample splitting is completed.

- 2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCI) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
- 3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
- 4. With Teflon lined septum.
- 5. Should only be used in the presence of residual chlorine. Ascorbic may be used instead.
- 6. Samples receiving no pH adjustments must be analyzed within 7 days. If 2-chlorovinylethylether is a target analyte, the sample should not be acidified.
- 7. The pH adjustment is not required if acrolein is not being measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.
- 8. 7 days until extraction, 40 days after extraction. (PCB only 1 year after extraction)
- 9. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more categories, the sample may be preserved by cooling to ≤ 6°C reducing residual chlorine with 0.0008 % sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9. Samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine) and footnotes 10 and 11(re the analysis of Benzidine).
- 10. If 1,2-diphenylhydrazine is likely to be present, adjust pH to of the sample to 4.0  $\pm$  0.2 to prevent rearrangement to benzidine.
- 11. Extracts may be stored up to 30 days before analysis if storage temperature is < 0°C.
- 12. For the analysis of diphenylnitrosamine, add 0.008 % Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- 13. Store in dark.
- 14. The pH adjustment may be performed upon receipt in the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.0008 % Na<sub>2</sub>S<sub>2</sub>O<sub>3.</sub>
- 15. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of "≤ °C" is used in place of the "4 °C" and "<4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

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Table 23-5
Holding Times, Preservation and Container Requirements: NPDES - Radiological

		PRESERVATION <sup>1,2</sup>		HOLDING	SAMPLE
PARAMETER	CONTAINER	Temp.	Chemical	TIME <sup>3</sup>	VOLUME
Alpha, Beta, Radium	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	6 months	1 L

### **Key to Table**

- Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
- 2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater).
- 3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

Table 23-6
Holding Times, Preservation and Container Requirements: RCRA - Aqueous

PARAMETER	CONTAINER <sup>1</sup>	PRES Temp. <sup>12</sup>	SERVATION <sup>2,3</sup> Chemical	HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
Carbonyl Compounds	Glass	4°C	None	3 <sup>13</sup>	1 L
Chloride	Plastic/Glass	4°C	None	28 days	100 mL
Cyanide -Total	Plastic/Glass	4°C	NaOH to pH >12 <sup>5</sup>	14 days	250 mL
Cyanide -Amenable	Plastic/Glass	4°C	NaOH to pH >12 <sup>5</sup>	14 days	250 mL
Hydrogen Ion (pH)	Plastic/Glass	4°C	None	24 hours <sup>11</sup>	100 mL
Nitrate	Plastic/Glass	4°C	None	48 hours	28 days
Oil and Grease	Glass	4°C	HCI	28 days	1 L
Organic carbon (TOC)	Plastic/Glass	4°C	pH to <2 <sup>6</sup> Store in dark	28 days	100 mL
Sulfate	Plastic/Glass	4°C	None	28 days	400 mL
Sulfide	Plastic/Glass	4°C	Add Zn Acetate	7 days	400 mL
Chromium VI	Plastic/Glass	4°C	None	24 hours	250 mL
Mercury	Plastic/Glass	None	HNO₃ to pH<2	28 days	250 mL

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PARAMETER	CONTAINER <sup>1</sup>	PRES Temp. <sup>12</sup>	SERVATION <sup>2,3</sup> Chemical	HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
Other Metals	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	6 months	250 mL
Acrolein and Acrylonitrile	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> Adjust pH to 4-5 <sup>14</sup>	14 days	1 L
Benzidines	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L
Chlorinated Hydrocarbons	Glass <sup>10</sup>	4ºC	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L
Dioxins and Furans	Glass <sup>10</sup>	4ºC	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	30 days	1 L
Haloethers	Glass <sup>10</sup>	4ºC	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L
Nitroaromatics and cyclic ketones	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> , store in dark	7 days <sup>8</sup>	1 L
Nitrosoamines	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> , store in dark	7 days <sup>8</sup>	1 L
Organochlorine Pesticides	Glass <sup>10</sup>	4ºC	None	7 days <sup>8</sup>	1 L
Organophosphorus Pesticides	Glass <sup>10</sup>	4°C	Adjust pH <sup>9</sup>	7 days <sup>8</sup>	1 L
PCBs	Glass <sup>10</sup>	4°C	None	7 days <sup>8</sup>	1 L
Phenols	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L
Phthalate Esters	Glass <sup>10</sup>	4°C	None	7 days <sup>8</sup>	1 L
Polynuclear Aromatic Hydrocarbons	Glass <sup>10</sup>	4ºC	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> , store in dark	7 days <sup>8</sup>	1 L
Purgeable Hydrocarbons	Glass <sup>10</sup>	4ºC	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> Adjust pH <2 <sup>2</sup>	14 days	40 mL
Purgeable Halocarbons	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	14 days	40 mL
Total Organic Halides (TOX)	Glass <sup>10</sup>	4°C	Adjust pH to <2 with H <sub>2</sub> SO <sub>4</sub>	28 days	1 L
Radiological Tests (Alpha, Beta, Radium)	Plastic/Glass	None	HNO₃ to pH<2	6 months	250 mL

## Key to Table

- 1. Plastic should be Polyethylene.
- 2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
- 3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The

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#### **Key to Table**

person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid ( $H_2SO_4$ ) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

- 4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
- 5. If oxidizing agents are present, add 5 mL 0.1 N NaAsO<sub>2</sub> or 0.06 g of ascorbic acid per Liter. See Cyanide SOP for additional information about other interferences.
- 6. Adjust pH to <2 with H<sub>2</sub>SO<sub>4</sub>, HCl, or solid NaHSO<sub>4</sub>. Free Chlorine must be removed prior to adjustment.
- 7. Free Chlorine must be removed by the appropriate addition of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.
- 8. 7 days until extraction. 40 days after extraction.
- 9. Adjust pH to 5-8 using NaOH or H<sub>2</sub>SO<sub>4</sub>.
- 10. With Teflon lined septum.
- 11. Holding Time is listed as "As Soon as Possible" in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hrs and in soil is 7 d. There are no mandated regulatory requirements.
- 12. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to ≤ 6°C is acceptable.
- 13. 3 days for derivatization & extraction. 7 days after extraction.
- 14. Per guidance from EPA MICE, if samples are received without pH adjustment, the holding time is 7 days.

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Table 23-7
Holding Times, Preservation and Container Requirements: RCRA – Non-Aqueous

			SERVATION	HOLDING	SAMPLE
PARAMETER	CONTAINER 1	Temp. <sup>7</sup>	Chemical	TIME <sup>2</sup>	WEIGHT
Carbonyl Compounds	Glass	4ºC	None	14 <sup>8</sup>	100 g
Chloride	Glass	4°C	None	28 days	50 g
Cyanide -Total	Glass	4°C	None	14 days	50 g
Cyanide -Amenable	Glass	4°C	None	14 days	50 g
Hydrogen Ion (pH)	Glass	4°C	None	7 days <sup>6</sup>	50 g
Nitrate	Glass	4°C	None	N/A	50 g
Oil and Grease	Glass	4°C	None	28 days	50 g
Sulfide	Glass	4°C	Add Zn Acetate, zero headspace	7 days	50 g
Chromium VI	Glass	4°C	None	24 hours	50 g
Mercury	Plastic/Glass	None	None	28 days	50 g
Other Metals	Plastic/Glass	None	None	6 months	50 g
Acrolein and Acrylonitrile	Glass <sup>4</sup>	4ºC	None	14 days	50 g
Benzidines	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Chlorinated Hydrocarbons	Glass <sup>4</sup>	4ºC	None	14 days <sup>3</sup>	50 g
Dioxins and Furans	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Haloethers	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Nitroaromatics and cyclic ketones	Glass <sup>4</sup>	4ºC	None	14 days <sup>3</sup>	50 g
Nitrosoamines	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Organochlorine Pesticides	Glass⁴	4°C	None	14 days <sup>3</sup>	50 g
Organophosphorus Pesticides	Glass <sup>4</sup>	4ºC	None	14 days <sup>3</sup>	50 g
PCBs	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Phenols	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Phthalate Esters	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g

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		PRESERVATION		HOLDING	SAMPLE
PARAMETER	CONTAINER <sup>1</sup>	Temp. <sup>7</sup>	Chemical	TIME <sup>2</sup>	WEIGHT
Polynuclear Aromatic Hydrocarbons	Glass <sup>4</sup>	4ºC	None	14 days <sup>3</sup>	50 g
Purgeable Hydrocarbons	Glass <sup>4</sup>	4ºC	None	14 days <sup>5</sup>	50 g
Purgeable Halocarbons	Glass⁴	4°C	None	14 days⁵	50 g
Total Organic Halides (TOX)	Glass <sup>4</sup>	4ºC	None	28 days	50 g

#### **Key to Table**

- 1. Plastic should be Polyethylene.
- 2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
- 3. 14 days until extraction. 40 days after extraction.
- 4. With Teflon Lined Septum
- 5. See Volatile SOP for more detailed preservation requirements.
- 6. Holding Time is listed as "As Soon as Possible" in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
- 7. For samples with a temperature requirement of  $4^{\circ}$ C, a sample temperature of just above the water freezing temperature to  $\leq 6^{\circ}$ C is acceptable.
- 8. 14 days until leaching, derivatization & extraction within 3 days of the completion of leaching, 7 days after extraction.

Table 23-8 Holding Times, Preservation and Container Requirements: Air Samples

		PRESERVATION		HOLDING	SAMPLE
PARAMETER	CONTAINER 1	Temp.	Chemical	TIME <sup>2</sup>	WEIGHT
Volatile Organics	Summa Canister	None	None	30 days	6L or 1L
Volatile Organics	Tedlar Bag	None	None	72 hrs <sup>3,4</sup>	1 L

## **Key to Table**

- 1. Plastic should be Polyethylene.
- 2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
- 3. Holding Time is based on SW 846 Method 0040 "SAMPLING OF PRINCIPAL ORGANIC HAZARDOUS CONSTITUENTS FROM COMBUSTION SOURCES USING TEDLAR® BAGS". Some states specifically enforce this holding time (e.g. Florida, New Jersey) and others have not specified this information in their regulatory requirements.
- 4. The holding time is 72 hours unless the laboratory has a documented validation study that indicates a longer HT is acceptable for the analytes of interest.

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#### **SECTION 24**

# HANDLING OF SAMPLES (NELAC 5.5.8)

Sample management procedures at *TestAmerica Ontario, CA* ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

# 24.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and can be initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 24-1.

# 24.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 24-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

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The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

**Note:** Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier or shipping company is retained with the COC; it lists all receipts each date.

## 24.1.2 <u>Legal / Evidentiary Chain-of-Custody</u>

If samples are identified for legal/evidentiary purposes on the COC, login will initiate an Internal COC (ICOC) (Figure 24-3) for laboratory use by analysts and a sample disposal record (Figure 24-4). Refer to the laboratory's Legal Custody Procedures SOP. This ICOC is attached to the door of the refrigerator where the samples are stored in a binder or on a clip board. The analysts sign the ICOC when samples are removed from the refrigerator for analysis. If the entire sample is to be used in the analysis, the analyst documents this on the ICOC. If there is sample remaining after analysis, it is placed back in the refrigerator, and the Analyst signs it back in. When the remaining sample is disposed of, the Sample Disposal Technician documents this on the ICOC. After the sample is used up or disposed of, the ICOC is removed from the refrigerator and filed with the original COC.

# 24.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

# 24.2.1 <u>Laboratory Receipt</u>

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Notice of Discrepancy Form (see Figure 24-6) and brought to the attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

## **24.2.1.1** Inspection of samples include a check for:

 Complete documentation to include sample identification, location, date and time of collection, collector's name, preservation type, sample type and any additional comments concerning the samples.

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- Complete sample labels to include unique identification in indelible ink.
- Use of appropriate sample containers (see Section 23)
- Adherence to holding times as specified in the test method and/or summarized in Section 23.
- Adequate sample volume for required analyses (see Section 23).
- Damage or signs of contamination to sample container. Volatile vials are also inspected for headspace
- 24.2.1.2 Check and record the temperature of the samples and/or temperature blanks that require thermal preservation. This is done by scanning the label of a sample or temperature blank the IR thermometer while the sample or temperature blank is still in the cooler
  - Samples shall generally be deemed acceptable if arrival temperature is just above freezing and less than or equal to 6.0° C. There are some exceptions to this range, including Microbiology samples that must be above freezing and below 10° C, and metals and radiological samples that do not require temperature preservation. Samples that are hand-delivered on the same day of collection may not have had time to reach the required temperatures. If there is evidence, however, that the chilling process has begun, such as the arrival on ice, the samples shall be considered acceptable. This will be documented on the COC.
  - If the samples were shipped in ice and solid ice is still present and in direct contact
    with samples, report the samples as "received on ice." Direct contact means
    samples must be surrounded by ice cubes or crushed ice. Ice present in a plastic
    bottle or other container does not constitute direct contact. Samples shipped with
    only "blue ice" may not be reported as "received on ice".
- 24.2.1.3 If samples are received without a COC, TestAmerica will provide a generic COC form to be completed by the client when the samples are brought to the laboratory. The client is always provided with a copy of the completed COC form for their records.
- 24.2.1.4 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in Sample Control Hold Refrigerator until the samples can be logged in and labeled. Samples for volatiles analysis are stored in the Volatiles Refrigerator if they cannot be logged in and labeled immediately.
- 24.2.1.5 Verify sample preservation as specified in the test method. For semi-volatile samples, check for correct pH and/or dechlorination as specified in the test method. The results are documented according to the laboratory's Sample Control SOP. In the case of volatiles, this check is performed at the time of analysis and documented on the sample prep sheet.
- **24.2.1.6** If analyses with short holding times are requested, the dates and times are inspected to ensure that holding times have not already expired.

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24.2.1.7 Samples received after normal working hours are left in their coolers and placed either in or in front of the Sample Control Hold Refrigerator. The person receiving the samples must record the date and time received, the presence or absence of ice and custody seals, the temperature of samples, presence and type of packing material, and initials. TestAmerica couriers may drop off sample coolers in Sample Control overnight when there is no one available to receive the samples. They will place the coolers in or in front of the Sample Control Hold Refrigerator, sign the COC, record the date and time that they are being dropped off, and include a "To Hold Refrigerator" or "To Hold Fridge" notation. The Sample Control Technician(s) will receive the samples when they arrive in the morning, sign the COC and record the actual date and time that they received the samples that morning.

- **24.2.1.8** Any deviations from the checks described in Section 24.2.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance criteria (Section 24.3) are not met, the laboratory shall either:
  - Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples (The project manager may be able to make decisions on samples with prior knowledge from the client, but documentation of acceptable scenarios must be provided and acknowledge by the client, and records of these decisions must be documented), or
  - Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

## 24.2.2 Sample Log-in

All samples that are received by the laboratory are logged into the LIMS to allow the laboratory to track and evaluate sample progress. Each group of samples that are logged in together (typically one project from a given client/sampling event) is assigned a unique job number. Within each job, each sampling point (or sample) receives a unique number. Sample numbers are generated sequentially over time, and are not re-assigned. A sample may be composed of more than one bottle since different preservatives may be required to perform all analyses requested. Even if multiple containers are received for a single sample, each container is uniquely identified with an alphabetic letter added to the sample number. The LIMS generates sample labels that are attached to each bottle for a given sample.

Each job/set of samples is logged into LIMS with a minimum of the following information:

- Client Name, Project Name, Address, Phone, Fax, Report to information, invoice to information (most of this information is "default information" that is stored in the LIMS).
- · Date and time sampled;
- Date and time received;
- Job and/or project description, sample description;
- Sample matrix, special sample remarks;
- Reporting requirements (i.e., QC level, report format, invoicing format);
- Turn-around-time requirements;

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 Parameters (methods and reporting limits or MDLs are default information for a given parameter)

## 24.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (Figure 24-5) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method;
- sample holding times must be adhered to;
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

## 24.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. Metals and Radiological samples that do not require temperature preservation may be stored on shelves in Sample Control. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve sample containers allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples are returned to the designated refrigerator. Empty sample containers are destroyed in the appropriate broken glass disposal containers. All unused portions of samples are kept in the refrigerators for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to a dry, room temperature sample archive area where they are stored for an additional four weeks before they are disposed of. This eight week holding period allows samples and their containers to be checked if a discrepancy or question arises. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be

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performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

#### 24.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only. For any sample that is known to be hazardous at the time of receipt or, if after completion of analysis the result exceeds the acceptable regulatory levels, a Hazardous Sample Notice must be completed by the analyst. This form may be completed by Sample Control, Project Managers, or analysts and must be attached to the report. The sample itself is clearly marked with a red stamp, stamped on the sample label reading "HAZARDOUS" or "FOREIGN SOIL" and placed in a colored and/or marked bag to easily identify the sample. The date, log number, lab sample number, and the result or brief description of the hazard are all written on the Hazardous & Foreign Soil Sample Notice. A copy of the form must be included with the original COC and Work Order and the original must be given to the Sample Disposal Technician. Analysts will notify Sample Control of any sample determined to be hazardous after completion of analysis by completing a Hazardous Sample Notice. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

#### 24.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). For sample shipments which include water/solid volatile organic analyses, a trip blank is enclosed when required by method specifications or state or regulatory programs. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

#### 24.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis,

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the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures as described in the Sample Archiving and Disposal section of the Sample Control SOP. All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, and return to the client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record (Figure 24-4) should be completed.

Figure 24-1

Example: Chain of Custody (COC)

	THE LEADER IN ENVIRONMENTAL TESTING	ESTING										101	East	t C00	lley D	)rive,	Suite	A,	1014 East Cooley Drive, Suite A, Colton, CA 92324	CA 9	2324		09) 37	(909) 370-4667	FAX (909) 370-1046	370-1046
				R	X	DRINKING WATER CHAIN OF CUSTODY FORM	×	ΙE	SC	¥	9	딩	STC	Z	임	ΣŽ								Page_	je of	
Client Name:											Р.	J./Pr	P.O./Project Name	Nan	ne:											
Address:											Pr	oject	Project Manager:	ager	, .											
City:		State:	.e:		Zip:	2:					ပိ	ildm	Compliance Sample: Yes	Sar	nple	. Υ	S	No.	0		# HOE	#				
Tel:	Fax:				Dat	Data to state's database? Yes (PWSID required)	state	's da	taba	se?	Yes	(PW	SID	requ	nired			No	-		PWs	PWS ID#				
Sampler(s) Name & Signature:											Sa	mple	s ac	idifie	∍d af	ter d	echl	orins	Samples acidified after dechlorination?		Yes		No	!		
Sample I.D.	(side T x Table)	Date Sampled	Number of Containers	S.4≤8 □.gəЯnU □.gəЯ səlisbbV	Trihalomethanes Only 524.2	Semivolatiles Reg. 🗆 UnReg. 🗆 525.2	EDB   DB Cb   1Cb 204.1	Pesticides and PCBs 505	Chlorinated Acids 515.4	Carbamates 531.1 Glyphosate 547	1.843 Ilentrobria	Diquat / Paraquat 549.2	Coliform, Total 🔲 Fecal 🔲 E.coli	Heterotrophic Plate Count (HPC)	Metals (Specify)	General Minerals (see fee schedule)	General Physical (see feeschedule)	Inorganic Chemicals (see fee schedule)	□sbioAcetic Acids□					Aluminum Arimony Arimony Besenic Benium Bevinim Becon Cadmium Cadmium Cadmium Cadmium Clackium Clackiu	Maganesium     Maganesium       Manganesium	Magnesium Manganese Mercuri Nickel Potassium Selenium Selenium Salver Sodium Vandium Vandium dher
	+	+					+	+	+	+	+	$\bot$	$\perp$					T					+			
	H	-				3		H	+	+												П	t			
							$\Box$	$\vdash$	$\vdash$	$\vdash$	$\vdash$	$\sqcup$	Ш	Ш								П	H			
	÷								+	+		$\perp$														
	H	H				╚	$\sqcap$	$\vdash$	$\vdash$	$\vdash$	$\vdash$	$\sqcup \downarrow$	П										$\forall$			
	+	+						+	+	+	+	$\dashv \downarrow$	$\Box$										$\dashv \dagger$			
Matrix Types	Reling	Relinquished by						$\dashv$	Date/Time	ije:	ğ	Received	by:	╛				1					_ime:	Turnarour	Turnaround Time*: (check one)	neck one)
SW - Surface Water RW - Raw Water (Source)	Reling	Relinquished by:	l.,					۵	Date/Time	::	ů.	Received by	by:									Date/Time	Lime:	72 Hours 24 Hours		48 hours
GW - Groundwater RW - Recreational Water	Reling	Relinquished by						Δ	Date/Time:	ne:	ű.	seived	Received in Lab by	b by:								Date/Time:	Lime:	*Surcharg	*Surcharges may be applied for remaining hold time <48 hours.	pplied for 8 hours.
TW - Treated Water (Point of Entry)	Remarks:	rks:																						Sample Integrity:		Temp:

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Figure 24-2

**Example: Custody Seal** 

CUSTODY SEAL	
Date	
Signature	

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Figure 24-3

**Example: Internal Chain of Custody (COC)** 

					K OR PL271					
Client: Client Code: Project Manage	r:				Project Nan Project Nur Printed:			r		
				Internal S Refrigera		Custody				
Sample	ln	Out	In	Out	In	Out	In	Out	Archived	Disposed
F1.2715-01G										
FL2715-01H					4			***		
F1.2715-011										
PL2715-01L						2.1 mil	- 4P.	(A)		
11.2715-01M										
II-L2715-01P				7 - <u>-</u>	7 5	1		48		
HL2715-01Q										
FL2715-01R				- Se-		ic we	n. vý		13 (13)-14 1 <b>2</b> (1 1) 1 1 <sub>2</sub>	-
11.2715-015										
IF:L2715-01T				4 -		E.		11.5		

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Figure 24-4

**Example: Sample Disposal Record** 

	М	onth/Year:			_	
		Matrix:				
SAMPLE I	NUMBERS ENDING	DATE REMOVED	INITIALS	DATE Disposed	INITIALS	COMMENTS
			3			

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### Figure 24-5

Sample Acceptance Policy (page 1)

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded and must be legible. noted on the laboratory report defining the nature and substance of the variation. The project manager will be notified if any sample is received in damaged Drinking Water Sample Acceptance Policy / Sampling Instructions 014 E. Cooley Dr., Suite A, Colton, CA 92324 (909) 370-4667 FAX (909) 370-1046 condition. Del Mar Analytical will request that a sample be resubmitted for analysis. Client name, address, phone number and fax number (if available) HE LEADER IN ENVIRONMENTAL TESTING estAmerico

Project name/number and/or P.O. number

Public Water System ID (if applicable)

Indicate whether lab data are for State compliance purposes and/or State's EDT requirements

Date, time and location of sampling

The collectors name and signature

Confirmation of acid preservation in the field for methods 524.2, 525.2 and 508.1

The matrix description

The total number of containers for each sample ID

Analysis requested

Requested turnaround time (TAT)

Any special instructions

The date and time that each person received or relinquished the sample(s), including their signed name.

enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (working Samples must be received in the laboratory with Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested. Insufficient 2) Proper sample containers with adequate volume for the analysis and necessary UC, are required to anomaly, samples to meet the method QC requirements will result in notation in the analytical reports to reflect the anomaly. Del Mar Analytical will make every effort to analyze samples within the regulatory holding time. Sample Holding Times

days) remaining on the holding time for us to ensure analysis.

Analyses that are "field" analyses (e.g. pH, DO, residual chlorine) will be analyzed within 24 hours from receipt of the samples in the laboratory. Field analyses samples received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless Pack samples in Ice rather than "Blue" ice packs. Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags. Fill extra cooler space with bubble wrap.

Page 1 of 3

## Sample Acceptance Policy (page 2)

23) The presence or absence of residual chicenes and the represerved. 3 ) The presence or absence of residual chicenes and the representation of the presence or absence of residual chicenes and the representation of the presence or absence of residual chicenes and the representation of the presence or absence of residual chicenes and return them to Del Mar Analytical. 4 ) Fill the additional FL containers falled "C") that are provided to meet the Outlity Control requirements for the additional FL containers and return them to Del Mar Analytical.  5 ) Veur gloses whenever handling acids, Recap the acid containers tightly affect they're used and return them to Del Mar Analytical.  6 ) Do not open naused containers and return them to Del Mar Analytical.  Farameter  7 ) Verification of the sequence of the supplied In CL (3 chops—U.28 m.) to pil < 2. Keep unopened Trip Blank with Sample without any headspace. Carefully and Chlorimated Sources.  8 ) Vener Gloses States and Trip Blank with the samples at 4 °C or less.  1.1.3. TCP (534.2 SIM) SIM	Drinking Water 500 Series Sam  1) The following are general sa drinking waters. Be sure to	eral sampling instructions f erre to consult your Regular	The following are general sampling instructions for collection and maintenance of sample integrity prior to chemical testing for various parameters in drinking waters. Be sure to consult your Regulator and/or Project Plan for any other specific sampling instructions.
e additional L-L containers (labeled cate and/or matrix spikes). Duplicat gloves whenever handling acids. Re sal. t open unused containers and return t open unused containers and return Methodology  S(524.2) SSIM) SSIM SS(524)		hat are pre-preserve ce of residual chlorin	<ul> <li>a.</li> <li>b. should be verified in the field prior to sample collection.</li> </ul>
gloves whenever handling acids. Resal.  t open unused containers and return  Methodology  S(524.2)  SIM)  EPA 524.2  SIM)  1 Sources  EPA 524.2  SIM)  1 Sources  EPA 524.2  EPA 524.2  SIM  1 Sources  EPA 524.2  EPA 524.1  EPA 525.2  EPA 525.2  EPA 525.2  EPA 531.1  EPA 547.1  EPA 547.1  EPA 549.2		L containers (labeled	"QC") that are provided to meet the Quality Control requirements for the analytical methods the are required for ICMB requirements
### Annual containers and return topen unused containers and return Methodology  ### Sizeron		er handling acids. R	ites are required for exerting equirements. ecap the acid containers tightly after they're used and return them to Del Mar Analytical for proper
### Appen unused containers and return    Methodology		)	
Methodology		containers and retur	n them to Del Mar Analytical.
ss(524.2)	Parameter	Methodology	Sam pling Instructions
ss (524) CA DHS 524.2 SIM) 1 Sources thanes EPA 524.2 EPA 524.2 EPA 524.2 EPA 524.2 EPA 524.1 EPA 524.2 EPA 524.2 EPA 524.1 EPA 524.1 EPA 524.1 EPA 531.1 EPA 525.2 Sources EPA 525.2 EPA 525.2 EPA 525.2 EPA 525.2 EPA 525.2 EPA 525.2 EPA 526.1 EPA 547.1 EPA 547.1 EPA 547.1	Volatile Organics(524.2) 1,2,3 TCP (524.2 SIM) Chlorinated sources	EPA 524.2 EPA 524.2 SIM	Fill 3 40 mL-VOA vials (preserved w/ascorbic acid) with sample without any headspace. Carefully and quickly add the supplied HCL(5 drops=0.25 mL) to pH <2. Keep unopened Trip Blank with samples. Store all samples at 4 $^{\circ}$ C or less.
thanes EPA 524.2  EPA 504.1  EPA 504.1  EPA 504.1  Sticides/PCBs EPA 505  EPA 515.4  ganies EPA 525.2  Sources EPA 525.2  EPA 525.2  EPA 527.1  EPA 547.1  EPA 547.1  EPA 549.2	Volatile Organics (524) 1,2,3, TCP (524.2 SIM) Non-Chlorinated Sources	CA DHS 524.2 SIM	Fill 3 40 mL-VOA with HCL. Keep unopened Trip Blank with samples. Store all samples at 4 $^{\circ}\mathrm{C}$ or less.
sticides/PCBs EPA 504.1 ds EPA 515.4 ganics EPA 525.2 ganics EPA 525.2 Sources EPA 525.2 EPA 527.1 EPA 547.1 EPA 549.2	Total Trihalomethanes	EPA 524.2	Fill 3 40 mL-VOA vials (preserved w/sodium thiosulfate) with sample without any headspace. <b>Keep unopened Trip Blank with samples.</b> Store all samples at 4 °C or less.
sticides/PCBs EPA 505  ds EPA 515.4  ganies EPA 525.2  Sources EPA 525.2  EPA 525.2  EPA 527.1  EPA 547.1  EPA 547.1  EPA 547.1	EDB/DBCP	EPA 504.1	Fill 3 40 mL-VOA vials (preserved w/sodium thiosulfate) with sample without any headspace. <b>Keep unopened Trip Blank with samples.</b> Store all samples at 4 °C or less.
ds EPA 515.4 ganies EPA 525.2 ganies EPA 525.2 Sources EPA 521.1 EPA 531.1 EPA 547.1 EPA 548.1 EPA 549.2	Organohalide Pesticides/PCBs	EPA 505	Fill 3 40 mL-VOA vials (preserved w/sodium thiosulfate) with sample. Store all samples at 4 °C or less.
ganies EPA 525.2  ganies EPA 525.2  Sources EPA 531.1  EPA 547.1  EPA 548.1  EPA 549.2	Chlorinated Acids	EPA 515.4	Fill 1-250 mL amber glass (preserved w/sodium sulfite) with sample. Store all samples at 4 °C or less.
Sources EPA 525.2  Sources EPA 531.1  EPA 547.1  EPA 547.1  EPA 549.2	Semivolatile Organics	EPA 525.2	Fill 2 1-Liter glass-amber containers (preserved w/sodium sulfite) with sample. Carefully and quickly add the supplied HCL (3 mL) to pH <2. Store all samples at 4 $^{\circ}$ C or less.
EPA 531.1 EPA 547.1 EPA 548.1 EPA 549.2	Semivolatile Organics Nonchlorinated Sources	EPA 525.2	Fill 2 1-Liter glass-amber containers with HCl. Store all samples at 4 °C or less.
EPA 547.1 EPA 548.1 EPA 549.2	Carbamates	EPA 531.1	Fill 3 60 mL-VOA vials (preserved w/sodium thiosulfate & monochloroacetic acid) with sample. Store all samples at 4 $^{\circ}$ C or less.
EPA 548.1 EPA 549.2	Glyphosate	EPA 547.1	Fill 3 amber 60 mL-VOA vials (preserved w/sodium thiosulfate) with sample. Store all samples at $4^{\circ}\text{C}$ or less.
EPA 549.2	Endothall	EPA 548.1	Fill 1-Liter glass-amber container (preserved w/sodium thiosulfate) with sample. Seal the container and shake vigorously for 1 minute. Store all samples at 4°C or less.
	Diquat/Paraquat	EPA 549.2	Fill 1-Liter <b>PVC-amber container</b> (preserved w/sodium thiosulfate & sulfuric acid) with sample. Store all samples at $4  ^{\circ}$ C or less.

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## **Sample Acceptance Policy (page 3)**

Parameter	Methodology	Sam pling Instructions
Haloacetic acids	EPA 552.2	Fill 3 60 mL-amber-VOA vials (preserved w/ammonium chloride) with sample. Seal the vials and agitate by hand for 1 minute. Store all samples at 4 °C or less.
Bacteriology (Coliform & HPC) General Physical:	EPA 9215/21/23 SM2120B.	Fill sterile 125 mL bottle (preserved w/sodium thiosulfate) with sample. Store all samples at 4 °C or less. Fill 500 mL clear olass bottle (unpreserved) with sample. Store all samples at 4 °C or less.
(Color, Odor, Turbidity)	SM2150B, EPA180.1	THE SOUTH CICAL BIASS COLDS (HIPPOSTIVE) WITH SAITIPIC. STOLE ALL SAITIPICS AT 1 COLDSS.
General Mineral:	EPA 150.1, 300.0,	Fill 1-Liter clear poly bottle (unpreserved) with sample. [for non-metals]
(Ca, Mg, Na, K, Fe, Cu, Mn, Zn, Hardness, Alkalinity, SOA, Cl.	200.7 SM4500F, SM2320B,	Fill 500 mL poly bottle (preserved w/nitric acid) with sample. [for metals]
NO3, F, pH, EC, TDS, MBAS)	SM2340B, SM2510B,	Store all samples at 4 °C or less.
Others that can be included: NO2, CLO4	SM2540C, SM5540C	
Inorganic Chemical Group: Al,	EPA 200.8, 300.0,	This group can be run from the "General Mineral" bottles plus "Cyanide" bottle [total of 3 bottles]
Sb, As, Ba, Be, Cd, Cr, Cu, CN, F, Pb, Hg, Ni, NO3, NO2, Se, Tl	SM4500-CN-C,E	
Cyanide	SM4500-CN-C,E	Fill 500 mL poly bottle (preserved w/sodium hydroxide) with sample. If sample is from a chlorinated source, it must first be de-chlorinated with ascorbic acid. Store all samples at 4 °C or less.
Lead & Copper Bule	FPA 200 8	Fill 1-1 iter poly hoffle (un-preserved) "first-draw" sample. Samples will be acidified upon receipt at the
read & cupper rails	EFA 200.0	The Flate poly boths (un-preserved) instrumy sample, samples will be actualled upon receipt at the laboratory. Acidification must be performed within 14 days of sampling.
Metals	EPA 200.8, 200.7	Fill 500 mL poly bottle (preserved w/nitric acid) with sample. Cooling is not required
Total Organic Carbon	SM5310B	Fill 3.40 mL-VOA vials (preserved w/hydrochloric acid) with sample without any headspace. Store all samples at 4 °C or less.
Perchlorate	EPA 314.0	Fill 500 mL poly bottle (unpreserved) with sample. Store all samples at 4 °C or less.
Radiochemistry - Gross Alpha	EPA 900.0	Fill 500 mL poly bottle (preserved w/nitric acid) with sample. Cooling is not required.
Radiochemistry - Gross Beta	EPA 900.0	Can be run from above bottle
Radiochemistry – Uranium	EPA 200.8	Fill 500 mL poly bottle (preserved w/nitric acid) with sample. Cooling is not required.
Radiochemistry – Uranium	ASTM D5174	Fill 500 mL poly bottle (preserved w/nitric acid) with sample. Cooling is not required.
Radiochemistry – Radium 226	EPA 903.1	Fill 1-Liter poly bottle (preserved w/nitric acid) with sample. Cooling is not required.
Radiochemistry – Radium 228	EPA 904.0	Fill 2 I-Liter poly bottle (preserved w/nitric acid) with sample. Cooling is not required.
Radiochemistry – Strontium 90	EPA 905.0	Fill 1-Liter poly bottle (preserved w/nitric acid) with sample. Cooling is not required.
Radiochemistry - Tritium	EPA 906.0	Fill 250 mL glass bottle (unpreserved) with sample. Cooling is not required.
Radiochemistry – Radon	ASTM D5072	Fill 2 40 mL-VOA vials (un-preserved) with sample without any headspace. Cooling is not required.
Asbestos	EPA 100.2	Fill 1-Liter poly bottle (un-preserved) with sample. Cooling is not required.
Dioxin (2,3,7,8-TCDD)	EPA 1613B	Fill 2 1-Liter glass-amber containers (un-preserved) with sample. Store all samples at 4 °C or less.
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Figure 24-6

**Example: Notice of Discrepancy Form** 

Test 1	merica al testing corporation	17461 Derlan Ave., Suite 190, Indree, CA 326 1914 E. Cookey Dr., Suite A., Colton, C 9830 South 51st Street, Suite B-120, Phosnic, A	A 92324 Ph (909) 370-466	7 Pax (909) 370-1046
		N OF DISCREPA F CUSTODY FOR		
CLIENT:	Choose a client from the li	ist below	DATE:	11/21/07
PROJECT NAME/NO:		v	ORK ORDER:	None Assigned
CLIENT CONTACT:			PHONE: #:	
PROJECT MANAGER:				
RUSH TAT?:	YES X NO	SHORT HOLDING TIME?:	X YES	□ NO
Clarification of Anal	ysis	Project Not Set Up		
Turnaround Status		Improper Preserva		
Sample Received Br		Analysis Requeste		
8015B for Gas or Di	ese: ole(s) listed on Chain-of-Custody F		Added - PM to Cor s to Add Analysis	ıfırm
	le(s) not listed on Chain-of-Custod		-	in Blank Received
	volume to perform analysis	volatile rilaryses	requested - 140 11	p Dank Received
	e Expired upon receipt			
No Project Name/No	umber on Chain-of-Custody Form			
No Purchase Order 1	Number			
Sample description(s	s) on Chain-of-Custody Form does	not match description(s) on sample lab	el(s)	
Other				
Further Explanation:				
Resolution:				
Client Approval by:		Date/Time:	PM I	nitials:
				Page 1 of 1

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#### SECTION 25.0

## ASSURING THE QUALITY OF TEST RESULTS (NELAC 5.5.9)

#### 25.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 21, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

#### 25.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include solvent extraction, solid phase extraction, filtration, and pH adjustment. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

#### 25.3 **NEGATIVE CONTROLS**

- **25.3.1 Method Blanks** are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
- 25.3.1.1 The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.
- **25.3.1.2** The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
- **25.3.1.3** The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
- **25.3.1.4** Evaluation criteria and corrective action for method blanks is defined in the specific standard operating procedure for each analysis. Generally, corrective action is taken if the concentration of a target analyte in the blank is at or above the reporting limit as established by the method or regulation:
  - The source of contamination is investigated
  - Measures are taken to minimize or eliminate the source of the contamination

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- Affected samples are reprocessed or the results are qualified on the final report.
- **25.3.2** <u>Calibration Blanks</u> are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
- **25.3.3 Instrument Blanks** are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
- **25.3.4 Trip Blanks** are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. A trip blank is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples. Trip Blanks are also sometimes referred to as Travel Blanks.
- **25.3.5** Field Blanks are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
- **25.3.6 Equipment Blanks** are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
- **25.3.7 Holding Blanks**, also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory (refer to section 24.4).
- **25.3.8** Field blanks, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. When known, blanks should not be selected for matrix QC, as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB".

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#### 25.3.9 Negative Controls for Microbiological Methods

Microbiological Methods utilize a variety of negative controls throughout the process to ensure that false positive results are not obtained. These controls are critical to the validity of the microbiological analyses. Some of these negative controls are:

- **25.3.9.1** Sterility checks of media are analyzed for each lot of pre-prepared media, ready-to-use media and for each batch of medium prepared by the laboratory.
- **25.3.9.2** Sterility checks on sample containers are performed on at least one container per lot of purchased, pre-sterilized containers. Container sterility checks are performed using non-selective growth media.
- **25.3.9.3** Sterility checks are performed on each batch of dilution water prepared by the laboratory and on each batch of pre-prepared dilution water. All checks are performed using non-selective growth media.
- 25.3.9.4 Negative culture controls demonstrate that a media does not support the growth of non-target organisms and ensures that there is not an atypical positive reaction from the target organisms. Prior to the first use of the media, each lot of pre-prepared selective media or batch of laboratory prepared selective media is analyzed with at least one known negative culture control as appropriate to the method.

#### 25.4 POSITIVE CONTROLS

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MSD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

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#### Method Performance Control - Laboratory Control Sample (LCS)

**25.4.1.1** The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

- 25.4.1.2 The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS.
- **25.4.1.3** Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).
- 25.4.1.4 As stated in the opening of this section, the LCS goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.). For some methods, the Calibration Verifications (CVs) are processed through all steps of the sample preparation process as well. In these cases, the LCS and CV may be the same sample but they must meet both sets of acceptance criteria.
- **25.4.1.5** The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.
- 25.4.1.6 If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.
- **25.4.1.6.1** For methods that have 1-10 target analytes, spike all components.
- **25.4.1.6.2** For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- **25.4.1.6.3** For methods with more than 20 target analytes, spike at least 16 components.

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**25.4.1.6.4** Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

- **25.4.1.6.5** Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project specific basis.
- **25.4.1.7** Accuracy Calculation: Percent Recovery (%R) Calculation (applies to LCS, CCV, Surrogates, and Matrix Spikes.

$$\% R = \frac{AV}{TV} \times 100$$

Where: AV = Analyzed Value TV = True Value

#### 25.4.2 Positive Controls for Microbiological Methods

- **25.4.2.1** Each lot of pre-prepared media (including chromofluorogenic reagent) and each batch of laboratory prepared media is tested with a pure culture of known positive reaction.
- **25.4.2.2** In addition, every analytical batch also contains a pure culture of known positive reaction.
- **25.4.2.3** A pure culture of known negative reaction is also tested with each analytical batch to ensure specificity of the procedure.

#### 25.5 SAMPLE MATRIX CONTROLS

#### 25.5.1 Matrix Spikes (MS)

- **25.5.1.1** The Matrix spike is used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used.
- 25.5.1.2 An MS is essentially a sample fortified with a known amount of the test analyte(s). At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.
- 25.5.1.3 If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number of the listed components (see LCS analytes 25.4.1.6 above) may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit-specified analytes and other client

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requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

**25.5.1.4** The percent recovery calculation for matrix spikes is essentially the same as the calculation shown in 25.4.1.7 except that:

$$AV = Sp - Sa$$

Where: Sp = Spike result Sa = Sample result

#### 25.5.2 Surrogate Spikes

- **25.5.2.1** Surrogate Spikes are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
- 25.5.2.2 Surrogate compounds are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method (also refer to Section 25.5). Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.

#### 25.5.3 **Duplicates**

- 25.5.3.1 For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure. Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.
- **25.5.3.2 Precision Calculation** (Relative Percent Difference RPD)

$$RPD = \frac{\mid S - D \mid}{\underbrace{\left(S + D\right)}} \times 100$$

Where: S=Sample Concentration

D=Duplicate Concentration

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#### 25.5.4 Internal Standards

25.5.4.1 In most organic analyses, internal standards are spiked into all environmental and quality control samples (including the initial calibration standards). An internal standard is also used with some metals analyses. It is added to sample extracts after the extraction (post-prep). The acceptance criteria in most methods are 50% to 200% of the responses in the mid-point of the corresponding calibration curve. Consult the method-specific SOPs for details on the internal standard compounds, calculations and acceptance criteria.

25.5.4.2 When the internal standard recoveries fall outside these limits, if there are not obvious chromatographic interferences, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets internal standard recovery criteria, the second run is reported (or both are reported if requested by the client).

#### 25.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

**25.6.1** Each individual analyte in the LCS, MS, or Surrogate Spike are evaluated against the control limits as published in the test method. Where there are no established acceptance criteria, the laboratory calculates control limits with the use of control charts or, in some cases, utilizes client project specific or regulatory mandated control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

**Note:** For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

- **25.6.2** Once control limits have been established, they are verified, reviewed, and updated if necessary on a semi-annual basis unless the method requires more frequent updating (e.g. EPA SW846 8000 series methods). Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.
- **25.6.2.1** The lab should consider the effects of the spiking concentration control limits, and to avoid censoring of data. The acceptance criteria for recovery and precision are often a function of the spike concentration used. Therefore, caution must be used when pooling data to generate control limits.
- 25.6.2.2 Not only should the results all be from a similar matrix, but the spiking levels should also be approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results should all be generated using the same set of extraction, cleanup and analysis techniques. For example, results from solid samples extracted by ultrasonic extraction are not mixed with those extracted by Soxhlet.
- 25.6.2.3 The laboratory should try and avoid discarding data that do not meet a preconceived notion of acceptable performance. This results in a censored data set, which, when used to develop acceptance criteria, will lead to unrealistically narrow criteria. For a 99% confidence interval, 1 out of every 100 observations likely will still fall outside

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the limits. For methods with long analyte lists this may mean occasional failures every batch or two. While professional judgment is important in evaluating data to be used to develop acceptance criteria, specific results are not discarded simply because they do not meet one's expectations. However, data points shall be discarded if they were the result of human or mechanical error or sample concentration exceeded spike level by > 4x. (Element users: Right clicking on the control chart and selecting View Data from the drop down menu allows the QA Staff to view a table of all the charted points with any qualifiers. This assists the QA Staff in determining if any points should be discarded prior to limit generation.)

- **25.6.3** Laboratory generated % Recovery acceptance (control) limits are generally established by taking  $\pm$  3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred). For labs using Promium Element: The system defaults to collecting the previous 3 months data. This time frame should be shortened if there are more than 200 points since the system slows down tremendously. The time frame should be extended if there fewer than 20-30 points.
- **25.6.3.1** Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).
- **25.6.3.2** In-house limits cannot be any wider than those mandated in a regulated analytical method.
- 25.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable.
- **25.6.3.4** The maximum acceptable recovery limit will be 150%.
- **25.6.3.5** The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.
- **25.6.3.6** If either the high or low end of the control limit changes by  $\leq$  5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.
- **25.6.4** The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. Refer to the laboratory's Control Charts and Statistical Process Control SOP.
- 25.6.4.1 The QA department generates a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for analyses performed at *TestAmerica Ontario*, *CA*, including whether they are fixed limits or whether Control Chart limits are required. This summary is used to generate Control Chart Update form for each method. This form includes all analytes reported by the laboratory for that method, and the Surrogate, LCS, LCS RPD, MS, and MS RPD limits. The parameters that have fixed limits are marked as such. The other parameters have the old limits, a space for the new limits, and a space for the date range used for the Control Chart, and the number of data points used in the Control

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Chart. These last two pieces of information can be used to regenerate the Control Charts if necessary. This form is filled out each time Control Chart limits are generated for a method and kept on file. The limits are updated in all test codes for the method in Element when the Control Chart is generated. The analysts are notified when new Control Limits have been generated and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory.

- **25.6.5** A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:
- **25.6.5.1** The analyte results are below the reporting limit and the LCS is above the upper control limit.
- **25.6.5.2** If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.
- **25.6.5.3** Or, for NELAC work, there are an allowable number of Marginal Exceedances (ME):
  - <11 analytes 0 marginal exceedances are allowed.
  - 11 30 Analytes 1 marginal exceedance is allowed
  - 31-50 Analytes 2 marginal exceedances are allowed
  - 51-70 Analytes 3 marginal exceedances are allowed
  - 71-90 Analytes 4 marginal exceedances are allowed
  - > 90 Analytes 5 marginal exceedances are allowed
  - **25.6.5.3.1** Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).
  - **25.6.5.3.2** Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.
  - **25.6.5.3.3** Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits and it may not be acceptable for regulatory compliance.
- **25.6.6** If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the individual method SOPs and in Section 13.

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**25.6.7** If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

#### 25.7 METHOD DETECTION LIMITS (MDLs)

MDLs, calculated as described in Section 20.7, are updated or verified annually, or more often if required by the method.

#### 25.8 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

- **25.8.1** The laboratory has written procedures to assure the accuracy of the test method including calibration (see Section 21), use of certified reference materials (see Section 22) and use of PT samples (see Section 16).
- **25.8.2** A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 20.
- **25.8.3** Use of formulae to reduce data is discussed in the method standard operating procedures and in Section 21.
- **25.8.4** Selection of appropriate reagents and standards is included in Section 9 and 22.
- **25.8.5** A discussion on selectivity of the test is included in Section 5.
- **25.8.6** Constant and consistent test conditions are discussed in Section 19.
- **25.8.7** The laboratories sample acceptance policy is included in Section 24.
- **25.8.8** A listing of the type of test result correlations that are looked at during report review (e.g. Total Chromium should be greater or equal to Hexavalent Chromium) is included in Section 20.13.4.5.

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#### SECTION 26.0

## REPORTING RESULTS (NELAC 5.5.10)

#### 26.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is a conflict between the client requested formats and accreditation requirements or data usability information, accreditation requirements and data usability information will take precedence over client requests. A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 20.

#### 26.2 TEST REPORTS

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

- **26.2.1** A report title (e.g. Laboratory Report) with a "sample result" column header.
- **26.2.2** Each report page is printed on company letterhead, which includes the laboratory name, address and telephone number.
- **26.2.3** A unique identification of the report (e.g. work order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:** Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

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- **26.2.4** A copy of the chain of custody (COC).
- Any COCs involved with Subcontracting are included.
- In most cases, the applicable COC is not paginated but is an integral part of the report. If the COC is not a paginated portion of the report then there will be a statement on the front of the report to effect of "The Chain of Custody, X page(s), is included and is an integral part of this report.". The number of pages of the CoC (X) is entered into Element so that it is correct for each report.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g. Sampling information).
- **26.2.5** The name and address of client and a project name/number, if applicable.
- **26.2.6** Client project manager or other contact
- **26.2.7** Description and unambiguous identification of the tested sample(s) including the client identification code.
- **26.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.
- **26.2.9** Date reported or date of revision, if applicable.
- **26.2.10** Method of analysis including method code (EPA, Standard Methods, etc).
- **26.2.11** Practical quantitation limits or reporting limit. In some cases, the regulatory required reporting limit (e.g. California Detection Limits Reportable (DLRs) are listed as the reporting limit. In these cases, the regulatory limit may or may not be the lowest calibration level for the analyte, but it may not be below the lowest calibration level.
- **26.2.12** Method detection limits (if requested)
- **26.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).
- **26.2.14** Sample results.
- **26.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.
- **26.2.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 26.2.4 Item 3 regarding additional addenda). This information is documented on the COC at the time of receipt, and is included in the report on the COC itself. This information is transferred into Element upon login of the samples and is included in the Case Narrative of the report as well.

- **26.2.17** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.
- **26.2.18** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory.
- **26.2.19** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.
- 26.2.20 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not. *Examples: "At the time of analysis the laboratory was in compliance with the current NELAC standards and held accreditation for all analyses performed unless noted by a qualifier. The lab's accreditation number is \_\_\_\_\_\_" OR "The report meets all applicable NELAC standards and shall not be reproduced except in full, without the written approval of the laboratory."*
- **26.2.21** The laboratory may include a cover letter if required or requested.
- **26.2.22** Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.
- **26.2.23** When Soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.
- **26.2.24** Appropriate laboratory certification number for the state of origin of the sample, if applicable.
- **26.2.25** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, draft, or summary), and that a complete report will follow once all of the work has been completed.
- **26.2.26** Any out of network subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All in-network subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

#### 26.3 REPORTING LEVEL OR REPORT TYPE

**TestAmerica Ontario, CA** offers several levels of reports and data packages.

- Normal reports include all of the features described in Section 26.2 above.
- Report summaries exclude QC data and list sample results compared to regulatory levels.
- Data Packages include the normal reports and COCs, as well as varying levels of supporting documentation including instrument calibration summaries, instrument calibration data, and sample raw data.

Specific information on data packages can be found in the laboratory's Data Package SOP.

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#### 26.3.1 <u>Electronic Data Deliverables (EDDs)</u>

EDDs are routinely offered as part of TestAmerica's services. *TestAmerica Ontario, CA* offers a variety of EDD formats including State regulatory specific, client specific, summaries, and Excel formats.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

#### 26.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report. Refer to Appendix 7 for a list of the laboratory's standard footnotes and qualifiers.

- **26.4.1** Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.
- **26.4.2** Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.
- **26.4.3** Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.
- **26.4.4** Opinions and Interpretations The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

**Note:** Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This

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necessitates a limited scope of interpretation, and this work is performed by the Group Leaders, Operations Manager, QA Department, and Project Managers through the CAR process. This is the only form of "interpretation" of data that is routinely performed by the laboratory and is generally only a comparison to the regulatory requirements.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

#### 26.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If **TestAmerica Ontario**, **CA** is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

#### 26.6 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

**Note:** This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

**Note:** Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

**26.6.1** Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please

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notify us immediately by telephone at the 1-909-370-4667 or by replying to the email, and delete this material from any computer).

#### 26.7 FORMAT OF REPORTS

The formats of reports are designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

#### 26.8 <u>AMENDMENTS TO TEST REPORTS</u>

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 13).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "\_Revision". The revised report will have the word "Revised" next to the date rather than the word "reported".

When the report is re-issued, a notation of why the report was revised is placed on the cover/signature page of the report or at the top of the narrative page with a brief explanation of reason for the re-issue and a reference back to the last final report generated. For Example: Report was revised on 11/3/07 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/07 at 10:47am.

### 26.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

#### 26.9.1 Sample Reanalysis Policy

Because there is a certain level of uncertainty with any analytical measurement a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g. sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client specific arrangements for reanalysis protocols can be established.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits
  for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples ≤ 5x the
  reporting limit, the original analysis will be reported. At the client's request, both results may
  be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

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 Due to the potential for increased variability, reanalysis may not be applicable to Nonhomogenous, Encore, and Sodium Bisulfate preserved samples. See the Operations Manager, QA Manager, or Laboratory Director if unsure.

#### 26.9.2 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely <u>no possible</u> impact on the interpretation of the analytical results and there is <u>no possibility</u> of the change being interpreted as misrepresentation by anyone inside or outside of our company.

#### 26.9.3 Multiple Reports

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

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#### Appendix 1

# TESTAMERICA EMPLOYEE ETHICS STATEMENTS

Refer to CA-L-P-001 for complete policy.

## TestAmerica EMPLOYEE ETHICS STATEMENT

I understand that TestAmerica is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

- With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:
- I will not intentionally report data values that are inconsistent with the actual values observed or measured.
- I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations.
- I will not intentionally misrepresent another individual's work as my own or represent my own work as someone else's.
- I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers; I shall not modify data (either sample or QC data) unless the modification can be technically justified through a measurable analytical process, such as one deemed acceptable to the laboratory's Standard Operating Procedures, Quality Assurance Manual or Technical Director. All such modifications must be clearly and thoroughly documented in the appropriate laboratory notebooks/worksheets and/or raw data and include my initials or signature and date.
- I shall not make false statements to, or seek to otherwise deceive, members of Management or their representatives, agents, or clients/customers. I will not, through acts of commission, omission, erasure, or destruction, improperly report measurement standards, quality control data, test results or conclusions.
- I shall not compare or disclose results for any Performance Testing (PT) sample, or other similar QA or QC requirements, with any employee of any other laboratory, including any other TestAmerica laboratory, prior to the required submission date of the results to the person, organization, or entity supplying the PT sample.
- I shall immediately inform my supervisor or other member of management regarding any intentional or unintentional reporting of my own inauthentic data. Such report shall be given both orally and in writing to the supervisor or other member of management contacted and to the local Quality Assurance Manager. The Quality Assurance Manager will initial and date the information and return a copy to me. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and will immediately report its occurrence. If I have actual knowledge of such acts committed by any other employees, and I do not report such information to designated members of Management, it shall be considered as serious as if I personally committed the offense. Accordingly, in that event, I understand that I may be subject to immediate termination of employment.
- I understand that if any supervisor, manager, or representative of TestAmerica management instructs, requests, or directs me to perform any of the aforementioned improper laboratory practices, or if I am in doubt or uncertain as to whether or not such laboratory practices are proper, I will not

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comply. In fact, I must report such event to all appropriate members of Management including, but not limited to, the Lab Director, all supervisors and managers with direct line reporting relationship between me and the Lab Director, and the local Quality Assurance representative, excluding such individuals who participated in such perceived improper instruction, request, or directive. In addition, I may contact Corporate Quality Assurance / Ethics Compliance Officer(s) for assistance.

- I understand the critical importance of accurately reporting data, measurements, and results, whether
  initially requested by a client, or retained by TestAmerica and submitted to a client at a later date, or
  retained by TestAmerica for subsequent internal use;
- I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the TestAmerica family of companies.
- I shall not accept gifts of a value that would adversely influence judgment.
- I shall avoid conflicts of interest and report any potential conflicts to the management (e.g. employment or consulting with competitors, clients, or vendors).
- I shall not participate in unfair competition practices (e.g. slandering competitors, collusion with other labs to restrict others from bidding on projects).
- I shall not misrepresent certifications and status of certifications to clients or regulators.
- I shall not intentionally discharge wastes illegally down the drain or onto the ground.
- I understand that any attempt by management or an employee to circumvent these policies will be subject to disciplinary action.

As a TestAmerica employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

I understand that if my job includes supervisory responsibilities, I shall not instruct, request, or direct any subordinate to perform any laboratory practice which is unethical or improper. Also, I shall not discourage, intimidate, or inhibit an employee who may choose to appropriately appeal my supervisory instruction, request, or directive which the employee perceives to be improper, nor retaliate against those who do.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination of my employment. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE	Date
Supervisor/Trainer:	Date
	Work Instruction No. CA-WI-005

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Date

Work Instruction No. CA-WI-006

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## TestAmerica CONFIDENTIALITY AND PROPRIETARY INFORMATION AGREEMENT

TestAmerica and their predecessors, in their businesses, have developed and use commercially valuable technical and non-technical information and to guard the legitimate interests of TestAmerica and its clients, it is necessary to protect certain information as confidential and proprietary.
I,, understand and acknowledge that during the term of my employment by TestAmerica, I will be privy to and entrusted with certain confidential information and trade secrets of TestAmerica and its clients.
Confidential information and trade secrets include, but are not limited to: customer and client lists; price lists; marketing and sales strategies and procedures; operational and equipment techniques; standard operating procedures; business plans and systems; quality control procedures and systems; special projects and technological research, including projects, research and reports for any government entity or client; client's plans and processes; client's manner of operation; the trade secrets of clients; client's data; vendor or supplier pricing; employee lists and personal information, and any other records, data, files, drawings, inventions, discoveries, applications, or processes which are not in the public domain.
I agree as follows:
1. I will not in any way, during the term of my employment, or at any time thereafter, except as authorized in writing by the Legal Department of TestAmerica or the client where client data is involved, disclose to others, use for my own benefit, remove from TestAmerica's premises (except to the extent off-site work is approved by my supervisor), copy or make notes of any confidential information and/or trade secrets of TestAmerica or its clients, excepting only that information which may be public knowledge. Technical and business information of any previous employer or other third party which I may disclose to TestAmerica shall be limited to that which was acquired legitimately and disclosed to me without restriction as to secrecy.
2. I agree that all inventions (whether or not patentable) conceived or made by me during the period of my employment by TestAmerica shall belong to TestAmerica, provided such inventions grow out of my work for TestAmerica and are related to the business of TestAmerica. I agree to disclose and assign such inventions to TestAmerica. In California, this provision shall not apply to any invention which qualifies fully under Section 2870 of the California Labor Code.
3. On termination of my employment from TestAmerica, I will deliver to TestAmerica all documents, records, notes, data, memoranda, files, manuals, equipment and things of any nature which relate in any way to confidential information and/or trade secrets of TestAmerica or its clients and which are in my possession or under my control.
4. I agree that during the period of my employment and for one (1) year from and after the termination (for any reason) of my employment with TestAmerica, I shall not directly or indirectly (without first obtaining the written permission of TestAmerica), recruit for employment, or induce to terminate his or her employment with TestAmerica, any person who is an active employee of TestAmerica on the last day of my employment with TestAmerica.
5. I acknowledge that if I were to breach any provision of this Confidentiality Agreement, money damages will be inadequate, and I hereby agree that TestAmerica shall be entitled, where appropriate, to specific performance and/or injunctive relief (i.e. to require me to comply with this Agreement). I further acknowledge that the willingness of TestAmerica to hire me or to continue my employment constitutes full and adequate consideration for the agreements, and obligations to which I have agreed as set forth in this document.
I have executed this Agreement, intending to be legally bound.

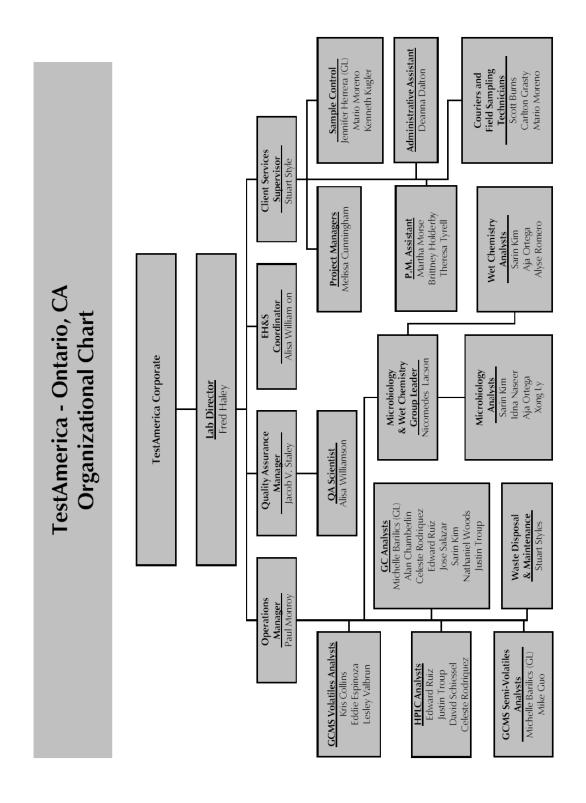
Signature

Printed Name

#### Appendix 2

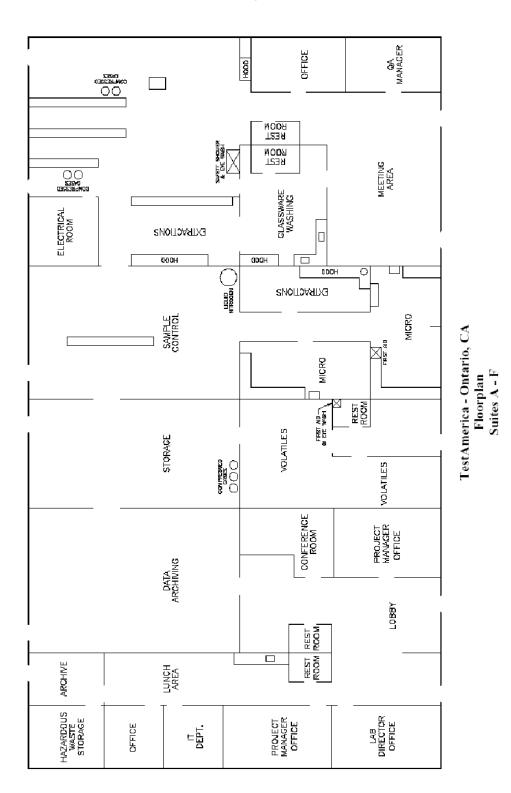
#### **Laboratory Organization Chart**

(The most current chart can be obtained from the QA Manager or Lab Director/Manager)



### **Appendix 3**

## **Laboratory Floor Plan**



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Appendix 4

Accredited methods

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## **Quality Control Limits Summary**

Method	Breakdown check or LPC (%)	Surrogat e (% Rec.)	Internal Standard ( % Rec.)	IDOC (+/- % Rec.)	IDOC (<= %RPD)	MDL Check (+/- %Rec.)		LFB Frequency (% of samples)	LFB/LFBD (<= %RPD)	LFM (+/- %Rec.)	LFM Frequency (% of samples)	LFM / LFMD (<= %RPD)	CV (+/- %Rec.)	Second Source QCS Frequency
EPA 504,1		сс	NA	30	RSD 20	40	30	10	сс	35	5	NA	Same as LFB	Quarterly
EPA 505	20	NA	NA	30	20		30, CC	5	CC (20)	35	10	20, CC	30	Quarterly
EPA 515,4		+/-30	+/- 50 from I Cal average	20	20		30, 50 low level		NA	30, 50 low level	5	30, 50 low level	30, 50 low level	Every new set of standards
CA SRL 524M 123 TCP		NA	> 80 from the I Cal average	7 reps, 20	20		20	10	NA (CC)	NA (CC)	NA (5)	20, CC	20	Quarterly
EPA 524,2		>30/50 *	>30/50*	20	20		30	5	сс	cc		сс	30	Quarterly
EPA 525,2	20	>30/50 *	>30/50*, <70**	30	30		30	5	сс	30	5	сс	30	Quarterly
EPA 531,1		NA	+/-30	20	20		20, CC		CC (20)	35	5	NA (CC)	20	Quarterly
EPA 547		NA	NA	30	RSD 30		30, CC		CC (30)	Use LFB Limits	10	Use LFB Limits	20	Quarterly
EPA 548,1	DFTPP	NA	+/-30 > 30/50 * for CCV	20	30				CC (30)	Use LFB Limits	10	Use LFB Limits	30	Quarterly
EPA 549,2		NA	NA	30	30		30, CC		CC (30)	Use LFB Limits	10	Use LFB Limits	20	Quarterly
EPA 552,2	LPC	+/-30	Mean, +/-15 from the daily CV	20	20				NA (CC,20)	30	10	NA (CC,20)	Same as LFB	Quarterly
EPA 8015B (MeOH / EtOH)			NA				30, CC		CC (30)	CC (30)	5	CC (30)	15	Every Calibration
EPA 8315A		NA	NA				30, CC		CC (30)	CC (30)	5	CC (30)	15	Every Calibration

<sup>\* 30%</sup> from the most recent CCV, or 50 % from the initial calibration.

<sup>\*\*</sup> Post extraction IS > 70% recovery

#### **Appendix 5 Glossary/Acronyms**

#### Glossary:

#### **Acceptance Criteria:**

Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

#### **Accreditation:**

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

## **Accrediting Authority:**

The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

#### Accuracy:

The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

#### Analyst:

The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

#### Assessment:

The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

#### **Assessment Criteria:**

The measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

#### **Assessment Team:**

The group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (NELAC)

#### Assessor:

One who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (NELAC) Audit:

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A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

#### Batch:

Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

#### Blank:

A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

#### **Blind Sample:**

A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

## Calibration:

To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

### **Calibration** Curve:

The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

#### **Calibration Method:**

A defined technical procedure for performing a calibration. (NELAC)

#### **Calibration Standard:**

A substance or reference material used to calibrate an instrument (QAMS)

#### <u>Certified Reference Material (CRM):</u>

A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

#### **Chain of Custody:**

An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

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#### **Clean Air Act:**

The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

## <u>Comprehensive Environmental Response, Compensation and Liability Act</u> (CERCLA/SUPERFUND):

The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

#### **Compromised Samples:**

Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

#### **Confidential Business Information (CBI):**

Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

#### **Confirmation:**

Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional Cleanup procedures

(NELAC)

#### **Conformance:**

An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

#### **Corrective Action:**

The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

#### Data Audit:

A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

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#### **Data Reduction:**

The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

#### **Deficiency:**

An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

#### **Detection Limit:**

The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

#### **Document Control:**

The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

#### **Duplicate Analyses:**

The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

#### **Environmental Detection Limit (EDL):**

The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

#### **Equipment Blank:**

Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

#### **External Standard Calibration:**

Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

#### Federal Insecticide, Fungicide and Rodenticide Act (FIFRA):

The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (NELAC)

#### Federal Water Pollution Control Act (Clean Water Act, CWA):

The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

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#### Field Blank:

Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

#### Field of Testing:

NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

#### Finding:

An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (NELAC)

#### Holding Times (Maximum Allowable Holding Times):

The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

#### Inspection:

An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

#### **Internal Standard:**

A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

#### **Internal Standard Calibration:**

Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

#### **Instrument Blank:**

A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

#### **Instrument Response:**

Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

#### Laboratory:

A defined facility performing environmental analyses in a controlled and scientific manner. (NELAC)

# <u>Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):</u>

A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in

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aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intralaboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

#### **Laboratory Duplicate:**

Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

#### **Least Squares Regression (1st Order Curve):**

The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.990 for organics and 0.995 for inorganics.

#### Limit of Detection (LOD):

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. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

#### Manager (however named):

The individual designed as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

#### Matrix:

The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

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Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device. (NELAC)

#### Matrix Spike (spiked sample or fortified sample):

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

#### Matrix Spike Duplicate (spiked sample or fortified sample duplicate):

A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be 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. (QAMS)

#### Method Blank:

A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

#### **Method Detection Limit:**

The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

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#### National Environmental Laboratory Accreditation Conference (NELAC):

A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

#### National Environmental Laboratory Accreditation Program (NELAP):

The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

#### **Negative Control:**

Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

#### **NELAC Standards:**

The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (NELAC)

#### **Performance Audit:**

The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

#### <u>Performance Based Measurement System (PBMS):</u>

A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

#### **Positive Control:**

Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

#### **Precision:**

The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

#### Preservation:

Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

#### **Proficiency Testing:**

A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

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#### **Proficiency Testing Program:**

The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

#### **Proficiency Test Sample (PT):**

A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

#### **Quality Assurance:**

An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

#### **Quality Assurance [Project] Plan (QAPP):**

A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

#### **Quality Control:**

The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

#### **Quality Control Sample:**

An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

#### **Quality Manual:**

A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

#### **Quality System:**

A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

#### **Quantitation Limits:**

The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

#### Range:

The difference between the minimum and the maximum of a set of values. (EPA-QAD)

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#### **Raw Results:**

The initial, uncorrected analyte result obtained from applying the calibration to the instrument response for the analyte, and possibly the internal standard response for internally calibrated methods. This result is not corrected for dilution or preparation factors and is the "on column" concentration, not the instrument response.

#### Reagent Blank (method reagent blank):

A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

#### **Reference Material:**

A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

#### Reference Method:

A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

#### **Reference Standard:**

A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

#### Replicate Analyses:

The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

#### Requirement:

Denotes a mandatory specification; often designated by the term "shall". (NELAC)

#### Resource Conservation and Recovery Act (RCRA):

The enabling legislation under 42 USC 321 et seg. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

#### Safe Drinking Water Act (SDWA):

The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

#### Sample Duplicate:

Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Second Order Polynomial Curve (Quadratic): The  $2^{nd}$  order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2<sup>nd</sup> order regression will generate a coefficient of determination (COD or r<sup>2</sup>) that is a measure of the "goodness of fit" of the quadratic curvature

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the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r<sup>2</sup> must be greater than or equal to 0.990.

#### **Selectivity:**

(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

#### Sensitivity:

The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

#### Spike:

A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.. (NELAC)

#### Standard:

The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

#### **Standard Operating Procedures (SOPs):**

A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

#### **Standardized Reference Material (SRM):**

A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

#### **Supervisor (Group Leader):**

The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

#### Surrogate:

A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

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Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

#### **Systems Audit (also Technical Systems Audit):**

A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

#### **Technical Director:**

Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

#### Test:

A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

#### **Test Method:**

An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

#### **Toxic Substances Control Act (TSCA):**

The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

#### Traceability:

The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

#### **Uncertainty:**

A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

#### <u>United States Environmental Protection Agency (EPA):</u>

The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

#### Validation:

The process of substantiating specified performance criteria. (EPA-QAD)

#### **Verification:**

Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

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NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

#### Work Cell:

A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

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#### **Acronyms:**

BS - Blank Spike

BSD – Blank Spike Duplicate

CAR – Corrective Action Report

CV – Calibration Verification

CF - Calibration Factor

CFR - Code of Federal Regulations

COC - Chain of Custody

CRS - Change Request Form

DOC - Demonstration of Capability

DQO - Data Quality Objectives

DU – Duplicate

DUP - Duplicate

EHS - Environment, Health and Safety

EPA – Environmental Protection Agency

GC - Gas Chromatography

GC/MS - Gas Chromatography/Mass Spectrometry

HPLC - High Performance Liquid Chromatography

ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy

ICV – Initial Calibration Verification

IDL - Instrument Detection Limit

IH – Industrial Hygiene

IS – Internal Standard

LCS - Laboratory Control Sample

LCSD - Laboratory Control Sample Duplicate

LIMS - Laboratory Information Management System

MDL – Method Detection Limit

MS - Matrix Spike

MSD - Matrix Spike Duplicate

MSDS - Material Safety Data Sheet

NELAC - National Environmental Laboratory Accreditation Conference

NELAP - National Environmental Laboratory Accreditation Program

PT – Performance Testing

QAM - Quality Assurance Manual

QA/QC - Quality Assurance / Quality Control

QAPP – Quality Assurance Project Plan

RF - Response Factor

RPD - Relative Percent Difference

RSD – Relative Standard Deviation

SD – Standard Deviation

SOP: Standard Operating Procedure

TAT - Turn-Around-Time

VOA - Volatiles

VOC - Volatile Organic Compound

#### Appendix 6

#### **Laboratory Certifications, Accreditations, Validations**

**TestAmerica Ontario, CA** maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Certificate Number	Organization	Certificate Number
Arizona DHS	AZ0062		
California ELAP	1169		
NELAP	04230CA		
Colorado	NA		
CNMI DEQ	MP0001		
Guam EPA	NA		
Hawaii DOH	NA		
Nevada	CA00242		
Oregon	CA200009		
Utah	Del1		
Washington	C2032		

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site and in the QA office.

#### **Claims of Accreditation Status**

**TestAmerica Ontario, CA** has agreed to make only valid claims as to its accreditation/certification status by any authority by ensuring that the expiration dates are not exceeded and the method-specific scope or parameter lists are supportable, as required by each. Any false claims would be reported to that authority. The agreement covers the use of the authority's name, such as "Authority-Accredited," logo, or certificate number. The only valid proof of accreditation/certification is the current certificate and scope of the authority. It is the responsibility of the laboratory to make these documents available to all staff, and it is the staff's duty to reference only the current documents.

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### Appendix 7 Data Qualifiers

Qualifier	DW Ok	AZ DW OK	AZ MAP	TextBody	Comments
Qualifier	UK	UK	AZ IVIAP	,	Comments
-				Negative Ion Balance	
+				Positive Ion Balance  Result is less than the indicated value.	Used only for Flashpoint or Micro, or Use "CSTM"
>				Result is greater than the indicated value.	Used only for Flashpoint or Micro, or use "CSTM"
A-01 A1	x		N1 A1	[Custom Value]	Type the qualifier in full sentences without abbreviations or uncommon acronyms. DO NOT USE ALL CAPs. AZ requires narrative.  Microbiology only (Put 'TNTC' in CSTM qualifier)
	1			Too numerous to count.	1
A2	Х		A2	Sample incubation period exceeded method requirement.	Microbiology only (NDs ONLY)
A3	.,		A3	Sample incubation period was shorter than method requirement.	Microbiology only
A4	Х		A4	Target organism detected in associated method blank.	Microbiology only (NDs ONLY)
A5			A5	Incubator/water bath temperature was outside method requirements	Microbiology only
A6			A6	Target organism not detected in associated positive control.	Microbiology only
A7	X		A7	Micro sample received without adequate headspace.	Microbiology only (Coliforms)
A8	х		N1	Result is greater than or equal to the indicated value.	Microbiology only. Won't really be used, 'CSTM' qualifier is used instead.
A9	X			Bacterial results confirmed	
A10			N1	Results based upon colony counts outside the acceptable range.	FL qualifier
A11				If the student t value is less than or equal to 2.78, the test lot is acceptable. If the student t value is greater than 2.78, the test lot produced significantly different results and the test lot is rejected.	For client specific Student-t test on reagent water.
A12			N1	Atypical growth	Microbiology only
A13			N1	Atypical growth appears to have a toxic effect on surrounding growth, thus affecting the plate count.	Microbiology only
AB1				Asbestos found in mastic	
AB2				Asbestos <1%	
AB3				Asbestos in soil	
AB4				Asbestos found in only in tile	

0 1111	DW	AZ DW			
Qualifier	Ok	OK	AZ MAP	TextBody	Comments
AB5	х		B1	Asbestos in mastic & tile  Analyte was detected in the associated Method Blank.	Requires internal CAR. Flag method blank and all associated samples with positive hits. Do not flag blank for J-flag hits unless regulatory limit has been exceeded
B1	х		B7	Analyte was detected in the associated method blank. Analyte concentration in the sample is greater than 10x the concentration found in the method blank.	Requires internal CAR.
B2			B2	Non-target analyte detected in method blank and sample, producing interference.	Requires internal CAR.
В3	Х		В3	Target analyte detected in calibration blank at or above the method reporting limit.	Requires internal CAR.  AZ - Metals and IC only. Requires internal
B4 B5	x		B4 B5	Target analyte detected in blank at/above method acceptance criteria.  Target analyte detected in method blank at or above the method reporting limit, but below the trigger level or MCL.	Usually AZ but may be used by others.
В6	х		B6	Target analyte detected in calibration blank at or above the method reporting limit, but below the trigger level or MCL.	Usually AZ but may be used by others.
BQC	x		N1	Reported for batch QC purposes only. See re-analysis (RE) for final result.	Lab to add in as place holder and then set to "Not reportable"
BQC1	х		N1	Reported for batch QC purposes only. See original analysis for final result.	Lab to add in as place holder and then set to "Not reportable"
С	Х		V1	Calibration Verification recovery was above the method control limit for this analyte. Analyte not detected, data not impacted.	Flag all affected sample results. Corrective action, such as re-calibration, is required.  Not to be used on a continuous basis.
<b>C</b> 1			V7	Calibration Verification recovery was above the method control limit for this analyte, however the average % difference for all analytes met method criteria. See Calibration Summary form. [Custom Value]	Used for NDs unless reanalysis confirms sample causing interference. 8000B series methods only. Flag all affected sample results. Requires internal CAR.
C2			V8	Calibration Verification recovery was below the method control limit for this analyte, however the average % difference for all analytes met method criteria. See Calibration Summary form. [Custom Value]	Used for NDs unless reanalysis confirms sample causing interference. 8000B series methods only. Flag all affected sample results. Requires internal CAR.

	DW	AZ			
Qualifier	DW Ok	DW OK	AZ MAP	TextBody	Comments
C4			N1	Calibration Verification recovery was below the method control limit for this analyte.	Corrective Action, such as re-calibration, is required. Not to be used on a continuous basis. Requires internal CAR.
C5	x		N1	Calibration Verification recovery was below the method control limit for this analyte. An additional check standard was analyzed at the reporting limit to ensure instrument sensitivity at the reporting limit. Samples ND.	Corrective Action, such as re-calibration, is required. Not to be used on a continuous basis. CAR not required for 8000 methods if average %R meets criteria. AZ requires narrative.
<b>C</b> 6			V4	CCV recovery was below method acceptance limits. The sample could not be reanalyzed due to insufficient sample.	CAR required.
<b>C</b> 7			N1	Calibration Verification recovery was below the method control limit for this analyte due to matrix interference carried over from analytical samples. The matrix interference was confirmed by reanalysis with the same result.	Re-extraction and/or re-analysis required for all bracketed samples. Needs internal CAR.
C8			N1	Calibration Verification recovery was above the method control limit for this analyte. A high bias may be indicated.	Samples should be reanalyzed unless there are Holding time constraints, insufficient sample, or samples are ND (then use C).  Requires internal CAR
СВР			No Map	Calibration verification recovery for this analyte is outside of limits as stated in BP-GCLN Technical Requirements however the calibration verification meets the requirements as stated in the analytical method.	BP work only.
CIG			W1	The % RSD for this compound was above 20%. The average % RSD for all compounds in the calibration met the 20% criteria specified in EPA method 8000B. See the attached Initial Calibration Criteria form.	For GC or HPLC 8000B series only. Used for NDs only.
CIN			W2	The % RSD for this compound was above 15%. The average % RSD for all compounds in the calibration met the 15% criteria specified in EPA methods 8260B/8270C. See the attached Initial Calibration Criteria form.	For GCMS 8000B series only. <b>Used for NDs only.</b>
CISM			No map	The concentration indicated for this analyte is derived from a single point calibration with no MDL study.	Only to be used for special requests. Client specific
CISP			No map	The concentration indicated for this analyte is derived from a single point calibration.	Only to be used for special requests. Client specific
CF1			C1	Confirmatory analysis not performed as required by the method.	Always use with N1
CF2	х		C4	Confirmatory analysis was past holding time.	Report both analyses if not confirmed. Probably need N1 also.

	DW	AZ DW			
Qualifier	Ok	oK	AZ MAP	TextBody	Comments
CF5			N1	The sample was originally analyzed with a positive result, however the reanalysis did not confirm the presence of the analyte.	Use for BP Ethanol Reporting
CF6	Х			Results confirmed by reanalysis.	Chariel qualifier for alient an exiting
cl			No Map	Compound reported based on total Chlordane result being less than the reporting limit.	Special qualifier for client specific requirements. Do not use for Arizona clients.
CN1			N1	The cyanide value was greater after chlorination than before chlorination due to the sample matrix. An additional Weak Acid Dissociable Cyanide analysis was performed.	AZ requires narrative.
CN2			N1	The cyanide value was greater after chlorination than before chlorination due to the sample matrix.	AZ requires narrative.
CN3			N1	Reactive cyanide results reported from total determination method.	
CN4			N1	Amenable cyanide results reported from total determination method.	
CR			N1	The carbon range of the fuel found in the sample = [Custom Value]	When requested, enter Carbon range of fuel at the prompt.
СЅТМ	x		No Мар	[Custom Value]	Use when results need to be reported as '<' or '>' or negative values. Enter exactly as it should appear on the report (e.g. "> 50" or "-3.2", or "DNQ") This is an "isRetain" qualifier
DNQ			No Map	Detected but not quantified.	For Boeing Project to use in conjunction with J flag. PM to add to report.
DR			No Map	Sample dried prior to screening.	Used for specific sieving project.
E			N1	Concentration exceeds the calibration range and therefore result is semi-quantitative.	Use when re-analysis is for multiple dilutions.
E1			E1	Concentration estimated. Analyte exceeded calibration range. Reanalysis not possible due to insufficient sample.	
E3			E3	Concentration estimated. Analyte exceeded calibration range. Reanalysis not performed due to holding time requirements.	
FT			No Map	This analysis was performed in the field by the sampler whose name appears on the attached Chain of Custody form.	When field analyses are entered into Element not performed by our laboratory (e.g. temperature, pH)
Н			H1	Sample analysis performed past method-specified holding time.	Requires client notification prior to release of data. Requires internal CAR.

	DW	AZ			
Qualifier	DW Ok	DW OK	AZ MAP	TextBody	Comments
H1			H1	Sample analysis performed past the method-specified holding time per client's approval.	MUST HAVE DOCUMENTED CLIENT APPROVAL. Requires internal CAR
H2			H2	Initial analysis within holding time. Reanalysis for the required dilution was past holding time.	Requires client notification prior to release of data.
Н3			H3	Sample was received and analyzed past holding time.	Requires client notification prior to release of data.
H4			H4	Sample was extracted past required extraction holding time, but analyzed within analysis holding time.	Requires client notification prior to release of data. Requires internal CAR.
H5	х		N1	The sample was prepared outside of the required 8 hour holding time, however it was stored at >0° and <4°C and prepared within the method allowed 24 hour holding time."	For HPC only
H6	x		N1	The sample was received at the laboratory either past, or with insufficient time remaining on, the required 8 hour holding time. However, it was stored at >0° and <4°C and prepared within the method allowed 24 hour hold time.	For HPC only
Н8			H3	The sample was extracted past the holding time.	Add N1 also where applicable)
Н9			N1	Sample analysis performed past the EPA recommended holding time	Use for Air samples in Tedlar bags.
нті			N1	The holding time for this test is immediate. The laboratory measurement, therefore, may not be suitable for compliance purposes.	Required for AZ, may also need for other agencies
HFT				Holding time for this test is defined as either immediately or 15 minutes from collection.  Analysis was performed at the laboratory.	To be used for tests with hold times of Immediate, 15 minutes, or tests classified as "Field Tests"
ı	х		E7	Internal Standard recovery was outside of method limits. Matrix interference was confirmed by reanalysis.	
ID	х		N1	Due to the low levels of analyte found in the sample, the analyte was qualitatively identified based on the compound's retention time and the presence of a single mass ion.	For GCMS when 2 mass ions cannot be detected. (e.g. low level TBA) AZ requires narrative.
ID2			N1	Secondary ion abundance outside of method requirements. Identification based on analytical judgment	
ID3				Due to matrix unable to resolve Benzofluoranthene isomers. Value reported only in Benzo(b) category represents Total Benzo(b,k)fluoranthene.	For Matrix interference issues only
ID4			Not used	Benzo(j)fluoranthene coelutes with Benzo(k)fluoranthene. The reported result is a summation of the isomers and the concentration is based on the response factor of Benzo(k)fluoranthene	Client Specific

	DW	AZ DW			
Qualifier	Ok	ok	AZ MAP	TextBody	Comments
ID5			Not Used	Benzo(e)pyrene concentration is based on the response factor of Benzo(a)pyrene, and has not been calibrated independently.	Client Specific
J			E4	Estimated value. Analyte detected at a level less than the Reporting Limit (RL) and greater than or equal to the Method Detection Limit (MDL). The user of this data should be aware that this data is of limited reliability.	When, on a project specific basis, reporting results down to the MDL is required.
J1				Due to matrix interference, estimated data below the PQL can not be determined.	Wisconsin only
K			K1	The sample dilutions set-up for the BOD analysis did not meet the oxygen depletion criteria of at least 2 mg/l. Therefore the reported result is an estimated value only.	
K1			K2	The sample dilutions set up for the BOD analysis failed to meet the criteria of a residual dissolved oxygen of at least 1 mg/L. Therefore the reported result is an estimated value only.	
K2			K4	The seed depletion was outside the method acceptance limits. Therefore, the reported result is an estimated value only.	
К3			K5	The dilution water D.O. depletion was > 0.2 mg/L.	
K4			N1	The seed depletion was not within method recommended limits. The LCS, which is a means of checking dilution water quality and seed effectiveness, was within acceptance limits.	
K5				Residual chlorine detected. Sample dechlorinated prior to analysis.	Only for BOD Analysis!
L	х		L3	Laboratory Control Sample and/or Laboratory Control Sample Duplicate recovery was above the acceptance limits. Analyte not detected, data not impacted.	Flag all affected sample results. Requires internal CAR.
L1			L3	Laboratory Control Sample and/or Laboratory Control Sample Duplicate recovery was above acceptance limits.	When there are positive hits. Requires internal CAR. Add N-1 or N-2 if for any additional clarification.
L1AZ		Х	L1	The associated blank spike recovery was above laboratory acceptance limits. Analyte not detected, data not impacted.	For Arizona Samples only. When there are positive hits. Requires internal CAR. Add N-1 or N-2 if for any additional clarification.
L2			L4	Laboratory Control Sample and/or Laboratory Control Sample Duplicate recovery was below acceptance limits.	Use only if samples cannot be reanalyzed. Requires internal CAR. Add N-1 or N-2 for any additional clarification.
L2AZ		X	L2	The associated blank spike recovery was below laboratory acceptance limits.	For Arizona samples only. Use only if samples cannot be reanalyzed. Requires internal CAR. Add N-1 or N-2 for any additional clarification.

		ΑZ			
Qualifier	DW Ok	DW OK	AZ MAP	TextBody	Comments
L4	<u> </u>		K6	Laboratory Control Sample and/or Laboratory Control Sample Duplicate recovery was below the acceptance limits. A low bias to sample results is indicated.	Generally for BOD only. However it could be used for BP-Amoco if technical requirements are met and local clients are o.k. with it.
L5			Not used	Analyte recovery outside of specified criteria. Individual analyte criteria exceedances allowed for multi-component analyses without disqualification of data per NELAC Standard, DOD QSM and/or AFCEE QAPP.	Only for NELAC and DOD work. The lab MUST follow ALL of the sporadic marginal exceedance criteria in order to use this qualifier.
L6			N1	Per the EPA methods, benzidine is known to be subject to oxidative losses during solvent concentration.	For Benzidine only.
M1	х	Х	M1	The MS and/or MSD were above the acceptance limits due to sample matrix interference. See Blank Spike (LCS).	Flag source sample AND MS and/or MSD only.
M2	х	Х	M2	The MS and/or MSD were below the acceptance limits due to sample matrix interference. See Blank Spike (LCS).	Flag source sample AND MS and/or MSD only.
М3	х		N1	Results exceeded the linear range in the MS/MSD and therefore are not available for reporting. The batch was accepted based on acceptable recovery in the Blank Spike (LCS).	Analyte Qualifier in the LCS. AZ requires narrative.
M4	x	X	M4	The sample required a dilution due to matrix interference. Because of this dilution, the matrix spike concentrations in the sample were reduced to a level where the recovery calculation does not provide useful information. See Blank Spike (LCS).	Must be diluted below Reporting Limit.
M5	х		N1	Due to CCV failure, the MS/MSD results were not available for reporting. The batch was accepted based on acceptable recovery in the Blank Spike (LCS).	Generally a sample qualifier though it could be used as an analyte qualifier if some analytes are to be reported. AZ requires narrative.
М6			N1	Any analyte not run due to matrix	Not Recommended
M7			N1	The MS and/or MSD were above the acceptance limits. See Blank Spike (LCS).	Non-matrix related Internal CAR required.  Add N-2 if Client CAR is needed.
M8			N1	The MS and/or MSD were below the acceptance limits. See Blank Spike (LCS).	Non-matrix related Internal CAR required.  Add N-2 if Client CAR is needed.
M9	х	Х	M6	Matrix Spike recovery was high. Data Reported per ADEQ policy 0154.000	AZ Only. Use only if BS/BSD have acceptable <b>Recovery AND RPD</b> .
M10		X	M7	Matrix Spike recovery was low. Data Reported per ADEQ policy 0154.000	AZ Only. Use only if BS/BSD have acceptable <b>Recovery AND RPD</b> .
M11			N1	The MS and/or MSD were above the acceptance limits. See calibration verification (CCV)	
M12			N1	The MS and/or MSD were below the acceptance limits. See calibration verification (CCV)	

	514	AZ			
Qualifier	DW Ok	DW OK	AZ MAP	TextBody	Comments
M13			N1	The sample spiked had a pH of less than 2. 2-Chloroethylvinylether degrades under acidic conditions.	For MS/MSD when batch spike has HCL and client samples unpreserved. <b>Mostly client specific.</b>
МСР	х		N1	No results were reported for the MS and/or MSD due to a clogged autosampler port. Batch was accepted based on Blank Spike (LCS) recoveries.	Requires internal CAR. AZ requires narrative.
MEN			N1	Unspiked sample results were determined from the sample portion received in an Encore sampler. The sample portions used for the MS/MSD were taken from an additional sample sleeve due to an insufficient number of Encore samplers supplied.	When insufficient Encores are available for MS/MSD. AZ requires narrative.
МНА	х	Х	M3	Due to high levels of analyte in the sample, the MS/MSD calculation does not provide useful spike recovery information. See Blank Spike (LCS).	Sample results > 4x spike level. Use whether or not the QC passes.
MNR	х		N1	No results were reported for the MS/MSD. The sample used for the MS/MSD required dilution due to the sample matrix. Because of this, the spike compounds were diluted below the detection limit.	Use as sample qualifier on the LCS. AZ requires narrative.
MNR1	х		Q8	There was no MS/MSD analyzed with this batch due to insufficient sample volume. See Blank Spike/Blank Spike Duplicate.	Use when there is not enough sample available to analyze MS/MSD. Use as a sample qualifier on the LCS. LCSD must be analyzed too.
MNR2	Х	Χ	Q12	Insufficient sample received to meet method QC requirements. See case narrative.	FOR AZ DRINKING WATERS ONLY.
MNR3	x			Insufficient sample received to meet method QC requirements.	Use when there is not enough sample available to analyze matrix QC. Use as a sample qualifier on the LCS. (no LCSD)
N1	Х		N1	See case narrative.	
N2	Х		N2	See corrective action report.	
Neg			Neg	The result is negative value.	For Redox Potential only.
NFP			No Map	Non-fuel pattern present.	
Р	х		Q3	The sample, as received, was not preserved in accordance to the referenced analytical method.	except for metals
P1			Q4	Sample received and analyzed without chemical preservation.	
P2			Q5	Sample received without chemical preservation, but preserved by the laboratory.	
P3			Q6	Sample was received above recommended temperature	
P4			Q10	Sample received in inappropriate sample container.	
P5	Х		Q9	Insufficient sample received to meet method QC requirements.	

Qualifier	DW Ok	AZ DW OK	AZ MAP	TextBody	Comments
P6				Sample received unpreserved, however the sample was analyzed within 7 days per EPA recommendation.	For unpreserved Volatiles (8000 Series)
P7			N1	Sample filtered in lab.	
P8			N1	Sample unable to be adjusted to correct pH due to matrix.	
P9			Q3	This analyte has been shown to degrade upon preservation with HCl and cannot accurately be quantitated.	For 2 CVE and Ac and Ac. Clients need to be made aware that acid preservation is not an option for 2-CVE, Acrolein and Acrylonitrile.
рН			N1	pH = [Custom value]	AZ requires narrative.
P-HS			Q2	Sample container contained headspace.	
PassY				Pass	"isRetain" for visual inspection for Oil
PassN				Not Pass	"isRetain" for visual inspection for Oil
Q1			No Map	Does not match typical pattern	When Client needs specific info on Fuel Patterns
Q2			No Map	The chromatographic pattern is consistent with diesel fuel.	When Client needs specific info on Fuel Patterns
Q3			No Map	The chromatographic pattern is not consistent with diesel fuel.	When Client needs specific info on Fuel Patterns
Q4			No Map	The hydrocarbons present are a complex mixture of diesel range and heavy oil range organics.	When Client needs specific info on Fuel Patterns
Q5			No Map	Results in the diesel organics range are primarily due to overlap from a gasoline range product.	When Client needs specific info on Fuel Patterns
Q6			No Map	Results in the diesel organics range are primarily due to overlap from a heavy oil range product.	When Client needs specific info on Fuel Patterns
Q7			No Мар	The heavy oil range organics present are due to hydrocarbons eluting primarily in the diesel range.	When Client needs specific info on Fuel Patterns
Q8			No Map	Detected hydrocarbons in the gasoline range appear to be due to overlap of diesel range hydrocarbons.	When Client needs specific info on Fuel Patterns
Q9			No Map	Hydrocarbon pattern most closely resembles [Custom Value].	When Client needs specific info on Fuel Patterns
Q10			No Map	Hydrocarbon pattern most closely resembles a blend of [Custom Value].	When Client needs specific info on Fuel Patterns
Q11			No Мар	Detected hydrocarbons in the diesel range do not have a distinct diesel pattern and may be due to heavily weathered diesel.	When Client needs specific info on Fuel Patterns

	DW	AZ DW			
Qualifier	Ok	OK	AZ MAP	TextBody	Comments
Q12			No Map	Detected hydrocarbons in the diesel range do not have a distinct diesel pattern and may be due to heavily weathered diesel or possibly biogenic interference.	When Client needs specific info on Fuel Patterns
Q13			No Map	Detected hydrocarbons do not have pattern and range consistent with typical petroleum products and may be due to biogenic interference.	When Client needs specific info on Fuel Patterns
QB			No Map	Quantitated against a Bunker C Oil standard.	Use as "Analyte Qualifier"
QC4			No Мар	Quantitation begun immediately before the retention time of tert-Butanol (TBA).	Only for TPH when C4 carbon range is requested. <b>Use as Analyte qualifier.</b>
QCM			No Map	Quantitation begun immediately following the methanol peak.	Only for TPH when C4 carbon range is requested. <b>Use as Analyte qualifier.</b>
QD			No Map	Quantitated against a diesel fuel standard.	Use as "Sample Qualifier"
QG			No Map	Carbon range C6-C12 quantitated against a gasoline standard.	Use as "Analyte Qualifier" To be used with the analyte "Volatile Fuel Hydrocarbons".
QG1			No Map	Quantitated against a gasoline standard.	Use as "Analyte Qualifier" for any carbon range other than C6-C12
QJ			No Map	Quantitated against a jet fuel standard.	Use as "Sample Qualifier"
QM			No Map	Quantitated against a motor oil standard.	Use as "Sample Qualifier"
QMS			No Map	Quantitated against a mineral spirits standard.	Use as "Analyte Qualifier"
QP			No Map	Hydrocarbon result partly due to individual peak(s) in quantitation range.	Use when individual non-HC peaks are present.
qr			No Map	Qualitative result based on chromatographic comparison with a known standard.	
QS			No Map	Quantitated against a Stoddard solvent standard.	Use as "Sample Qualifier"
QSG			No Map	Silica Gel clean-up performed on extracts.	When Silica Gel used on DRO/ORO extracts.
QT			No Map	Quantitated against a therminol standard.	Use as "Sample Qualifier"
QU			No Map	Unquantitated hydrocarbons present in the sample outside of the reported carbon range.	Use for EFH when there are HCs above the quant range.
QV			No Map	The molecular weight of 100 was used to convert Volatile Fuel Hydrocarbons from mg/m3 to ppm by volume (ppmv).	
R	х		R4	The RPD exceeded the method control limit due to sample matrix effects. The individual analyte QA/QC recoveries, however, were within acceptance limits.	Apply to MSD only
R1			C6	The RPD between the primary and confirmatory analysis exceeded 40%. Per method 8000B, the higher value was reported.	

	DW	AZ DW			
Qualifier	Ok	OK	AZ MAP	TextBody	Comments
R2	х		R1	The RPD exceeded the method control limit.	Narrative required for AZ. (narrative likely for all). Add N-2 if Client CAR is needed.
R3	Χ		Q11	The RPD exceeded the method control limit due to sample matrix effects.	
R4	х	Х	R9	Due to the low levels of analyte in the sample, the duplicate RPD calculation does not provide useful information.	Duplicates Only. NOT for MS/MSD.
R6			R11	The RPD calculation does not provide useful information due to varying sample weights when Encore samplers are used.	Encore Samples only.
R7	Х		R6	LFB/LFBD RPD exceeded the method control limit. Recovery met acceptance criteria.	Apply to LCSD only.
R9	Х	Χ	R9	Sample RPD exceeded the laboratory control limit.	For Sample Duplicates
R10			C7	The RPD between the primary and confirmatory analysis exceeded 40%. Per method 8000B, the lower value was reported due to apparent chromatographic problems.	
R11	Х	Χ	R2	RPD exceeded the laboratory control limit. See case narrative.	When there are no "Method" Limits.
RL1	Χ	Χ	D1	Reporting limit raised due to sample matrix effects.	
RL2		Χ	D1	Reporting limit raised due to high concentrations of hydrocarbons.	
RL3		Χ	D1	Reporting limit raised due to high concentrations of non-target analytes.	
RL4	Χ	Χ	D3	Reporting limit raised due to insufficient sample volume.	
RL5		Χ	D1	Reporting raised due to high single peak analyte.	For TPH (DRO or GRO) only.
RL6		Χ	D1	Reporting limit raised due to high toxaphene concentrations.	
RL7	Χ	Χ	D2	Sample required dilution due to high concentration of target analyte.	
s			M5	Analyzed by standard addition.	Will probably only be used for metals in rare instances.
<b>S</b> 1				The correlation coefficient (r) from MSA for this analyte is less than 0.995.	
S10			N1	Insufficient sample available for reanalysis.	Narrate if EDF being reported
S2			N1	Compound is a common lab solvent and contaminant.	
S3			N1	Post digestion spike is out of acceptance limits for this analyte	
<b>S</b> 4			No Map	Sample was received by the laboratory with moisture in the charcoal tube. Sample results may be biased low.	
<b>S</b> 5			No Map	The fineness factor used to calculate the ECCE was determined by Servi-Tech Laboratories.	
S6			N1	Sediment present.	
<b>S7</b>			N1	Sample breakthrough to 2nd section is > 10%. Results may be biased low.	
S8			No Map	Acid concentration not matched	

	DW	AZ DW			
Qualifier	Ok	OK	AZ MAP	TextBody	Comments
S9			N1	Unable to digest full amount of sample due to matrix problem.	
S11			N1	Direct sample analysis. No preparation performed.	Generally for metals and special matrices prevent sample digestion. <b>Do not use on routine dissolved metals.</b>
SB			No Map	Sustained burning when exposed to open flame.	For Ignitability only. For all positive hits.
sc			No Map	Analytical results not reliable due to potential sample container contamination	For low level Volatiles when contamination is the likely cause of the result.
SF				Reactive sulfide results reported from total determination method.	
SR			No Map	Rogers Ratio is not applicable for this sample. Concentrations of Dissolved Gasses do not exceed PGE specified limits.	client specific
SME			No Map	In the presence of high concentrations of MTBE, there is a potential for breakdown of MTBE into Methanol.	Use only with positive MeOH hits and there is a significant MTBE peak also in the chromatogram.
T1			T1	Method approved by EPA, but not yet licensed by ADHS.	AZDHS only
Т3			T3	Method not promulgated either by EPA or ADHS.	AZDHS only
T4	х		T2	The cited licensed method does not contain this analyte as part of the method compound list.	
T5			N1	Less than the prescribed sample amount was available to perform the leachate extraction.  The volume of extraction fluid was adjusted proportionately based on the method prescribed ratio of extraction fluid to sample weight.	Internal CAR not required if documented in extraction log.
Т6			N1	The temperature during the 18 hour TCLP extraction exceeded the 21-25 degrees C range stated in EPA Method 1311. The temperature range during the extraction was [Custom Value] degrees C.	Enter the temperature range during the extraction when prompted (e.g. 20-27)
Т7			T4	Tentatively identified compound. Concentration is estimated based on the closest internal standard.	
TMP	Х		No Map	Temperature taken in the field at the time of sampling.	Only when lab is reporting temperature into an ELMNT analysis code.
TRM			No Map	Per client request, the sample was digested according to section 4.1.4 of "Methods for the Chemical Analysis of Water and Wastes 1983". The sample was subsequently prepared and analyzed by EPA Method 245.1.	Boeing Total Recoverable Mercury ONLY.
TVO			No Map	Based on the sum of the concentrations of the compounds in the EPA 8010/8020 list.	Client Specific for special Air test code.

	DW	AZ DW			
Qualifier	Ok	ок	AZ MAP	TextBody	Comments
Х	Х		No Map	Exceeds regulatory limit.	PM to apply as an "Analyte" Qualifier.
X1	Х		No Map	Exceeds specified permit limit.	PM to apply as an "Analyte" Qualifier.
Z	х	Х	S6	Due to sample matrix effects, the surrogate recovery was below the acceptance limits.	Re-extraction and/or re-analysis required
<b>Z</b> 1			S10	Surrogate recovery was above acceptance limits.	AZ requires narrative. (Narrative likely for all.)
Z2	Х		S4	Surrogate recovery was above the acceptance limits. Data not impacted.	Only use if sample results are ND.
<b>Z</b> 3	Х	Х	S8	The sample required a dilution due to the nature of the sample matrix. Because of this dilution, the surrogate spike concentration in the sample was reduced to a level where the recovery calculation does not provide useful information.	Only if diluted below calibration range for surrogate. Surrogates in MB and LCS must pass to use this qualifier.
<b>Z</b> 5	х		N1	Due to sample matrix effects, the surrogate recovery was outside acceptance limits.  Secondary surrogate recovery was within the acceptance limits.	For Volatile TPH and could be used for PCBs in some cases. AZ requires narrative.
<b>Z</b> 6			S7	Surrogate recovery was below acceptance limits.	When reanalysis not performed
<b>Z</b> 7	х	Х	S11	Surrogate recovery was high. Data reported per ADEQ policy 0154.000.	For AZDHS only. Surrogate passes in LCS but not in sample.
<b>Z8</b>	х	Х	S12	Surrogate recovery was low. Data reported per ADEQ policy 0154.000.	For AZDHS only. Surrogate passes in LCS but not in sample.
<b>Z</b> 9			N1	Unable to calculate surrogate recovery due to matrix interference.	When matrix effects prevent the reporting of surrogates.
ZX	Х		N1	Due to sample matrix effects, the surrogate recovery was outside acceptance limits.	Use for High bias. Re-extraction and/or reanalysis required (Narrate for AZ)



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10.5.09

# Title: LOW BACKGROUND GAS FLOW PROPORTIONAL COUNTING (GFPC) SYSTEM ANALYSIS

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#### This SOP was previously identified as SOP No. ST-RD-0403 Rev. 7

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#### 1.0 SCOPE AND APPLICATION

- 1.1 This SOP is applicable to all Low Background Proportional Counting instruments. TestAmerica St. Louis performs Radium 226, 228, Strontium 89, 90, gross alpha/beta and Chlorine 36.
- 1.2 This SOP is based on SW846 method 9310, 9315 and 9320; EPA methods 900.0, 903.0, 904.0, 905.0; and DOE EML HASL 300 method Ba-01-R.
- 1.3 The SOP applies to GFPC analysis of liquid and solid matrices.
- 1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Information Management System (QuantIMS). A copy of the Structure and Analysis Code (SAC), which lists this information, is included in the appendix of this SOP.

#### 2.0 SUMMARY OF METHOD

2.1 This procedure provides instructions for the daily calibration and maintenance of the Low Background Proportional Counting instrumentation.

#### 3.0 **DEFINITIONS**

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (QAM) for a glossary of common terms and data qualifiers.
- 3.2 IQC a computerized Quality Control Program where the counting results of Radioactive check sources are entered and compared to statistical average data. A measurement within  $\pm$  3 standard deviations indicates the detector is operating within acceptable parameters.
- 3.3 αLL discriminator setting indicating the alpha lower voltage limit.
- 3.4 Alpha Voltage Only detector voltage capable of collecting ions created by alpha radiation only. Ion pairs created by beta radiation are not collected.
- 3.5 αUL discriminator setting indicating the instruments alpha upper voltage limit.
- 3.6 BLL discriminator setting indicating the beta lower voltage limit.
- 3.7 βUL discriminator setting indicating the beta upper voltage limit.
- 3.8 Crosstalk a measure of the amount of beta radiation that is collected in the alpha radiation channel; it is also a measure of alpha radiation collected in the beta channel.
- 3.9 Plateau a point on a graph of count rate vs. detector bias voltage where further increases in bias will not result in an increase in measured counting rate.
- 3.10 LB4100 LBPC (Low background Gas Flow Proportional Counting instrument).

#### 4.0 INTERFERENCES

4.1 A detector contaminated with radioactive material will result in a high background and interfere with the correct measurement of a sample.

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4.1.1 If a sample "times out" reaching 10000 counts before the allotted time, and the sample count rate is 60 cpm or greater, then another daily background check is performed on that detector. If the detector background check is unacceptable, the detector is taken Out Of Service until action is taken to bring the background check within acceptable limits. If the chamber requires action to remove contamination and a new background check is acceptable, then a 30 minute empty chamber count should be performed to determine if a new long background needs to be performed on that detector.

- 4.2 The actual counting efficiency for alpha radiation decreases greatly with a density > 6.0 mg/cm2. Therefore, the maximum acceptable mass density is typically 5 mg/cm 2 or less that 100 mg for a 2" planchet.
- 4.3 For beta radiation, reliable data may be obtained counting samples with a density as high as 10 mg/cm 2 or greater.
- 4.4 Sample thickness as well as moisture content may impact the alpha and/or beta results.

#### 5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS 5.2.1.1 None.

#### 5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

#### 6.0 EQUIPMENT AND SUPPLIES

- 6.1 Low Background Proportional Counter, equivalent to the Canberra/Oxford/Tennelec LB4100, or Protean MPC9604.
- 6.2 Gas mixture, 90% argon, 10% Methane
- 6.3 Blank planchets
- 6.4 PC based data acquisition system, OSUM software, IQC software

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#### 7.0 STANDARDS AND REAGENTS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision
- 7.2 Radioactive sources to measure beta radiation,: Sr-90 and Ra-228 sources.
- 7.3 Radioactive sources to measure alpha radiation: Am-241, Th-230 and Ra-226

#### 8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 See associated sample preparation SOPs ST-RC-0020, ST-RC -0021, ST-RC -0036, ST-RC -0040, ST-RC -0041 and ST-RC -0050, for more detailed information.

#### 9.0 QUALITY CONTROL

#### 9.1 **Batch**

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a <u>method blank</u>, a <u>Laboratory Control Sample</u> (LCS), and Matrix Spike (MS)/ Sample Duplicate (Dup). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
  - 9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

#### 9.2 **Method Blank (MB)**

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.

#### 9.3 Laboratory Control Sample (LCS)

- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every sample batch.

#### 9.4 **Matrix Spike**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

#### 9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

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#### 9.6 Procedural Variations/ Nonconformance and Corrective Action

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

#### 10.0 CALIBRATION AND STANDARDIZATION

#### 10.1 Voltage Plateau Determination

#### 10.1.1 **Frequency**:

10.1.1.1 Performed as a part of the Intial Calibration.

#### 10.1.2 Voltage Plateau Determination on LB4110 Red

- 10.1.2.1 Place the Am-241 sources in Drawers A and B.
- 10.1.2.2 Select the auto-sequence file "Plateau"
- 10.1.2.3 Highlight the detectors in Drawers A and B
- 10.1.2.4 Click on 'Run"
- 10.1.2.5 Type 'Alpha' for the sample ID for the detectors with the alpha source
- 10.1.2.6 Click 'Done'. The voltage plateau will begin automatically.
- 10.1.2.7 When the alpha counts are complete, the highlighted detectors will flash. Click 'Unit status' and highlight the voltage plateau file name. Click on 'Re-load'. Enter 'Beta' as the sample ID.
- 10.1.2.8 Place the  $Sr^{90}$  beta source on the detector.
- 10.1.2.9 Click 'Done'. The voltage plateau will complete automatically.
- 10.1.2.10 When the plateau is complete click on 'Data Output'. Select the plateau file. Print the graphs and data for calibration packages. Archive the data file.
- 10.1.2.11 Repeat the steps above for drawers C and D.

#### 10.1.3 Voltage Plateau Determination on Protean MPC 9604

10.1.3.1 The manufacturer has counted plateaus and permanently set the discriminator voltages. The manufacturer does not recommend recounting plateaus. The detectors are manufactured to have a high dead time. However, a cross talk test may be performed using different nuclide sources to indicate acceptable discriminator settings. This procedure is outlined in the discriminator setting section of this SOP.

#### 10.2 **Discriminator Settings**

#### 10.2.1 Frequency:

10.2.1.1 Performed as a part of the Intial Calibration.

#### 10.2.2 Discrimator Settings on LB4110 Red

- 10.2.2.1 From the unit menu, select 'Change ROI'
- 10.2.2.2 Place a beta source in each detector
- 10.2.2.3 Highligh a detector with a beta source
- 10.2.2.4 The alpha upper limit should be set at 100% and the beta lower limit should be set a 0%. The alpha lower limit and the beta upperlimit should both be set at 50%.
- 10.2.2.5 Select 'Count'. Accumulate at least 100,000 beta counts
- 10.2.2.6 Reduce the beta upper limit/alpha lower limit until there is 3.5% beta into alpha crosstalk
- 10.2.2.7 Raise the alpha lower limit until there is 0.10% beta into alpha crosstalk.
- 10.2.2.8 Raise the beta upper limit until it is 5% less than the alpha lower limit.
- 10.2.2.9 Select 'Halt'.
- 10.2.2.10 Repeat steps above until all detectors have been set.

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10.2.2.11 Select 'Close'. Discriminator settings are updated automatically.

#### 10.2.3 Discrimator Settings on Protean MPC 9604

- 10.2.3.1 Collect a minimum of 10,000 counts for each of Am-241, Th-230 and Po-210 sources
- 10.2.3.2 Calculate the percentage of crosstalk and compare the results to historical and expected values. Consult the Technical director if the values fall out of range.

#### 10.3 **Initial Calibration**:

#### 10.3.1 **Frequency**:

- 10.3.1.1 The Gas Flow Proportional Counter (GFPC) is calibrated initially and each year thereafter. Recalibration may be required if indicated during the operation of the instrument.
- 10.3.2 The specific calibration source preparations can be found in the file containing the previous calibration.
- 10.3.3 All nuclide sources shall be NIST traceable.
- 10.3.4 The efficiency calibration shall consist of at least seven single or dual sets of mass attenuated calibration standards, unless a single point source efficiency is to be determined.
- 10.3.5 The standards shall have enough activity to generate at least 10000 counts in 90 minutes of count time for the most highly attenuated source. The count rate shall not exceed 5000 counts per second.
  - 10.3.5.1 For alpha and beta analysis, separate sets of calibration sources shall be prepared.
- 10.3.6 The mass attenuation is accomplished by utilization of a salt solution with comparable make up to the majority of samples seen in the laboratory.
  - 10.3.6.1 Alternatively, the mass attenuation may be accomplished by using the same carrier solution used in a specific analysis.
- 10.3.7 Each standard shall be counted in every detector to be calibrated.

#### 10.3.8 Criteria for a Single or Dual Set Calibration

- 10.3.8.1 The efficiency of the detector (the dependent variable) shall be plotted on a single graph against the masses (the independent variable) for all data points.
- 10.3.8.2 The equation of the calibration curve shall be determined using polynomial functions and be included on the plot of the curve. The curve shall be continuous and smooth.
- 10.3.8.3 The degree of the polynomial shall not exceed three. The number of discreet source pairs shall be two more than the degree of the polynomial.
- 10.3.8.4 The percent difference of the measured efficiency and theoretical efficiency shall be calculated for all data points.
- 10.3.8.5 Points that are visual outliers or demonstrate less than 15 percent difference between the measured efficiency and theoretical efficiency may be removed at the analyst's discretion. Low residual mass sources and samples are difficult to plate with acceptable duplicate precision. Therefore, high outliers may not necessarily be removed from the calibration if they mimic live sample masses. In any case outliers above 15 percent shall be removed from the calibration curve. No more than 20 percent of the data points may be removed. Reasons for removal or inclusion of outliers shall be documented in the calibration narrative. Once outliers are removed, the percent difference between the measured efficiency and theoretical efficiency must be recalculated using the new polynomial coefficients generated from removal of data points. If outliers over 15 percent difference remain between the measured efficiency and theoretical efficiency the Radiochemistry Manager/QA must be consulted before calibration may continue.

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- 10.3.8.6 The coefficient of determination  $(r^2)$  shall be calculated and displayed on the plot with the equation of the trend line. An  $r^2$  greater than or equal to 0.9 is required to proceed to counting of verification sources.
- 10.3.8.7 If the coefficient of determination (r²) is not greater than or equal to 0.9 on the plot of all data points (with or without outliers removed), the data may be plotted using the mean of the paired values for both the efficiencies and the masses. This action is acceptable to reduce the variability caused when plating low mass sources. Calculate the percent difference of each datum value from the mean of the paired points. If the percent difference for any datum value is greater than 10 percent for alpha or 7.5 percent for beta delete the data pair and perform another statistical fit to the data. More than 20 percent of all data points may not be removed from the curve. The coefficient of determination (r²) shall be calculated and displayed on the plot with the equation of the trend line. An r² greater than or equal to 0.9 is required to proceed to counting of verification sources.

#### 10.4 Independent Calibration Verification (ICV)

- 10.4.1 **Frequency**:
  - 10.4.1.1 Performed with every intial calibration
- 10.4.2 GFPC initial calibrations must be verified by a second source standard.
- 10.4.3 The ICV standard is NIST traceable.
- 10.4.4 <u>ICV for Dual Set Calibrations</u>:
  - 10.4.4.1 Prepare at least one set of calibration verification sources consisting of a low, medium and high residual mass within the calibration range of the curve.
  - 10.4.4.2 Prepare a blank at the same time.
  - 10.4.4.3 The sources shall contain radionuclide from a second source. Second sources may include a second dilution from the same primary source used for the calibration curve.
    - 10.4.4.3.1 Alternatively, verification source nuclides may consist of different nuclides than the calibration curve if it is customary to do so.
  - 10.4.4.4 Count each secondary source and the blank in all detectors that were calibrated.
  - 10.4.4.5 Calculate the results in terms of percentage recovery.
  - 10.4.4.6 Calculate the mean results of all masses across each detector.
  - 10.4.4.7 Criteria:
    - 10.4.4.7.1 Individual points are within 30 percent of the true value
    - 10.4.4.7.2 The mean result of all masses across all detectors is less than 10 percent.
    - 10.4.4.7.3 If any detector fails the validation tests the Technical Director must be consulted to provide corrective action.

#### 10.4.5 **ICV for Single Set Calibrations**:

- 10.4.5.1 Prepare at least one set of verification sources varying in expected mass within the calibration range of the curve.
- 10.4.5.2 Prepare a blank at the same time.
- 10.4.5.3 The sources shall contain radionuclide from a second source. Second sources may include a second dilution from the same primary source used for the calibration curve.
  - 10.4.5.3.1 Alternatively, verification source nuclides may consist of different nuclides than the calibration curve if it is customary to do so.
- 10.4.5.4 Count the secondary source and the blank in all detectors that were calibrated.
- 10.4.5.5 Calculate the results in terms of percentage recovery.
- 10.4.5.6 Calculate the mean results of all masses across each detector.
- 10.4.5.7 Criteria:
  - 10.4.5.7.1 Individual points are within 30 percent of the true value

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- 10.4.5.7.2 The mean result of all masses across all detectors is less than 10 percent.
- 10.4.5.7.3 If any detector fails the validation tests the Technical Director must be consulted to provide corrective action.

#### 10.5 Setting Performance Check Criteria After Calibration

- 10.5.1 Twenty background check samples are counted and used to establish quality control limits for the daily background checks.
- 10.5.2 Twenty alpha/beta check sources are counted after calibration and used to establish quality control limits for the daily source checks.
- 10.5.3 The limits will be a running average of the four months post calibration.
  - 10.5.3.1 The limits are to be documented.
  - 10.5.3.2 The limits will be re-established monthly at the following frequency
    - 10.5.3.2.1 1<sup>st</sup> month take first five data points from the new month and fifteen data points from the initial calibration.
    - 10.5.3.2.2 2<sup>nd</sup> month take first five points from new month, five from prior month and ten from initial calibration.
    - 10.5.3.2.3 3<sup>rd</sup> month take first five points from new month, five points each from the previous two months and five from the initial calibration.
    - 10.5.3.2.4 4<sup>th</sup> month take first five data points from new month and five points each from the previous three months.
  - 10.5.3.3 Limits are set.

#### 10.6 Alpha to Beta Crosstalk Determination

- 10.6.1 The mean mass is determined for each data point used to calculate the mass attenuation curve.
  - 10.6.1.1 These curves are calculated and plotted and the percent of alpha into beta crosstalk is determined. This is done by dividing the beta counts per minute as observed in the beta channel from the alpha calibration source counts by the sum of the alpha and beta counts per minute.
  - 10.6.1.2 The mean percent of alpha into beta is determined for each mass point by using the count data accumulated for two sets of alpha sources.
  - 10.6.1.3 The crosstalk curve is plotted as mean crosstalk values relative to the mean mass for the two sets of data.
    - 10.6.1.3.1 In this manner the crosstalk factor can be determined for any given mass.
  - 10.6.1.4 The equation of the curve shall be determined using polynomial functions.
  - 10.6.1.5 The coefficient of determination (R<sup>2</sup>) shall be calculated and displayed on the plot as well as the equation for the trendline.

#### 10.7 **Beta to Alpha Crosstalk Determination**

- 10.7.1 Since beta to alpha crosstalk does not vary across mass, a mean beta to alpha crosstalk correction factor is calculated.
- 10.7.2 The percent of beta into alpha is determined by dividing the alpha counts per minute as observed in the alpha channel from the beta calibration source counts by the sum of the alpha and beta counts per minute.
- 10.7.3 The mean percent of beta into alpha is determined for all mass points. The mean percent is insignificant in calculating results, therefore is not applied to the result calculation.

#### 10.8 Long Background

#### 10.8.1 **Frequency**:

10.8.1.1 Monthly or whenever instrument conditions have significantly changed since the previous background was performed (e.g. detector replaced, etc.)

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- 10.8.1.2 Minimum count time: 1000 minutes.
- 10.8.2 Wash the planchet holder and clean the drawers with a 20% radiac wash or ethyl alcohol.
  - 10.8.2.1 <u>Do not spray cleaner directly onto the drawers. Spray cleaner on a Kimwipe, a</u> cotton ball, or paper towel and wipe out the drawers.
- 10.8.3 Check that instrument settings are as specified in 11.1.

#### 10.8.4 Red Long Background Count Set Up

- 10.8.4.1 Place the cursor on the red box in the upper left hand corner of the screen and right click on the mouse.
- 10.8.4.2 Select 'edit parameters'. Verify the count time on the screen is set to 1000 minutes and the iterations is set to 1. If they are different than 1000 min and 1 iteration, change them to 1000 min and 1 iteration. Then select 'close' to exit this window.
- 10.8.4.3 Place clean empty planchets in instruments.
- 10.8.4.4 Place the cursor on the red box in the upper left hand corner of the screen and right click on the mouse.
- 10.8.4.5 Select 'create batch' from the instrument menu.
- 10.8.4.6 Select 'background'
- 10.8.4.7 Select the detectors that are to be counted by double clicking the mouse on the drawer desired which selects all detectors in that drawer or on each individual detector in the display.
- 10.8.4.8 Select 'run'
- 10.8.4.9 Select 'done'
- 10.8.4.10 When backgrounds are complete, review the printouts for acceptance.

#### 10.8.5 **Protean Long Background Count Set Up**

- 10.8.5.1 Double click on detector 0.
- 10.8.5.2 Select the 'source' bullet.
- 10.8.5.3 Select LB0 by clicking on the file list arrow. (L for long. B for background. 0 for detector).
- 10.8.5.4 Ensure count time is set to 1000 minutes.
- 10.8.5.5 Select 'start'.
- 10.8.5.6 Continue these steps with detectors 1-15.
- 10.8.5.7 Review the data for acceptance when the backgrounds are complete.

#### 10.8.6 Orange Long Background Count Set Up

- 10.8.6.1 Select detector 0
- 10.8.6.2 Select 'source' bullet
- 10.8.6.3 Select 'monthly long background' by clicking on the file list arrow.
- 10.8.6.4 Ensure count time is set to 1000 minutes
- 10.8.6.5 Select 'start'
- 10.8.6.6 Continue these steps with detectors 1-15.
- 10.8.6.7 Review the data for acceptance when the backgrounds are complete.

#### 10.8.7 **Long Background Criteria:**

- 10.8.7.1 The CPM for the alpha are <0.2 and the beta <2.0, the detector may be used.
  - 10.8.7.1.1 The data printout must include initials and date to indicate that has been reviewed.
  - 10.8.7.1.2 If a detector is above this limit, discard planchet.
  - 10.8.7.1.3 Clean the planchet holder with radiac wash, ethyl alcohol or a detergent spray cleaner and dry thoroughly.
  - 10.8.7.1.4 Place a clean planchet in the holder and repeat steps for that detector (s) only.
  - 10.8.7.1.5 Perform a new background.
    - 10.8.6.1.5.1 Note: the detector is tagged out of service until a successful background has been achieved.

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#### 11.0 PROCEDURES

#### 11.1 Initial Setup

- 11.1.1 Check the normal instrument settings for all controls as described below:
  - 11.1.1.1 Tank Flow 8 psi
  - 11.1.1.2 Flow Cells >/= 0.3 SCFH, the flow will vary, the target range is 0.15 to 0.20 SCFH.
- 11.1.2 The High Voltage is set as indicated in the Manuals for the LB4000/LB4100 located in the count room file cabinet. The Protean remains as set by the manufacturer and does not require adjustment.
- 11.1.3 If counting gas has just been changed or turned on, allow a minimum purge time of 30 minutes prior to operation. Record gas tank changes in the maintenance logbook.
- 11.2 Record date of Daily Background and Check Source Data in runlog logbook.

#### 11.3 Maintenance

- 11.3.1.1 Change out the counting gas when the gauge reads under 500 psi. This usually occurs every 3 to 4 days. Record in the instrument maintanence logbook.
- 11.3.1.2 Allow gas to purge a minimum of 30 minutes prior to operation.
- 11.4 Data Acquisition: Daily Background Check and Source Check

#### 11.4.1 **Daily Background Check**:

#### 11.4.1.1 **Red Instrument**:

- 11.4.1.2 Open the drawer by rotating the knob at the front of the drawer to the 'DOWN' position and pull the sample drawer out slowly. Place clean empty planchets in the sample holders.
  - 11.4.1.3 Before inserting the drawer, confirm that none of the planchets extend above the sample holder. Failure to observe this note can result in damage to the detector.
  - 11.4.1.4 Slowly insert sample drawer into the instrument and slowly rotate the positioning knob into the 'UP' position.
  - 11.4.1.5 Place the cursor on the red box in the upper left hand corner of the screen and right click on the mouse.
  - 11.4.1.6 Select 'create batch'
  - 11.4.1.7 Select 'daily background check'
  - 11.4.1.8 Select the detectors that are to be loaded by double clicking the mouse on the drawer desired or on each individual detector in the display.
  - 11.4.1.9 Select 'run'
  - 11.4.1.10 Select 'done'
  - 11.4.1.11 Measure the detector background for 200 minutes. The count time is predetermined by the protocol selected. i.e. 'daily background check'.
  - 11.4.1.12 The detector display will be yellow when the detector is counting. The detector display will turn green when the count is complete.
  - 11.4.1.13 When counting is complete, place the cursor on the red box in the upper left hand corner of the screen and <u>right click</u> on the mouse. Select 'data output' and select the data file generated by your background counts for that instrument, "DAY-###". Select 'ok' to print data.
  - 11.4.1.14 On any work station, i.e. "PC computer in the count room", double click on the IQC icon.
  - 11.4.1.15 Select 'import data'
  - 11.4.1.16 Select 'Red'. Enter the current date. Click on the file list arrow.

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- 11.4.1.17 From the file list select <u>each</u> file generated above <u>individually</u>, and then select import data. i.e. import each "Day" or "DQC" file for that instrument individually.
- 11.4.1.18 Select 'close'
- 11.4.1.19 Select 'reporting'. Verfiy the current date in both the 'start' and 'end' date fields. Select 'print' to generate the report.

### 11.4.2 **Protean Instrument**:

- 11.4.2.1 Open each detector drawer. Place clean empty planchets into each sample holder and slowly insert each sample drawer into the instrument.
- 11.4.2.2 Double click detector 0 on the Protean computer screen.
- 11.4.2.3 Select 'source log'
- 11.4.2.4 Set the time for 200 minutes.
- 11.4.2.5 Type 'DB0' in the sample id box. (D for daily. B for background. 0 for detector.)
- 11.4.2.6 Select 'start'
- 11.4.2.7 Double click detector 1 on the computer screen. Repeat steps 11.6.30 through 11.6.32 for each detector, making sure to change the number to coincide with the detector the background is counting for.
- 11.4.2.8 Remove planchets from detector drawers when counting is complete.
  - 11.4.2.9 On any work station, i.e. "PC computer in the count room", double click on the IOC icon.
  - 11.4.2.10 Select 'import data'
  - 11.4.2.11 Select 'Protean'. Enter the current date. Click on the file list arrow.
  - 11.4.2.12 Select 'close'
  - 11.4.2.13 Select 'reporting'. Verfiy the current date in both the 'start' and 'end' date fields. Select 'print' to generate the report.

### 14.1.1 **Orange Instrument**:

- 14.1.1.1 Open each detector drawer. Place clean empty planchets into each sample holder and slowly insert each sample drawer into the instrument.
- 14.1.1.2 Select detector 0.
- 14.1.1.3 Select 'source log'.
- 14.1.1.4 Select 'daily background check' by clicking on the file list arrow.
- 14.1.1.5 Select 'start'
- 14.1.1.6 Repeat these steps with detectors 1-15.

### 11.4.3 **Daily Background Criteria:**

- 11.4.3.1 Review the IQC printouts for each detector.
  - 11.4.3.1.1 If a detector fails background criteria (3 sigma), clean the detector with radiac wash or ethyl alcohol and re-run.
  - 11.4.3.1.2 If the detector fails a second time, but the CPM for the alpha are <0.2 and the beta < 2.0, the detector may be used. The data printout must include initials and date to indicate that it was checked.
  - 11.4.3.1.3 Circle any failed detector on the printout.
  - 11.4.3.1.4 Place a planchet upside down in the planchet holder to indicate that the detector is out of service for that day.

### 11.5 **Daily Source Check**

### 11.5.1 **Red Instrument**:

- 11.5.1.1 Open the drawer by rotating the knob at the front of the drawer to the 'DOWN' position and pull the sample drawer out slowly.
- 11.5.1.2 Place alpha sources in the sample holders of the A and B drawer. Place beta sources in the sample holders of the C and D drawer.

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- 11.5.1.3 Place the cursor on the red box in the upper left hand corner of the screen and right click on the mouse.
- 11.5.1.4 Select 'create batch'
- 11.5.1.5 Select 'daily source check'
- 11.5.1.6 Select the detectors that are to be loaded by double clicking the mouse on the desired drawer or on each individual detector in the display.
- 11.5.1.7 Select 'run'
- 11.5.1.8 Select 'done'
- 11.5.1.9 Measure the detector source for 2 min. The count time is predetermined by the protocol selected. i.e. 'daily source check'
- 11.5.1.10 The detector display will be yellow when the detector is counting.

  The detector display will turn green when the count is complete.
- 11.5.1.11 When counting is complete, place the cursor on the red box in the upper left hand corner of the screen and <u>right click</u> on the mouse. Select 'data output' and select the data file generated by your background counts for that instrument, "DQC-###". Select 'ok' to print data.
- 11.5.1.12 Open the drawer by rotating the knob at the front of the drawer to the 'DOWN' position and pull the sample drawer out slowly. Place beta sources in the sample holders of the A and B drawer. Place alpha sources in the sample holders of the C and D drawer.
- 11.5.1.13 Repeat steps 11.5.1.12 to 11.5.1.13.
  - 11.5.1.14 On any work station, i.e. "PC computer in the count room", double click on the IOC icon.
  - 11.5.1.15 Select 'import data'
  - 11.5.1.16 Select 'Red'. Enter the current date. Click on the file list arrow.
  - 11.5.1.17 From the file list select <u>each</u> file generated above <u>individually</u>, and then select import data. i.e. import each "Day" or "DQC" file for that instrument individually.
  - 11.5.1.18 Select 'close'
- 11.5.1.19 Select 'reporting button'. Verify the current date in both the 'start' and 'end' date fields. Select 'print' to generate the report

### 11.5.2 **Protean Instrument:**

- 11.5.2.1 Slowly open each detector drawer. Place alpha sources in sample holders of detectors 0-7. Place beta sources in sample holders of detectors 8-15 and slowly insert each drawer into the instrument.
- 11.5.2.2 Double click detector 0 on the Protean computer screen.
- 11.5.2.3 Select 'source log'.
- 11.5.2.4 Set the time for 2 minutes.
- 11.5.2.5 Type "SA0" in the sample id box. (S for source. A for alpha. 0 for detector.)
- 11.5.2.6 Select 'start'
- 11.5.2.7 Double click detector 1 on the computer screen. Repeat steps 11.5.3.5 to 11.5.3.6 for each detector, making sure to change A to B when starting the beta sources on detectors 8-15 and changing the number to coincide with the detector the source is on.
- 11.5.2.8 When the counting is complete, slowly open each detector drawer. Place beta sources in detectors 1-7. Place alpha sources in detectors 8-15.
- 11.5.2.9 Double click detector 0 on the Protean computer screen.
- 11.5.2.10 Type "SB0" in the sample id box. (S for source. B for beta. 0 for the detector.)
- 11.5.2.11 Double click detector 1 on the computer screen. Repeat steps 11.5.3.8-11.5.3.10. for each detector, making sure B to A when starting the alpha sources on detectors 8-15.
- 11.5.3 Remove sources from detector drawers when counting is complete.
- 11.5.4 Orange Instrument:

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- 11.5.4.1 Slowly open each detector drawer. Place alpha sources in sample holders of detectors 0-7. Place beta sources in sample holders of detectors 8-15. Slowly insert each drawer into the instrument.
- 11.5.4.2 Select detector 0.
- 11.5.4.3 Select 'source log' bullet.
- 11.5.4.4 Select 'daily alpha check 00'.
- 11.5.4.5 Select 'start'
- 11.5.4.6 Repeat these steps for detectors 1-7 using the correlating detector number. For detectors 8-15 select 'daily beta check 08', 'daily beta check 09', and so on for each correlating detector number.
- 11.5.4.7 Slowly open each detector drawer when counting is complete. Place beta sources in detectors 1-7 and place alpha sources in detectors 8-15.
- 11.5.4.8 Select detector 0.
- 11.5.4.9 Select 'daily beta check 00'.
- 11.5.4.10 Select 'start'.
- 11.5.4.11 Repeat these steps for detectors 1-7 using the correlating detector number. For detectors 8-15, select 'daily alpha check 08', 'daily alpha check 09' and so on for each correlating detector number.
- 11.5.4.12 Remove sources from detector drawers when counting is complete.

### 12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis QAM.
- 12.2 Result calculations are performed by TestAmerica St. Louis' Rad Capture software program. These calculations are found in the TestAmerica St. Louis QAM.

### 13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036. Below is a subset of the data assessment and QC excursion types within Clouseau; the text in underline is the exact "type" line in Clouseau. For a complete and current listing, please access the software program.

### 13.2 Method Blank

- 13.2.1 Acceptance Criteria:
  - 13.2.1.1 No target analytes may be present in the method blank above the reporting limit.
  - 13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the client requirements sheet.
- 13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:
  - 13.2.2.1 Method Blank Contamination See Clouseau NCM for corrective action (e.g. reprep/reanalysis, narration). Note certain analytes are common laboratory contaminants which require special narrative comment. These compounds are so designated in Clouseau.
- 13.3 Laboratory Control Sample (LCS)
  - 13.3.1 Acceptance Criteria:

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- 13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).
- 13.3.2 Corrective Action for LCS not meeting acceptance criteria:
  - 13.3.2.1 <u>LCS Spike Recovery excursion (high)</u> See Clouseau NCM for corrective action (e.g. , reanalysis, narration).
  - 13.3.2.2 <u>LCS Spike Recovery excursion (low)</u> See Clouseau NCM for corrective action (e.g., reanalysis, narration).
- 13.3.2.3 <u>RPD/RER Duplicate excursion</u> See Clouseau NCM for corrective action (e.g., reanalysis, narration).
- 13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)
  - 13.4.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).
  - 13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:
    - 13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. See Clouseau NCM to determine if re-preparation reanalysis is required.
- 13.5 Sample result evaluation
  - 13.5.1 Tracer/Carrier recovery must be within specified limits.
  - 13.5.2 Tracer/Carrier recovery low– See Clouseau NCM for corrective action.
  - 13.5.3 <u>Tracer/Carrier recovery high</u>— See Clouseau NCM for corrective action.
    - 13.5.3.1 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:
    - 13.5.3.2 minimum number of tracer counts
    - 13.5.3.3 level of uncertainty
    - 13.5.3.4 client project requirements/approval
  - 13.5.4 These expections will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.
- 13.6 Insufficient Sample
  - 13.6.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis and narrative comment stating such is included in the report narrative. The insufficient sample description is included in the the Clouseau NCM within the type defining the excursion.

### 14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of this SOP.
- 14.2 Demonstration of Capability
  - 14.2.1 Initial and continuing demonstrations of capability requirements are established in QAM section 18.3.
- 14.3 Training Qualification
  - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
  - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in QAM section 18.3.

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Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in QAM section 18.3.

### 15.0 VALIDATION

15.1 Laboratory SOPs are based on standard reference EPA and DOE methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica ST Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

### 16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

- All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
  - 16.2.1 The following waste streams are produced when this method is carried out.
    - 16.2.1.1 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

### 17.0 REFERENCES

- 17.1 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, method Ba-01-R, Beta Radioassay
- 17.2 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 1, Method 900.0 Gross Alpha and Gross Beta Radiochemistry
- 17.3 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 6, Method 903.0 Alpha-Emitting Radium Isotopes
- 17.4 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 8, Method 904.0 Radium 228
- 17.5 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 9, Method 905 Radioactive Strontium in Drinking Water
- 17.6 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9310, Gross Alpha and Gross Beta
- 17.7 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9315, Alpha-Emitting Radium Isotopes

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- 17.8 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9320, Radium 228
- 17.9 TestAmerica St. Louis Quality Assurance Manual, current revision
- 17.10 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions
- 17.11 Associated SOPs:
  - 17.11.1 ST-PM-0002 "Sample Receipt and Chain of Custody"
  - 17.11.2 ST-QA-0002, "Standards and Reagent Preparation."
  - 17.11.3 ST-QA-0036, "Non-Conformance Memorandum (NCM) Process"
  - 17.11.4 ST-RC-0004, "Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis".
  - 17.11.5 ST-RC-0020, "Determination of Gross Alpha/Beta Activity"
  - 17.11.6 ST-RC-0021, "Gross Alpha Radition in Water using Copreciptation"
  - 17.11.7 ST-RC-0036, "Determination of Chlorine-36 in Various Matrices by GFPC"
  - 17.11.8 ST-RC-0040, 'Total Alpha Emitting Isotopes of Radium"
  - 17.11.9 ST-RC-0041, "Radium 228 in Water"
  - 17.11.10ST-RC-0050, "Preparation of Strontium 89 and 90"
  - 17.11.11 ST-RC-0300, "New Jersey 48-hour Gross Alpha Testing for Private Well Testing ACT (PWTA)

### 18.0 MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 TestAmerica St. Louis uses Thorium-230 to calibrate the GFPC system for Ra-226. Th-230 has similar alpha energies and a sufficiently long half life to eliminate the need for purification. The laboratory has historically performed well on PE programs for Ra-226, demonstrating the laboratory's ability to accurately calibrate for this isotope. Calibrating with a Ra-226 source presents a severe bias in the quantitated result. Ra-226 can be purified and separated from all other alpha emitting isotopes, but the moment after separation, alpha emitting daughters begin to grow (i.e. Radon-222, Polonium-28 and Polonium-214). As the daughters in-grow, the alpha activity changes and due to the higher alpha energies of these daughters, the measured efficiency of the GFPC changes as well. After three weeks the alpha activity from purified Ra-226 increases by a factor of four. Due to their short half lives, these daughters can not be isolated long enough to mathematically correct for the bias brought on by them. Calibrating the GFPC with Ra-226 is actually calibrating with a mix of the four isotopes and not a legitimate calibration under the cited regulation.
- Strontium 89's short half life makes it impractical to use as a calibration standard for both Radium 228 analysis, as stated in EPA method 904 and SW method 9310, and Strontium 89 analysis, as stated in EPA method 905. TestAmerica St. Louis uses a mixed Strontium 90/Yittrium 90 standard for its' GFPC beta calibration used in Gross Beta, Strontium 90, Strontium 89, and Radium 228 analyses. TestAmerica St. Louis has selected the Strontium 90/Yittrium 90 standard because it produces a stable beta emission which can be reliably used for initial and continuing calibration. By using this standard mix, we have beta emissions at the lower and upper energetic spectrum whose average is in the middle of the beta range.
- 18.3 For Radium 228 analysis, TestAmerica St. Louis uses chemical separation techniques to eliminate other potential beta emitters.
- 18.4 TestAmerica St. Louis does not perform a direct Strontium 89 analysis. TestAmerica St. Louis provides calculated results based on the difference between Total Strontium and Strontium 90.

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### 19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 Updated Sections 5, 14 and 17
- 19.2 Removed all references to the "Yellow" and "Blue" GFPC instruments.
- 19.3 Added all information pertaining to the "Orange" GFPC instrument

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Structured Analysis Code: I-FR-ZA-01-06 Target Analyte List: All Analytes	I-FR-ZA-01-0	9				Matrix: Extraction: Method: QC Program: Location:	Matrix: Extraction: Method: Program: Location:	WATER Evaporat GROSS / STANDA TestAme	WATER Evaporative Preparation, Total GROSS A/B BY GFPC SW846 9310 MOD STANDARD TEST SET TestAmerica St. Louis	, Total W846 931	10 MOD		i i
Analyte List		Detection Limits	n Limits			Check List 6501	ist 6501		v	Spike List 6548	6548		
Syn Compound	R	Units	MDL	Units	Run Date	T A Amt Units		UCL RPI	LCL UCL RPD T A Amt Units	Units	LC LC	LCL UCL RPD	
4170 Gross Alpha	က	pCi/L			0	۲ ک	73	73 136 40 C Y	≻ 0		44 150 40	0 40	
4171 Gross Beta	4	pCi/L			0	<b>∠</b> ⊃	73	73 122 40	≻		66 147	17 40	

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Structured Analysis Code: I-HZ-ZA-01-06 Target Analyte List: All Analytes	I-HZ-ZA-01-06 All Analytes						ac P	Matrix: Extraction: Method: QC Program: Location:	WATER Evaporative Preparation GROSS A/B BY GFPC (STANDARD TEST SET TestAmerica St. Louis	WATER Evaporative Preparation, Dissolved GROSS A/B BY GFPC SW846 9310 MOD STANDARD TEST SET TestAmerica St. Louis	olved 9310 MOD	RD-0403
Analyte List		<b>Detection Limits</b>	Limits				Check	Check List 6501		Spike I	Spike List 6548	
Syn Compound	RL	Units	MDL	Units	Run Date	<b>∀</b> ⊢	T A Amt Units		LCL UCL RPD T A Amt Units	A Amt Uni	IS LCL UCL RPD	
5630 Gross Alpha, Dissolved	ю	pCi/L			0	≻ ∪		73	136 40 C Y	<b>&gt;</b>	44 150 40	
5632 Gross Beta, Dissolved	4	pCi/L			0	≻ C		73	122 40 C	z	72 139 40	

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Structured Analysis Code: I-H6-ZA-01-06 Target Analyte List: All Analytes	I-H6-ZA-01-06 All Analytes						ď	Matrix: Extraction: Method: QC Program: Location:		WATER Evaporative Preparation GROSS A/B BY GFPC STANDARD TEST SET TestAmerica St. Louis	WATER Evaporative Preparation, Suspended GROSS A/B BY GFPC SW846 9310 MOD STANDARD TEST SET TestAmerica St. Louis	uspende 346 9310	MOD	RD-0403
Analyte List		Detection Limits	Limits				ç	Check List 6501	501		Spik	Spike List 6548	148	
Syn Compound	RL	Units	MDL	Units	Run Date	<b>4</b>	T A Amt Units		TON TOT	RPD T A	Amt	Juits	LCL UCL RPD T A Amt Units LCL UCL RPD	
5629 Gross Alpha, Suspended	ო	pCi/L			0	z U			73 136 40 C N	40 C N		•	44 150 40	
5631 Gross Beta, Suspended	4	pCi/L			0	z O			73 122 40	40 C		Ĭ	66 147 40	

Structured Analysis Code: A-FS-ZA-01-06 Target Analyte List: All Analytes	A-FS-ZA-01- Ali Analytes	90				:	၂၂ ၁၀	Matrix: Extraction: Method: QC Program: Location:	SOLID Dry, Gri GROSS STAND, TestAm	SOLID Dry, Grind, Thin Layer on Planchet GROSS A/B BY GFPC SW846 9310 MOD STANDARD TEST SET TestAmerica St. Louis	r on Planche S SW846 93 T	et 110 MOD		
Analyte List		Detection Limits	n Limits				Chec	Check List 6501			Spike List 6548	6548		
Syn Compound	귛	Units	MDL	Units	Run Date	۲ ۲	T A Amt Units		. UCL RP	LCL UCL RPD T A Amt Units LCL UCL	t Units	רכר ח	CL RPD	
4170 Gross Alpha	10	pCi/g			0	≻ C		42	42 111 40	≻ `		43 123	23 40	
4171 Gross Beta	10	pCi/g			0	≻ C		38	126 40	<b>≻</b>		55 1	125 40	

Structured Analysis Code: A-HI-ZA-01-06 Target Analyte List: All Analytes	A-HI-ZA-01-06 All Analytes						Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Total Dissolution GROSS A/B BY GFPC SW846 9310 MOD STANDARD TEST SET TestAmerica St. Louis	issolution FPC SW846 9 SET	310 MOD
Analyte List		Detection Limits	ı Limits				Check List 6527	st 6527		Spike List 6528	it 6528
Syn Compound	RL	Units	MDL	Units	Run Date	<b>4</b>	Amt Units	רכרו	JCL RPD T A /	Amt Units	T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD
4170 Gross Alpha	10	pCi/g			0	≻ ט		70 1	70 130 40 C Y		70 130 40
4171 Gross Beta	10	pCi/g			0	≻ C		70 1	130 40 C Y		70 130 40

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Structured Analysis Code: A-K2-ZA-01-06 Target Analyte List: All Analytes	A-K2-ZA-01-01	49					Matrix: Extraction: Method: QC Program: Location:		SOLID As Received, Thin Layer on Planchet GROSS A/B BY GFPC SW846 9310 MOD STANDARD TEST SET TestAmerica St. Louis	shet 10 MOD
Analyte List		Detection Limits	Limits			S	Check List 6501	.01	Spike List 6548	6548
Syn Compound	RL	Units	MDL	Units	Run Date	T A Amt	Units	T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD	Amt Units	LCL UCL RPD
1170 Gross Alpha	10	pCi/g			0	<b>≻</b>	4	42 111 40 C Y		43 123 40
1171 Gross Beta	10	pCi/g			0	≻ Տ	e	38 126 40 C Y		55 125 40

Structured Analysis Code: I-FR-Y9-01-06 Target Analyte List: All Analytes	I-FR-Y9-01-0	9				)	Matrix: Extraction: Method: QC Program: Location:		WATER Evaporative Preparation, Total Gross Alpha/Beta EPA 900 STANDARD TEST SET TestAmerica St. Louis	n, Total 900		
Analyte List Syn Compound 4170 Gross Alpha 4171 Gross Beta	<u>ጸ</u> 6 4	Detection Limits Units MDL pCi/L pCi/L	n Limits MDL	Units	Run Date 0 0	C Y C Y C Y	¥	1501 LCL UCL RPD T A 73 136 40 C Y 73 122 40 C Y	Amt	Spike List 6548 Units LCL 44	6548 LCL UCL RP 44 150 40 66 147 40	. <b>RPD</b> 40 40

Structured Analysis Code: I-HZ-Y9-01-06 Target Analyte List: All Analytes	I-HZ-Y9-01-0 All Analytes	و					Matrix: Extraction: Method: QC Program: Location:		WATER Evaporativ Gross Alph STANDARI TestAmeric	WATER Evaporative Preparation, Dissolved Gross Alpha/Beta EPA 900 STANDARD TEST SET TestAmerica St. Louis	n, Dissolve 900	Pe	
Analyte List Syn Compound 5630 Gross Alpha, Dissolved 5632 Gross Beta, Dissolved	8 8 4	Detection Limits Units MDL pCi/L	n Limits MDL	Units	Run Date 0	<b>∀</b> <b>∀</b> ≻≻ □	C Y C Y Check List 5584 CC C Y 63 CC Y 80	st 6584 LCL U 63 1 80 1	4 L UCL RPD 130 40 120 40	Spike List 6585         LCL UCL RPD T A Amt Units LCL UCL RPD         63 130 40 C Y       63 130 40         80 120 40 C Y       80 120 40	Spike List 6585 Units LCL 63	6585 LCL UCL RP 63 130 40 80 120 40	:L RPD 0 40 0 40

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Structured Analysis Code: I-H6-Y9-01-06 Target Analyte List: All Analytes	I-H6-Y9-01-06 All Analytes						] oo	Matrix: Extraction: Method: QC Program: Location:	WATER Evaporative Preparation, Suspended Gross Alpha/Beta EPA 900 STANDARD TEST SET TestAmerica St. Louis	reparation, Su eta EPA 900 EST SET t. Louis	rspended	
Analyte List		Detection Limits	Limits				Chec	Check List 6582	2	Spike	Spike List 6583	13
Syn Compound	R	Units	MDL	Units	Run Date	T A	Amt Ur	its LC	T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD	A Amt U	nits L(	CL UCL RPD
5629 Gross Alpha, Suspended	ო	pCi/L			0	<b>≻</b>		63	130 40 C Y	<b>&gt;</b>	63	63 130 40
5631 Gross Beta, Suspended	4	pCi/L			0	≻ 0		80	120 40 C Y	>	8	80 120 40

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Structured Analysis Code: I-J6-Y9-01-06 Target Analyte List: All Analytes	<b>I-J6-Y9-01-06</b> All Analytes						ď	Matrix: Extraction: Method: QC Program: Location:		WATER 48 HOUR - EVAPORA Gross Alpha/Beta EPA STANDARD TEST SE TestAmerica St. Louis	WATER 48 HOUR - EVAPORATIVE PREPARATION Gross Alpha/Beta EPA 900 STANDARD TEST SET TestAmerica St. Louis	VE PREP	ARATION
Analyte List		Detection Limits	Limits				Ç	Check List 6501	6501		S	Spike List 6548	5548
Syn Compound	RL	Units	MDL	Units	Run Date	<b>∀</b>	Amt (	T A Amt Units	TCF NCF	RPD T	A Amt	Units	LCL UCL RPD T A Amt Units LCL UCL RPD
4170 Gross Alpha	က	pCi/L			0	≻ C			73 136	136 40 C Y	<b>&gt;</b>		44 150 40
4171 Gross Beta	4	pCi/L			0	≻ ე			73 122 40		۲ ک		66 147 40

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Strı	Structured Analysis Code: I-MH-Y9-01-06 Target Analyte List: All Analytes	I-MH-Y9-01-06 All Analytes						Extr N QC Pr Lo	Matrix: Extraction: Method: QC Program: Location:	WATER 48 hour- E Gross Alph STANDAR TestAmeric	WATER 48 hour- Evaporative Prep Suspended Gross Alpha/Beta EPA 900 STANDARD TEST SET TestAmerica St. Louis	ep Suspen	ded	RD-0403
	Analyte List		Detection Limits	Limits				Check List 6501	.ist 6501		•	Spike List 6548	548	
Syn	Compound	RL	Units	MDL	Units	Run Date	T A	T A Amt Units		ICL RPD	T A Amt	Units	LCL UCL RPD T A Amt Units LCL UCL RPD	
4170	Gross Alpha	2	pCi/L			0	≻ `		73 1	73 136 40 C Y	<b>∠</b> ⊃		44 150 40	
4171	Gross Beta	2	pCi/L			0	≻ C		73 1	122 40	۲ ک		66 147 40	

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Structured Analysis Code: I-MI-Y9-01-06 Target Analyte List: All Analytes	I-MI-Y9-01-06 All Analytes						Matrix: Extraction: Method: QC Program: Location:		WATER 48 hour- Evaporative Prep Dissolved Gross Alpha/Beta EPA 900 STANDARD TEST SET TestAmerica St. Louis	rep Dissolv	pe
Analyte List		Detection Limits	imits				Check List 6501	it 6501		Spike List 6548	6548
Syn Compound	RL	Units	MDL	Units	Run Date	T A A	T A Amt Units	LCL UCL RF	OT A Amt	Units	LCL UCL RPD T A Amt Units LCL UCL RPD
4170 Gross Alpha	2	pCi/L			0	<b>≻</b>		73 136 40	≻ ၁		44 150 40
4171 Gross Beta	2	pCi/L			0	<b>≻</b>		73 122 40	د ≺		66 147 40

Str	Structured Analysis Code: A-FS-Y9-01-06 Target Analyte List: All Analytes	A-FS-Y9-01-(	90					Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind Gross Alpt STANDAR TestAmeri	SOLID Dry, Grind, Thin Layer on Planchet Gross Alpha/Beta EPA 900 STANDARD TEST SET TestAmerica St. Louis	n Planchet 00			
	Analyte List		Detection Limits	n Limits			S	Check List 6501	6501		S	Spike List 6548	5548		
Syn	Compound	RL	Units	MDL	Units	Run Date	T A Amt Units	Units	rcr nc	CL RPD	LCL UCL RPD T A Amt	Units	LCL UCL RPD	. RPD	
4170	4170 Gross Alpha	10	pCi/g			0	≻ O		42 11	42 111 40 C Y	<b>∠</b> ∨		43 123	40	
4171	4171 Gross Beta	10	pCi/g			0	≻ C ≺		38 12	126 40	<b>≻</b>		55 125	40	

Structured Analysis Code: A-HI-Y9-01-06 Target Analyte List: All Analytes	A-HI-Y9-01-0 All Analytes	9(					ð	Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Gross Alpha STANDARD TestAmerica	SOLID Dry, Grind, Total Dissolution Gross Alpha/Beta EPA 900 STANDARD TEST SET TestAmerica St. Louis	ution 900			
Analyte List		Detection Limit	n Limits				Che	Check List 6527	527		•	Spike List 6528	6528		
Syn Compound	R	Units	MDL	Units	Run Date	<b>∀</b>	T A Amt Units		וכר חכ	L RPD	LCL UCL RPD T A Amt Units LCL UCL RPD	Units	LCL U	ICL RPD	
4170 Gross Alpha	10	pCi/g			0	<b>≻</b>			70 130	70 130 40 C Y	> ()		70 1	130 40	
4171 Gross Beta	10	pCi/g			0	≻ C			70 130 40		≻		70 1	130 40	

Structured Analysis Code: I-J6-3Z-01-06 Target Analyte List: All Analytes	I-J6-3Z-01-06 All Analytes						Matrix: Extraction: Method: QC Program: Location:		WATEF 48 HOU Gross A STANDA TestAme	WATER 48 HOUR - EVAPORATIVE PREPARATION Gross A/B by NJ EPA 900 PWTA STANDARD TEST SET TestAmerica St. Louis	PORATIV EPA 900 T SET	E PREP	ARATIC	z.	
Analyte List		<b>Detection Limits</b>	. Limits				Check List 6501	st 6501			Sp	Spike List 6548	6548		
Syn Compound	RL	Units	MDL	Units	Run Date	T A An	T A Amt Units		UCL RP	LCL UCL RPD T A Amt Units LCL UCL RPD	Amt	Units	רכר ח	CL RP	٥
4170 Gross Alpha	7	pCi/L			0	<b>≻</b> O		. 22	136 40 C Y	<b>≻</b>			44	44 150 40	
4171 Gross Beta	2	pCi/L			0	≻ 0		73	73 122 40	ک ن			66 147	47 40	

Structured Analysis Code: I-FR-ZA-DO-06 Target Analyte List: All Analytes	I-FR-ZA-DO-	90					Matrix: Extraction:		WATER Evaporative Preparation, Total GROSS A/B BY GFPC SW846 9310 MOD DOE QSAS TestAmerica St. Louis	paration, Tot: SFPC SW84.	al 6 9310 MO	Q
Analyte List		Detection Limits	n Limits				Check List 6880	ist 6880		Spike	Spike List 6881	
Syn Compound	R	Units	MDL	Units	Run Date	T A A	T A Amt Units		LCL UCL RPD T A Amt Units LCL UCL RPD	Amt Un	its LCL	UCL RPD
4170 Gross Alpha	က	pCi/L			0	<b>≻</b>		75 125 25	25 C Y		9	60 140 25
4171 Gross Beta	4	pCi/L			0	<b>ک</b>		75 125 25	25 C Y		9	140 25

Structured Analysis Code: A-FS-ZA-DO-06	A-FS-ZA-DO	90-						Matrix: Extraction: Method:	· <del>-</del>	SOLID Dry, Grir GROSS	SOLID Dry, Grind, Thin Layer on Planchet GROSS A/B BY GFPC SW846 9310 MOD	ayer on FPC SM	Planche /846 93	it 10 MOE	0
Target Analyte List: All Analytes	All Analytes						o	QC Program: Location:		DOE QSAS TestAmerica	DOE QSAS TestAmerica St. Louis	ouis	:		
Analyte List		Detection Limit	n Limits				Che	Check List 6880	088			Sp	Spike List 6881	6881	
Syn Compound	RL	Units	MDL	Units	Run Date	<b>∢</b> ⊢	T A Amt Units		rcr u	CL RPI	<b>∀</b> ⊥ 0	Amt	Units	רכרו	LCL UCL RPD T A Amt Units LCL UCL RPD
4170 Gross Alpha	10	pCi/g			0	≻ O			75 12	125 25	≻ C				140 25
4171 Gross Beta	10	pCi/g			0	<b>≻</b> ⇔			75 12	125 25	<b>≻</b> O				140 25

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Structured Analysis Code: I-FR-Y9-DO-06 Target Analyte List: All Analytes	I-FR-Y9-DO-	90				:	Ö	Matrix: Extraction: Method: QC Program: Location:		WATER Evaporative Preparation, Total Gross Alpha/Beta EPA 900 DOE QSAS TestAmerica St. Louis	on, Total	
Analyte List		Detection Limits	n Limits				Che	Check List 6880	1880		Spike List 6881	6881
Syn Compound	RL	Units	MDL	Units	Run Date	<b>∀</b>	T A Amt Units		LCL UCL RP	'D T A Ami	Units	LCL UCL RPD T A Amt Units LCL UCL RPD
4170 Gross Alpha	ю	pCi/L			0	≻ C			75 125 25	<b>∠</b> 2		60 140 25
4171 Gross Beta	4	pCi/L			0	≻ C			75 125 25	≻ C		60 140 25

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Structured Analysis Code: A-FS-Y9-DO-06 Target Analyte List: All Analytes	Analysis Code: A-FS-Y9-E Target Analyte List: All Analytes	A-FS-Y9-DO	90-1					ŏ	Matrix: Extraction: Method: QC Program: Location:	1	SOLID Dry, Grind, ' Gross Alpha DOE QSAS TestAmerica	SOLID Dry, Grind, Thin Layer on Planchet Gross Alpha/Beta EPA 900 DOE QSAS TestAmerica St. Louis	ayer on f EPA 900 ouis	Planchet		
Analyte List	ist		Detection Limits	n Limits				Che	Check List 6880	9880			Spi	Spike List 6881	5881	
Syn Compound		RL	Units	MDL	Units	Run Date	٠ ٧ ١	T A Amt Units		רכר ת	il RP(	LCL UCL RPD T A Amt Units	Amt	Units	TCT N	LCL UCL RPD
4170 Gross Alpha		10	pCi/g			0	<b>≻</b>			75 12	125 25	≻ C			60 14	140 25
4171 Gross Beta		10	pCi/g			0	> O			75 12	125 25	≻ C			60 14	140 25

		Location:	Radium 226 by SW846 9315 MOD STANDARD TEST SET TestAmerica St. Louis
Analyte List Detection Limits Syn Compound RL Units MDL Units Run Date 2257 Radium (226) 1 pCi/L 0	Units	C Y C > Check List 6504	6504         Spike List 6550           LCL UCL RPD T A Amt Units LCL UCL RPD           52 150 40 C Y         50 130 40

Structured Analysis Code: 1-G3-ZW-01-06 Target Analyte List: All Analytes	I-G3-ZW-01-0 All Analytes	9					Matrix: Extraction: Method: QC Program: Location:	,	WATER Precipitate, Separation - 14 day Ingrowth Radium 226 by SW846 9315 MOD STANDARD TEST SET TestAmerica St. Louis	ay Ingrowth MOD
Analyte List		<b>Detection Limits</b>	ι Limits				Check List 6504	6504	Spike	Spike List 6550
Syn Compound	RL	Units	MDL	Units	Run Date	T A Ami	T A Amt Units	LCL UCL RPD	T A Amt Un	LCL UCL RPD T A Amt Units LCL UCL RPD
2257 Radium (226)	-	pCi/L			0	<b>≻</b>		52 150 40 C Y	<b>&gt;</b> 0	50 130 40

Structured Analysis Code: I-NR-ZW-01-06 Target Analyte List: All Analytes	I-NR-ZW-01-	90	:			Matrix: Extraction: Method: QC Program: Location:			WATER Precipitate, Separation - 21 day Ingrowth, Dissolved Radium 226 by SW846 9315 MOD STANDARD TEST SET TestAmerica St. Louis	21 day Ing 315 MOD	growth, Dis:	olved
Analyte List Syn Compound	RL	Detection Limits Units MDL	ı Limits MDL	Units	Run Date	Check List 6504 Spike List 6550 T A Amt Units LCL UCL RPD T A Amt Units LCL UCL	ist 6504 LCL 1	UCL RPD	S <sub>F</sub> T A Amt	Spike List 6550 Units LCL	TCT NCT	RPD
5656 Radium (226), Dissolved	<del>-</del>	pCi/L			0	<b>≻</b> 3	25	52 150 40 C Y	<b>≻</b>		50 130 40	40

STL Reference D	erence Data Summary	51-	ST-
	Matrix:	SOLID	RD-
Structured Analysis Code: A-G1-ZW-01-06	Extraction:	Extraction: Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth	-04
Target Analyte List: All Analytes	QC Program:	STANDARD TEST SET	0.3
	Location:	TestAmerica St. Louis	

						     	ocation: TestAme	TestAmerica St. Louis		
Analyte List		Detection Limit	Limits			Check	Check List 6504	S	Spike List 6550	20
Syn Compound	R	Units	MDL	Units	Run Date	T A Amt Units LCLUCL RPD T A Amt Units LCLUCL RPD	S LCL UCL RPD	T A Amt	Units	CL UCL RPD
2257 Radium (226)	-	pCi/g			0	<b>≻</b> ∪	64 130 40 C Y	<b>≻</b>	7	72 140 40

								•		
Structured Analysis Code: A-G5-ZW-01-06 Target Analyte List: All Analytes	A-G5-ZW-01-	90					Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Digest, Precipitate, Ser Radium 226 by SW846 9315 MOD STANDARD TEST SET TestAmerica St. Louis	SOLID Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth Radium 226 by SW846 9315 MOD STANDARD TEST SET TestAmerica St. Louis
Analyte List Syn Compound 2257 Radium (226)	귛 ~	Detection Limits Units MDL	n Limits MDL	Units	Run Date	T A Am	Check List 6504 t Units LCL 64	6504 LCL UCL RPD T A 64 130 40 C Y	Spi	C Y C Y Spike List 6550

Str	Structured Analysis Code: A-RC-ZW Target Analyte List: All Analytes	A-RC-ZW-01-06 All Analytes	90					Matrix: Extraction: Method: QC Program: Location:		SOLID Ra-226, ASTM 3987-85/ SW846 9315 Radium 226 by SW846 9315 MOD STANDARD TEST SET TestAmerica St. Louis	85/ SW846 9 6 9315 MOD :T	315
<b>Syn</b> 2257	Analyte List Syn Compound 2257 Radium (226)	R +	Detection Limits Units MDL pCi/L	n Limits MDL	Units	Run Date	<b>T A An</b>	Check List 6504  T A Amt Units LCL  C Y 64	6504 LCL UCL RPD T A 64 130 40 C Y	PD T A Am	Spike List 6550 t Units LCL 72	Spike List 6550  LCL UCL RPD T A Amt Units LCL UCL RPD  64 130 40 C Y 72 140 40

Structured Analysis Code: I-G2-Z3-01-06 Target Analyte List: All Analytes	I-G2-Z3-01-06 All Analytes						Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation - 21 day Ingrowth Radium 226 by DOE RA-06-RC MOD STANDARD TEST SET TestAmerica St. Louis	- 21 day Ing A-06-RC MO	growth
Analyte List Syn Compound 2257 Radium (226)	R -	Detection Limits Units MDL pCi/L	n Limits MDL	Units	Run Date	<b>→ A</b> Am	C Y C Aeck List 6504	6504 LCL UCL RPD T A 52 150 40 C Y	Amt	Spike List 6550 : Units LCL	5550 LCL UCL RPD 50 130 40

Structured Analysis Code: I-G3-Z3-01-06 Target Analyte List: All Analytes	I-G3-Z3-01-06 All Analytes						Extra M QC Pro Loc	Matrix: Extraction: Method: QC Program: Location:	WATER Precipitate Radium 22 STANDAR TestAmeric	WATER Precipitate, Separation - 14 day Ingrowth Radium 226 by DOE RA-06-RC MOD STANDARD TEST SET TestAmerica St. Louis	- 14 day Ing 4-06-RC MC	growth DD	:
Analyte List Syn Compound 2257 Radium (226)	Ā -	Detection Limits Units MDL pCi/L	n Limits MDL	Units	Run Date	<b>∀</b> ≻	C Y C C Check List 6504	<del>,</del>	UCL RPD T A	T A Amt	Spike List 6550 Units LCL	Spike List 6550  LCL UCL RPD T A Amt Units LCL UCL RPD 52 150 40 C Y 50 130 40	

D-0403	
SOLID Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth Radium 226 by DOE RA-06-RC MOD STANDARD TEST SET TestAmerica St. Louis	L RPD
paration - OD	6550 LCL UCL RP 72 140 40
ipitate, Se 06-RC M	Spike List 6550 t Units LCL 72
SOLID Dry, Grind, Digest, Precipitate, Separa Radium 226 by DOE RA-06-RC MOD STANDARD TEST SET TestAmerica St. Louis	× A Amt
SOLID Dry, Grind, Digest, Prec Radium 226 by DOE R/ STANDARD TEST SET TestAmerica St. Louis	<b>RPD T</b>
	6504 Spike List 6550 LCL UCL RPD T A Amt Units LCL UCL RPD 64 130 40 C Y 72 140 40
Matrix: Extraction: Method: QC Program: Location:	
J	Am
	<b>∢</b> ≻ ⊢ ∪
	Run Date 0
	Units
	MDL
90	Detection Limits Units MDL pCi/g
Structured Analysis Code: A-G1-Z3-01-06 Target Analyte List: All Analytes	ጃ -
Analysis Code: A-G1-Z3-C Target Analyte List: All Analytes	List
uctured Ana Targe	Analyte List Syn Compound 2257 Radium (226)
Strı	Syn 2257

Structured Analysis Code: A-G5-Z3-01-06 Target Analyte List: All Analytes	A-G5-Z3-01-C	90				σ	Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth Radium 226 by DOE RA-06-RC MOD STANDARD TEST SET TestAmerica St. Louis
Analyte List		Detection Limits	n Limits			Che	Check List 6504	.04 S	Spike List 6550
Syn Compound	Ŗ	Units	MDL	Units	Run Date	T A Amt U	Inits L	T A Amt Units LCL UCL RPD T A Amt	Units LCL UCL RPD
2257 Radium (226)	-	pCi/g			0	≻ ′	Ó	64 130 40 C Y	72 140 40

ge number 1

	STL Refe	erence Data Summary	51-
Structured Analysis Code: A-K4-Z3-01-06 Target Analyte List: All Analytes	: <b>A-K4-Z3-01-06</b> All Analytes	Matrix: Extraction: Method: QC Program:	Matrix: SOLID  traction: As Received, Digest, Precipitate, Seperation - 14 Day Ingro O  Method: Radium 226 by DOE RA-06-RC MOD  Program: STANDARD TEST SET  Occasion: TestAmerica St Louis

Analyte List		<b>Detection Limits</b>	Limits				Check	heck List 6504			Spike	Spike List 6550	50
Syn Compound	묎	Units	MDL	Units	Run Date	<b>∀</b> ⊥	Aπt Un	its LCI	T A Amt Units LCL UCL RPD T A Amt	1 A 1	lmt U	its L	Units LCL UCL RPD
2257 Radium (226)	-	pCi/g			0	≻ C		64	64 130 40 C Y	> 0		_	72 140 40

Sti	Structured Analysis Code: I-G2-ZY-01-06 Target Analyte List: All Analytes	I-G2-ZY-01-06 All Analytes						Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation - 21 day Ingrowth Radium 226 by EPA 903.0 MOD STANDARD TEST SET TestAmerica St. Louis	03.0 MOD	growth
<b>Syn</b> 2257	Analyte List Syn Compound 2257 Radium (226)	ר או	Detection Limits Units MDL pCi/L	Limits MDL	Units	Run Date	Check Li TAAmt Units CY	st (	5504 LCL UCL RPD T A 52 150 40 C Y	PD T A Amt	Spike List 6550 . Units LCI 50	Spike List 6550  LCL UCL RPD T A Amt Units LCL UCL RPD  52 150 40 C Y 50 130 40

Structured Analysis Code: I-G3-ZY-01-06 Target Analyte List: All Analytes	I-G3-ZY-01-0	9					Matrix: Extraction: Method: QC Program: Location:		WATER Precipit Radium STAND/	WATER Precipitate, Separation - 14 day Ingrowth Radium 226 by EPA 903.0 MOD STANDARD TEST SET TestAmerica St. Louis	- 14 day ln	growth		
Analyte List Syn Compound 2257 Radium (226)	귛	Detection Limits Units MDL PCI/L	n Limits MDL	Units	Run Date	Check Li	Check List 6504 t Units LCL 52	t 6504 LCL t	504 LCL UCL RPD 52 150 40	A Amt	Spike List 6550 Units LCL	6550 LCL 1 50 .	UCL RPD 130 40	
3030 Radium (220), Dissolved	-	Ę Į			>	- د		<b>7</b> 6	150 40	- د		2	130 40	

		STL Refe	Refe	ren	ce Dat	rence Data Summary	ary		ST-F
Structured Analysis Code: I-P3-ZY-01-06 Target Analyte List: All Analytes	I-P3-ZY-01-06 All Analytes					Extr M QC Pro Lo	Matrix: Extraction: Method: QC Program: Location:	WATER Precipitate, Separation - 14 day Ingrowth, Dissolved Radium 226 by EPA 903.0 MOD STANDARD TEST SET TestAmerica St. Louis	dth, Dissolved
Analyte List		Detection Limits	Limits			Check List 6504	ist 6504	Spike List 6550	
Syn Compound	R	Units	MDL	Units	Run Date	T A Amt Units		LCL UCL RPD T A Amt Units LCL UCL RPD	L UCL RPD
5656 Radium (226), Dissolved	-	pCi/L			0	<b>≻</b> 0	52	52 150 40 C Y 50	50 130 40

Structured Analysis Code: A-G1-ZY-01-06 Target Analyte List: All Analytes	A-G1-ZY-01-	90					Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth Radium 226 by EPA 903.0 MOD STANDARD TEST SET TestAmerica St. Louis
Analyte List		Detection Limits	ι Limits				Check List 6504	6504	Spike List 6550
Syn Compound	RL	Units	MDL	Units	Run Date	T A Amt Units	it Units	LCL UCL RPD T A Amt	t Units LCL UCL RPD
2257 Radium (226)	-	pCi/g			0	<b>≻</b>		64 130 40 C Y	72 140 40

					cuec cara cammary				
Structured Analysis Code: A-G5-ZY-01-06 Target Analyte List: All Analytes	A-G5-ZY-01-(	90					Matrix: Extraction: Method: QC Program: Location:	c. SOLID  1: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth  2: Radium 226 by EPA 903.0 MOD  1: STANDARD TEST SET  1: TestAmerica St. Louis	ation - 21day ingrowth
	ō	Detection Limits	n Limits	- - - -	ote C	Check Li	st (	Spike List 6550	20
2257 Radium (226)	÷ +	pCi/g			0	<b>€</b> ≻		64 130 40 C Y 72	72 140 40

Structi	Structured Analysis Code: A-S7-ZY-01-06 Target Analyte List: All Analytes	A-S7-ZY-01- All Analytes	90					Matrix: Extraction: Method: QC Program: Location:		SOLID As received, Digest, Precipitate, Radium 226 by EPA 903.0 MOD STANDARD TEST SET TestAmerica St. Louis	ecipitate, Se 13.0 MOD	SOLID As received, Digest, Precipitate, Separation 21 day ingrowth Radium 226 by EPA 903.0 MOD STANDARD TEST SET TestAmerica St. Louis
Syn Cor 2257 Ra	Analyte List Compound Radium (226)	R +	Detection Limits Units MDL pCi/g	Limits MDL	Units	Run Date	Check Lis TAAmt Units CY	Check List 6504 t Units LCL 64	6504 LCL UCL RF 64 130 40	UCL RPD T A Amt	Spike List 6550 Units LCL	5504 Spike List 6550 LCL UCL RPD T A Amt Units LCL UCL RPD 64 130 40 C Y 72 140 40

		STL Refe	Refe		rence Data Summary	ia S	E	mar	>				ST-F
Structured Analysis Code: I-G2-ZY-DO-06 Target Analyte List: All Analytes	I-G2-ZY-DO-(	96					00	Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation - 21 day Ingrowth Radium 226 by EPA 903.0 MOD DOE QSAS TestAmerica St. Louis	n - 21 day lı 903.0 MOD	ıgrowth	RD-0403
Analyte List		Detection Limits	Limits				Chec	Check List 6882	12		Spike List 6883	6883	
Syn Compound	RL	Units	MDL	Units	Run Date	, A ⊤	\mt	nits LC	CL UCL RF	'D T A Am	t Units	T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD	
2257 Radium (226)	•	pCi/L			0	≻ O		75	75 125 25 C Y	≻ `		60 140 25	

		SIL Kere			rence Data Summary	5	5	·
Structured Analysis Code: A-G5-ZY-DO-06 Target Analyte List: All Analytes	A-G5-ZY-DO All Analytes	90-					Matrix: Extraction:	SOLID  By, Grind, Digest, Precipitate, Separation - 21day ingrowth Radium 226 by EPA 903.0 MOD DOE QSAS TestAmerica St. Louis
Analyte List Svn Compound	RL	Detection Limits Units MDL	n Limits MDL	Units	Run Date	Check List	Check List 6882 t Units LCL	Spike List 6883 LCL UCL RPD T A Amt Units LCL UCL RPD
	-	pCi/g			0	<b>≻</b>	12	75 125 25 C Y 60 140 25

Structured Analysis Code: I-G2-Z3-DO-06 Target Analyte List: All Analytes	I-G2-Z3-DO-0 All Analytes	9				Extr M QC Prr	Matrix: Extraction: Method: QC Program: Location:	WATER Precipitate, Separation - 21 day Ingrowth Radium 226 by DOE RA-06-RC MOD DOE QSAS TestAmerica St. Louis	21 day Ingr -06-RC MOI	owth C
Analyte List		Detection Limits	n Limits			Check List 6882	ist 6882	Ś	Spike List 6883	383
Syn Compound	RL	Units	MDL	Units	Run Date	T A Amt Units	רכרו	T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD	Units	LCL UCL RPD
2257 Radium (226)	-	PC!/L			0	C ≺	75	75 125 25 C Y	_	60 140 25

		STL Rete	Kete	ren	ce Da	rence Data Summary	T-R
Structured Analysis Code: A-G5-Z3-DO-06 Target Analyte List: All Analytes	A-G5-Z3-DO.	90				Matrix: Extraction: Method: QC Program: Location:	Matrix: SOLID  Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth Defended: Radium 226 by DOE RA-06-RC MOD  Program: DOE QSAS  Location: TestAmerica St. Louis
Analyte List Syn Compound 2257 Radium (226)	RL 1	Detection Limits Units MDL pCi/g	n Limits MDL	Units	Run Date 0	Check List 6882  TAAmt Units LCL	C Y C Y Spike List 6883 Spike List 6883 C Y Spike List 6883

Structured Analysis Code: 1-G2-ZW-DO-06 Target Analyte List: All Analytes	I-G2-ZW-DO-	90					Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation - 21 day Ingrowth Radium 226 by SW846 9315 MOD DOE QSAS TestAmerica St. Louis	21 day Ing 3 9315 MOD	growth
Analyte List Syn Compound 2257 Radium (226)	RL 1	Detection Limits Units MDL pCi/L	Limits MDL	Units	Run Date	T A Am	C Y C Y Check List 6882	6882 LCL UCL 75 125	1882 LCL UCL RPD T A Amt 75 125 25 C Y	Spike List 6883 Units LCL	8882 Spike List 6883 LCL UCL RPD T A Amt Units LCL UCL RPD 75 125 25 C Y 60 140 25

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Structured Analysis Code: A-G5-ZW-DO-06 Target Analyte List: All Analytes	A-G5-ZW-DC	90-(				-	Matrix: Extraction: Method: QC Program: Location:	SOLID Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth Radium 226 by SW846 9315 MOD DOE QSAS TestAmerica St. Louis	Precipitate, Separa 846 9315 MOD iis	ion - 21day ingrowth
Analyte List		Detection Limits	n Limits			๋	Check List 6882	2	Spike List 6883	
Syn Compound	RL	Units	MDL	Units	Run Date	T A Amt Units	Units LC	LCL UCL RPD T A Amt Units LCL UCL RPD	mt Units LC	UCL RPD
2257 Radium (226)	-	pCi/g			0	<b>≻</b>	75	75 125 25 C Y	09	60 140 25

Target Analyte List: All Analytes	Structured Analysis Code: I-G2-Z5-01-06 Target Analyte List: All Analytes						Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation - 21 day Ingrowth Radium 228 by GFPC SW846 9320 MOD STANDARD TEST SET TestAmerica St. Louis	ttion - 21 day PC SW846 9 SET uis	Ingrowth 320 MOD	
Analyte List		Detection Limits	imits.				Check List 6505	1 6505		Spike List 6551	st 6551	
Syn Compound	R	Units	MDL	Units	Run Date	T A A	T A Amt Units		LCL UCL RPD T A Amt Units LCL UCL RPD	\mt Units	י רכר חכ	L RPD
2259 Radium 228	_	pCi/L			0	≻ C		64 14	64 140 40 C Y		64 150 40	0 40
4152 Ra-228	_	pCi/L			0							

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Structured Analysis Code: I-G3-Z5-01-06 Target Analyte List: All Analytes	I-G3-Z5-01-06 All Analytes						ac i	Matrix: Extraction: Method: QC Program: Location:	WATER Precipitate, Separation - 14 day Ingrowth Radium 228 by GFPC SW846 9320 MOD STANDARD TEST SET TestAmerica St. Louis	- 14 day Ingrowth W846 9320 MOD	D-0403
Analyte List Syn Compound 2259 Radium 228	RL 1	Detection Limits Units MDL pCi/L	Limits MDL	Units	Run Date	<b>∀</b> ≻	Check Amt Uni	Check List 6505 t Units LCL 64	505 LCLUCL RPD T A Amt 64 140 40 C Y	Check List 6505  T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD  C Y 64 140 40 C Y 64 150 40	

Structured Analysis Code: I-NR-Z5-01-06 Target Analyte List: All Analytes	I-NR-Z5-01-06 All Analytes					Matrix: Extraction: Method: QC Program: Location:	Matrix: WATER action: Precipitate, Separation - 21 day Ingrowth, Dissolved lethod: Radium 228 by GFPC SW846 9320 MOD ogram: STANDARD TEST SET cation: TestAmerica St. Louis
Analyte List		Detection Limits	ı Limits			Check List 6505	5505 Spike List 6551
Syn Compound	귙	Units	MDL	Units	Run Date	T A Amt Units	LCL UCL RPD T A Amt Units LCL UCL RPD
5657 Radium 228, Dissolved	-	pCi/L			0	<b>≻</b> 0	64 140 40 C Y 64 150 40

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Structured Analysis Code: A-G1-Z5-01-06 Target Analyte List: All Analytes	<b>A-G1-Z5-01-(</b> All Analytes	90				Matrix: Extraction: Method: QC Program: Location:	Matrix: SOLID  Extraction: Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth  Method: Radium 228 by GFPC SW846 9320 MOD  Program: STANDARD TEST SET  Location: TestAmerica St. Louis	D-0403 €
Analyte List		Detection Limits	Limits			Check List 6505	. 6505 Spike List 6551	
Syn Compound	RL	Units	MDL	Units	Run Date	T A Amt Units	LCL UCL RPD T A Amt Units LCL UCL RPD	
2259 Radium 228	-	pCi/g			0	۲ ک	57 140 40 C Y 50 150 40	

					ience Data Cammaiy	) 3		, m				
Structured Analysis Code: A-G5-Z5-01-06 Target Analyte List: All Analytes	A-G5-Z5-01-06 All Analytes						Ex OC P	Matrix: Extraction: Method: QC Program: Location:	_ ' '	SOLID Dry, Grind, Digest, Precipitate, Separatior Radium 228 by GFPC SW846 9320 MOD STANDARD TEST SET TestAmerica St. Louis	e, Separal 6 9320 MC	SOLID Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth Radium 228 by GFPC SW846 9320 MOD STANDARD TEST SET TestAmerica St. Louis
Analyte List		Detection Limits	Limits				Check	Check List 6505		Spike	Spike List 6551	
Syn Compound	RL	Units	MDL	Units	Run Date	, ▲ T	T A Amt Units		. UCL RPD T	LCL UCL RPD T A Amt Units LCL UCL RPD	nits LC	L UCL RPD
2259 Radium 228	-	pCi/g			0	≻ C		22	140 40 C Y	<b>&gt;</b>	50	50 150 40

Structured Analysis Code: A-RD-Z5-01-06 Target Analyte List: All Analytes	A-RD-Z5-01-0	90					Matrix: Extraction: Method: QC Program: Location:		SOLID Ra-228, AS Radium 22( STANDARI	SOLID Ra-228, ASTM 3987-95/ SW846 9320 Radium 228 by GFPC SW846 9320 MOD STANDARD TEST SET TestAmerica St. Louis	/ SW846 9; W846 932C	320 MOD	
Analyte List Syn Compound	R	Detection Limits Units MDL	ו Limits MDL	Units	Run Date	Check Li T A Amt Units	Check List 6505 rt Units LCL	16505 LCL UC	K RPD	Amt	Spike List 6551 Units LCL	issa LCL UCL F	RPD
2259 Radium 228	-	pCi/L			0	<b>≻</b>		57 14	140 40 C Y	≻ o		50 150 40	0

Structured Analysis Code: I-G2-Z6-01-06 Target Analyte List: All Analytes	<b>I-G2-Z6-01-06</b> All Analytes						OC P. L	Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation - 21 day Ingrowth Radium 228 by GFPC DOE RA-06-RC Mod STANDARD TEST SET TestAmerica St. Louis	DOE RA-06	ngrowth 3-RC Mod	
Analyte List		Detection Limits	ı Limits				Check	Check List 6505			Spike List 6551	6551	
Syn Compound	RL	Units	MDL	Units	Run Date	T A Ar	T A Amt Units		. UCL RPI	LCL UCL RPD T A Amt Units LCL UCL RPD	Units	TCL UCI	. RPD
2259 Radium 228	~	pCi/L			0	<b>≻</b>		64	64 140 40 C Y	<b>≻</b> 0		64 150 40	40
4152 Ra-228	•	pCI/L			0								

Structured Analysis Code: I-G3-Z6-01-06 Target Analyte List: All Analytes	I-G3-Z6-01-0 All Analytes	9					Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation Radium 228 by GFPC   STANDARD TEST SE TestAmerica St. Louis	WATER Precipitate, Separation - 14 day Ingrowth Radium 228 by GFPC DOE RA-06-RC Mod STANDARD TEST SET TestAmerica St. Louis	14 day Ing DE RA-06-	Irowth RC Mod	
Analyte List Syn Compound	RL	Detection Limits Units MDL	n Limits MDL	Units	Run Date	Check Lis T A Amt Units	Check List 6505 t Units LCL	6505 LCL UCI	, RPD T	Ami	Spike List 6551 t Units LCL	iss1 LCL UCL	RPD
2259 Radium 228	-	pCi/L			0	<b>≻</b> ∪		64 140	64 140 40 C Y	<b>&gt;</b>		64 150 40	40

Structured Analysis Code: A-G1-Z6-01-06 Target Analyte List: All Analytes	A-G1-Z6-01-	90					Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth Radium 228 by GFPC DOE RA-06-RC Mod STANDARD TEST SET TestAmerica St. Louis	Precipitate, Se PC DOE RA-00 SET uls	eparation - 14 6-RC Mod	lay ingrowth
Analyte List		Detection Limits	n Limits				Check List 6505	505		Spike List 6551	t 6551	
Syn Compound	RL	Units	MDL	Units	Run Date	T A Amt Units		רכר חכ	LCL UCL RPD T A Amt Units LCL UCL RPD	mt Units	רכר חכר ו	(PD
2259 Radium 228	_	pCi/g			0	<b>≻</b>		57 14(	140 40 C Y		50 150 40	0

Structured Analysis Code: A-G5-Z6-01-06 Target Analyte List: All Analytes	5- <b>Z6-01-06</b>					Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Digest, Precipitate, Separation - Radium 228 by GFPC DOE RA-06-RC Mod STANDARD TEST SET TestAmerica St. Louis	SOLID Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth Radium 228 by GFPC DOE RA-06-RC Mod STANDARD TEST SET TestAmerica St. Louis
Analyte List		<b>Detection Limits</b>	Limits			Check List 6505	t 6505	Spike	Spike List 6551
Syn Compound	RL	Units	MDL	Units	Run Date	T A Amt Units	LCL UCL RPD	LCL UCL RPD T A Amt Units	ts LCL UCL RPD
2259 Radium 228	-	pCi/g			0	<b>≻</b> ∪	57 140 40 C Y	<b>≻</b> ∨	50 150 40

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Structured Analysis Code: A-K4-Z6-01-06 Target Analyte List: All Analytes	<b>A-K4-Z6-01-0</b> All Analytes	9				Extra  Mc  QC Pro  Loc	Matrix: SOLID Extraction: As Rec Method: Radium QC Program: STAND Location: TestAm	SOLID As Received, Digest, Precipitate, Seperation - 14 Day Ingro Radium 228 by GFPC DOE RA-06-RC Mod STANDARD TEST SET TestAmerica St. Louis	on - 14 Day Ingro 0 - 1
Analyte List Syn Compound	RL	Detection Limits Units MDL	Limits	Units	Run Date	Check List 6505 T A Amt Units LCL	st 6505 LCL	Check List 6505  T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD	CL RPD
2259 Radium 228	<del></del>	pCi/g			0	د ≺	22	57 140 40 C Y 50 150 40	50 40

ST-RD-0403

Structured Analysis Code: 1-G2-Z4-01-06 Target Analyte List: All Analytes	<b>I-G2-Z4-01-0</b> All Analytes	စ္					Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation - 21 day Ingrowth Radium 228 by GFPC EPA 904 MOD STANDARD TEST SET TestAmerica St. Louis	eparation - y GFPC EF 'EST SET it. Louis	21 day Ing วA 904 MC	growth 3D	
Analyte List		Detection Limits	n Limits			J	Check List 6505	6505		ซี	Spike List 6551	3551	
Syn Compound	RL	Units	MDL	Units	Run Date	T A Amt Units	Units	רכר חכו	LCL UCL RPD T A Amt Units LCL UCL RPD	A Amt	Units	רכר חכר	. RPD
2259 Radium 228	-	pCi/L			0	> 0		64 140	140 40 C Y	>		64 150	40
4152 Ra-228	-	pCi/L			0								

								1				
Structured Analysis Code: I-G3-Z4-01-06 Target Analyte List: All Analytes	I-G3-Z4-01-06 All Analytes						Ex OC F	Matrix: Extraction: Method: QC Program: Location:	WATER Precipitate, Separation - 14 day Ingrowth Radium 228 by GFPC EPA 904 MOD STANDARD TEST SET TestAmerica St. Louis	paration - 14 c GFPC EPA 9 EST SET t. Louis	lay Ingrowth 04 MOD	
Analyte List		Detection Limits	Limits				Check	Check List 6505		Spike	Spike List 6551	
Syn Compound	RL	Units	MDL	Units	Run Date	۲ ۲	T A Amt Units		LCL UCL RPD T A Amt Units LCL UCL RPD	A Amt Ur	its LCL UC	RPD
2259 Radium 228	-	pCi/L			0	<b>≻</b> 0		64	140 40 C Y	>-	64 150 40	40
5657 Radium 228, Dissolved	-	pCi/L			0	≻ C		64	140 40 C Y	<b>&gt;</b>	64 150	40

Str	Structured Analysis Code: I-P3-Z4-01-06 Target Analyte List: All Analytes	I-P3-Z4-01-06 All Analytes		į				o	Matrix: Extraction: Method: QC Program: Location:	·	WATER Precipitate, Separation Radium 228 by GFPC E STANDARD TEST SET TestAmerica St. Louis	WATER Precipitate, Separation - 14 day Ingrowth, Dissolved Radium 228 by GFPC EPA 904 MOD STANDARD TEST SET TestAmerica St. Louis	14 day Ing 2A 904 MC	growth, D OD	ssolved
<b>Syn C</b> 5657 F	Analyte List Compound Radium 228, Dissolved	RL +	Detection Limits Units MDL pCI/L	Limits	Units	Run Date	<b>∀</b> ≻	Check Lis TAAmtUnits CY	<b>.</b>	05 CL UCL   4 140 4	505 LCL UCL RPD T A 64 140 40 C Y	Ami	Spike List 6551 t Units LCI 64	6551 LCL UCL RP 64 150 40	L RPD

		STL Refe	Refe	renc	rence Data Summary	ta Sui	nma	ry
Structured Analysis Code: A-G1-Z4-01-06 Target Analyte List: All Analytes	<b>A-G1-Z4-01-0</b> All Analytes	91					Matrix: Extraction: Method: QC Program: Location:	Matrix: SOLID action: Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth fethod: Radium 228 by GFPC EPA 904 MOD ogram: STANDARD TEST SET cation: TestAmerica St. Louis
Analyte List Syn Compound	RL	Detection Limits Units MDL	Limits MDL	Units	Run Date	T A Amt	Check List 6505 Units LCL	Check List 6505  T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD

50 150 40

ک د

57 140 40

≻ C

0

pCi/g

2259 Radium 228

Structured Analysis Code: A-G5-Z4-01-06 Target Analyte List: All Analytes	A-G5-Z4-01-0	90				Ö	Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth Radium 228 by GFPC EPA 904 MOD STANDARD TEST SET TestAmerica St. Louis
Analyte List Syn Compound 2259 Radium 228	R +	Detection Limits Units MDL pCi/g	Limits MDL	Units	Run Date	Check List 6 TAAmt Units CY	Check List 6505 t Units LCL 57	UCL RPD T A Amt	Spike List 6551 Units LCL UCL RPD 50 150 40

					lence Data Summary			•	
Structured Analysis Code: A-S7-Z4-01-06 Target Analyte List: All Analytes	: <b>A-S7-Z4-01-0</b> All Analytes	9(					Matrix: Extraction: Method: QC Program: Location:		SOLID As received, Digest, Precipitate, Separation 21 day ingrowth Radium 228 by GFPC EPA 904 MOD STANDARD TEST SET TestAmerica St. Louis
Analyte List Syn Compound 2259 Radium 228	R +	Detection Limits Units MDL pCi/g	n Limits MDL	Units	Run Date	7 A Amt	Check List 6505 t Units LCL 57	UCL RPD T A Amt	Spike List 6551 Units LCL UCL RPD 50 150 40

Structured Analysis Code: I-G2-Z4-DO-06 Target Analyte List: All Analytes	3: I-G2-Z4-DO. :: All Analytes	90					AC EX	Matrix: Extraction: Method: QC Program: Location:	WATER Precipitate, Separation - 21 day Ingrowth Radium 228 by GFPC EPA 904 MOD DOE QSAS TestAmerica St. Louis	eparation - y GFPC EF St. Louis	21 day Ing A 904 MC	yrowth ND	
Analyte List Syn Compound 2259 Radium 228	. R	Detectio Units PCi/L	Detection Limits Units MDL	Units	Run Date	<b>∢</b> ≻ <b>⊢</b> ○	Check List T A Amt Units C Y	Ξ.	884 LCL UCL RPD T A 75 125 25 C Y	S A Amt	Spike List 6885 Units LCL	\$884 Spike List 6885 LCL UCL RPD T A Amt Units LCL UCL RPD 75 125 25 C Y 60 140 25	
	<b></b>	pCi/L			0				•			} }	

	SIL Reference Data Summary	ımary	
Structured Analysis Code: A-G5-Z4-DO-06 Target Analyte List: All Analytes		Matrix: SOLID Extraction: Dry, Gr Method: Radium QC Program: DOE Q? Location: TestAm	Matrix: SOLID xtraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth Method: Radium 228 by GFPC EPA 904 MOD Program: DOE QSAS Location: TestAmerica St. Louis

Analyte List Syn Compound RL Units MDL Units Run Date T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD 2259 Radlum 228 1 pCl/g 0 C Y 75 125 25 C Y 60 140 25							Loc	Location: TestAmerica St. Louis	
Compound RL Units MDL Units Run Date T A Amt Radium 228 1 pCl/g 0 C Y	Analyte List		Detection	n Limits			Check Li	st 6884	Spike List 6885
Radium 228 125 25 C Y 60 140		RL	Units	MDL	Units	Run Date	T A Amt Units	LCL UCL RPD T A Am	t Units LCL UCL RPD
	2259 Radium 228	<del></del>	pCi/g			0	۲ ک	75 125 25 C Y	60 140 25

Structured Analysis Code: I-G2-Z6-DO-06	I-G2-Z6-DO-	90				:	Extra	Matrix: Extraction:	WATER Precipitat	WATER Precipitate, Separation - 21 day Ingrowth	- 21 day In	ngrowth		
Target Analyte List: All Analytes	All Analytes						QC Program: Location:		DOE QSAS TestAmerica	Nadidili 220 by Of POR DOE QSAS TestAmerica St. Louis	200			
Analyte List		Detection Limits	n Limits				Check List 6505	st 6505			Spike List 6551	6551		
Syn Compound	RL	Units	MDL	Units	Run Date	T A A	nt Units	רכר ו	JCL RPD	T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD	Units	רכר חנ	L RPD	
2259 Radium 228	-	PCI/L			0	<b>≻</b>		64	64 140 40	<b>≻</b>		64 15	150 40	
4152 Ra-228	-	DCi/L			0									

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Structured Analysis Code: A-G5-Z6-DO-06 Target Analyte List: All Analytes	A-G5-Z6-DO. All Analytes	90-				00	Matrix: Extraction: Method: QC Program: Location:	SOLID Dry, Grind, Radium 228 DOE QSAS TestAmerica	SOLID Dry, Grind, Digest, Preci Radium 228 by GFPC D DOE QSAS TestAmerica St. Louis	pitate, Ser	SOLID Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth Radium 228 by GFPC DOE RA-06-RC Mod DOE QSAS TestAmerica St. Louis
Analyte List		Detection	<b>Detection Limits</b>			Chec	Check List 6505		S	Spike List 6551	5551
Syn Compound	R	Units	MDL	Units	Run Date	T A Amt Units		UCL RPD	T A Amt	Units	LCL UCL RPD T A Amt Units LCL UCL RPD
2259 Radium 228	-	pCi/g			0	۲ ک	25	57 140 40 C Y	<b>∠</b> ⊃		50 150 40

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Str	Structured Analysis Code: I-G2-Z5-DO-06 Target Analyte List: All Analytes	I-G2-Z5-DO- All Analytes	90					Extr QC Pro Lo	Matrix: Extraction: Method: QC Program: Location:	WATER Precipitate, Radium 228 DOE QSAS TestAmerics	WATER Precipitate, Separation - 21 day Ingrowth Radium 228 by GFPC SW846 9320 MOD DOE QSAS TestAmerica St. Louis	- 21 day lı SW846 93;	ngrowth 20 MOD	
Syn G 2259 R 4152 R	Analyte List Syn Compound 2259 Radium 228 4152 Ra-228	R + +	Detection Limit Units MD pCi/L pCi/L	n Limits MDL	Units	Run Date 0	<b>ĕ</b> <b>∀</b> ≻ ⊢ ∪	C Y C Check List 6505	4	505 LCLUCL RPD T A 64 140 40 C Y	<b>T A A</b> mi C Y	Spike List 6551 Units LCI	Spike List 6551   LCL UCL RPD T A Amt Units LCL UCL RPD 64 140 40 C Y 64 150 40	PD (

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Structured Analysis Code: A-G5-Z5-DO-06 Target Analyte List: All Analytes	A-G5-Z5-DO-0	91				, σ	Matrix: Extraction: Method: QC Program: Location:	x: SOLID n: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth d: Radium 228 by GFPC SW846 9320 MOD n: DOE QSAS n: TestAmerica St. Louis
Analyte List Syn Compound	RL	Detection Limits Units MDL	Limits	Units	Run Date	Ch <sub>r</sub>	Check List 6505 t Units LCL	Check List 6505  T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD

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57 140 40

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pCi/g

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2259 Radium 228 Syn Compound

50 150 40

Structured Analysis Code: I-LP-2Z-01-06 Target Analyte List: All Analytes	de: I-LP-2Z-01-0 ist: All Analytes	9					Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation Total Alpha Radium EPA 903.0 MOD STANDARD TEST SET TestAmerica St. Louis	ion EPA 903.0 M( SET is	ОО
Analyte List Syn Compound 2261 Radium (total)	Ā -	Detectio Units pCi/L	Detection Limits Units MDL pCi/L	Units	Run Date	<b>V</b> > 0	Check List 6503 mt Units LCL	t 6503 LCL U	C heck List 6503  Spike List 6596  T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD  C Y 72 125 40 C Y 70 130 40	Spike List 6596 mt Units LCL 70	6596 LCL UCL RPD 70 130 40

SOLID	Dry, Grind, Precipitate, Separation	Total Alpha Radium EPA 903.0 MOD	STANDARD TEST SET	TestAmerica St. Louis
Matrix: SOLID	Extraction:	Method:	QC Program:	Location:
	A-I O-27-01-06		All Analytes	
	tructured Analysis Code: A-I O-27-01-06		Target Analyte List: All Analytes	
	Struct	;		

Analyte List		Detection Lin	Limits				Che	Check List 6503	5503	S	Spike List 6596	6596	
Syn Compound	R	Units	MDL	Units	Run Date	<b>∀</b>	\mt	nits	T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD	A Amt	Units	LCL UCL RPD	
2261 Radium (total)	<del>-</del>	pCi/g			0	≻ C			52 114 40 C Y	<b>&gt;</b>		70 130 40	

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Structured Analysis Code: A-L1-2Z-01-06 Target Analyte List: All Analytes	A-L1-2Z-01-06 All Analytes					ο	Matrix: Extraction: Method: QC Program: Location:	SOLID As Received, Precipitate, Separation Total Alpha Radium EPA 903.0 MOD STANDARD TEST SET TestAmerica St. Louis	sparation 3.0 MOD
Analyte List		Detection Limits	ı Limits			Che	Check List 6503		Spike List 6596
Syn Compound	R	Units	MDL	Units	Run Date	T A Amt Units		LCL UCL RPD T A Amt Uni	Units LCL UCL RPD
2261 Radium (total)	<del>-</del>	pCi/g			0	≻	52	52 114 40 C Y	70 130 40

ge number 1

Analyte List Detection Limits Check List 6890 Spike List 6891 Syn Compound RL Units MDL Units Run Date T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD 2261 Radium (total) 1 pCl/L 6891	Structured Analysis Code: I-LP-20-DO-06 Target Analyte List: All Analytes	Code: yte List:	Analysis Code: I-LP-20-DO-C	90					ŏ	Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, 3 Total Radium DOE QSAS TestAmerica	WATER Precipitate, Separation Total Radium by DOE RA-06-RC DOE QSAS TestAmerica St. Louis	A-06-RC		
	Analyte List Syn Compound 2261 Radium (total)		R -	Detection Units pCi/L	n Limits MDL	Units	Run Date	<b>∀</b> ≻ <b>⊢</b> O	Che Amt U		890 .cL UCL 75 125	<b>RPD 1</b>	. A Amt	pike List Units	6891 LCL UCL RPD 60 140 25	

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Structured Analysis Code: A-LQ-20-DO-06 Target Analyte List: All Analytes	A-LQ-20-DO-	90-				Matrix: Extraction: Method: QC Program: Location:	SOLID Dry, Grind, Precipitate, Separation Total Radium by DOE RA-06-RC DOE QSAS TestAmerica St. Louis	RD-0403
Analyte List	i	Detection Limits	Limits	:		Check List 6890	190 Spike List 6891	
Syn Compound	R.	Onits	MDL	Units	Kun Date	T A Amt Units L	T A AMT UNITS LCLUCL RPD T A AMT UNITS LCLUCL RPD	
2261 Radium (total)	-	pCi/g			0	C Y 7	75 125 25 C Y 60 140 25	

		STL Refe	Refe		ce Da	rence Data Summary	ary	ST-R
Structured Analysis Code: I-FX-0K-01-06 Target Analyte List: All Analytes	I-FX-0K-01-06 All Analytes					Matrix: Extraction: Method: QC Program: Location:	Matrix: WATER Extraction: Precipitate, Separation Method: SR-89 BY GFPC EPA 905.0 MOD Program: STANDARD TEST SET Location: TestAmerica St. Louis	2D-0403
Analyte List Syn Compound 4109 Strontium 89	<b>8</b> 8	Detection Limits Units MDL pCi/L	Limits MDL	Units	Run Date	C Y C Y Check List 6524	1 6524 Spike List 6516 LCL UCL RPD T A Amt Units LCL UCL RPD 70 130 40 C Y 70 130 40	st 6516 : LCL UCL RPD 70 130 40

alysis Code: A-FW-0K-01-06 et Analyte List: All Analytes List RL										
List Detection Limits  RL Units MDL Units Run Date  3 pCi/q 0	Structured Analysis Code: Target Analyte List:	A-FW-0K-01-0 All Analytes	91			i	Extrac Extrac Met QC Prog	1	OLID ry, Grind, Digest, Precipitate, Se R-89 BY GFPC EPA 905.0 MOD TANDARD TEST SET sstAmerica St. Louis	eparation 5
RL Units MDL Units Run Date	Analyte List		Detection	Limits			Check Lis	t 6524	Spike List	t 6516
3 pCi/a 0 C Y	Syn Compound	RL	Units	MDL	Units	Run Date	T A Amt Units	רכר חכ	L RPD T A Amt Units	LCL UCL RPD
	4109 Strontium 89	က	pCi/g			0	≻ ∵	70 13	0 40 C Y	70 130 40

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Structured Analysis Code: I-FX-0J-01-06 Target Analyte List: All Analytes	I-FX-0J-01-06 All Analytes			ł		Matrix: Extraction: Method: QC Program: Location:	rix: WATER on: Precipitate, Separation od: SR-89 BY GFPC DOE SR-01-RC MOD im: STANDARD TEST SET on: TestAmerica St. Louis	МОВ
Analyte List Syn Compound 4109 Strontium 89	3 <b>R</b>	Detection Limits Units MDL pCi/L	Limits MDL	Units	Run Date	C Y Check List 6524	C Y C Y Spike List 6524 Spike List 6516  Spike List 6516  Spike List 6516  Spike List 6516  Character Spike List 6516  Spike List 6516  Spike List 6516  Spike List 6516	6516 LCL UCL RPD 70 130 40

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Structured Analysis Code: A-FW-0J-01-06 Target Analyte List: All Analytes	A-FW-0J-01-	90				Matrix: Extraction: Method: QC Program: Location:	Matrix: SOLID  traction: Dry, Grind, Digest, Precipitate, Separation Method: SR-89 BY GFPC DOE SR-01-RC MOD  Program: STANDARD TEST SET  cocation: TestAmerica St. Louis	D-0403
Analyte List Syn Compound 4109 Strontium 89	3 2	Detection Limits Units MDL pCi/g	Limits MDL	Units	Run Date	C Y C 7	Spike List 6 . UCL RPD T A Amt Units 130 40 C Y	516 LCL UCL RPD 70 130 40

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Structured Analysis Code: A-K3-0J-01-06 Target Analyte List: All Analytes	A-K3-0J-01-0	90			* ************************************	Matrix: Extraction: Method: QC Program: Location:	Matrix: SOLID action: As Received, Digest, Precipitate, Seperation fethod: SR-89 BY GFPC DOE SR-01-RC MOD ogram: STANDARD TEST SET cation: TestAmerica St. Louis
Analyte List Syn Compound 4109 Strontium 89	RL 3	Detection Limits Units MDL pCi/g	Limits MDL	Units	Run Date	Check List 6524  T A Amt Units LCL  C Y 70	6524 Spike List 6516  LCL UCL RPD T A Amt Units LCL UCL RPD 70 130 40 C Y 70 130 40
4109 Strontium 89	က	pCi/g			0	C Y	70 130 40 C Y

	<i>,</i>	SIL Kete	Kere		lence Data Summary	ב כ		<b>&gt;</b>		
Structured Analysis Code: I-FX-0J-DO-06 Target Analyte List: All Analytes	I-FX-0J-DO-06 All Analytes						Matrix: Extraction: Method: QC Program: Location:	1	WATER Precipitate, Separation SR-89 BY GFPC DOE S DOE QSAS TestAmerica St. Louis	WATER Precipitate, Separation SR-89 BY GFPC DOE SR-01-RC MOD DOE QSAS TestAmerica St. Louis
Analyte List		Detection Limits	Limits				Check List 6886	6886		Spike List 6887
Syn Compound	RL	Units	MDL	Units	Run Date	T A An	T A Amt Units	LCL UCL RPI	J T A Amt	LCL UCL RPD T A Amt Units LCL UCL RPD
4109 Strontium 89	ო	pCi/L			0	<b>≻</b>		75 125 25 C Y	ر د	60 140 25

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	N	Matrix: WATER
Structured Analysis Code: LEY-01-DO-06		Extraction: Precipitate, Separation
ou actual dilarysis code.		Method: SR-89 BY GFPC DOE SR-01-RC MOD
Target Analyte List: All Analytes	All Analytes QC Program:	ram: DOE QSAS
	Poc	Location: TestAmerica St. Louis

Analyte List		Detection Limits	Limits				Check	Sheck List 6886	S	Spike List 6887	5887
Syn Compound	R	Units	MDL	Units	Run Date	T A Amt	t Units	LCL UCL RPD T A Amt	T A Amt	Units	Units LCL UCL RPD
4109 Strontium 89	ю	pCi/L			0	<b>≻</b>		75 125 25 C Y	<b>≻</b>		60 140 25

	STL Reference Data Summary	ummary		ST-F
Structured Analysis Code: A-FW-0J-DO-06 Target Analyte List: All Analytes	A-FW-0J-DO-06 All Analytes	Matrix: Extraction: Method: \$ QC Program: [ Location: 7	SOLID Dry, Grind, Digest, Precipitate, Separation SR-89 BY GFPC DOE SR-01-RC MOD DOE QSAS TestAmerica St. Louis	RD-0403

Analyte List		Detection Limits	Limits				S	heck List 6886	6886			υ,	Spike List 6887	t 6887	
Syn Compound	꿉	Units MI	MDL	Units	Run Date	¥ +	4mt	Units	רכר ח	CL RP	-	A Amt	Units	T A Amt Units LCLUCL RPD T A Amt Units LCLUCL RPD	CL RPD
4109 Strontium 89	ဗ	pCi/g			0	≻ C			75 125 25 C Y	25 25	ပ	<b>&gt;</b>		60 140	0 25

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Structured Analysis Code: I-FX-0K-DO-06 Target Analyte List: All Analytes	I-FX-0K-DO-06 All Analytes					Matrix: Extraction: Method: OC Program: Location:	Matrix: WATER xtraction: Precipitate, Separation Method: SR-89 BY GFPC EPA 905.0 MOD Program: DOE QSAS Louis Location: TestAmerica St. Louis	р мор	RD-0403
Analyte List Syn Compound	RL	Detection Limits Units MDL	Limits MDL	Units	Run Date	Check List 6886 T A Amt Units LCL	UCL RPD T A Amt	Spike List 6887 Units LCL UCL RPD	
109 Strontium 89	က	pCi/L			0		75 125 25 C Y	60 140 25	

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Str	Structured Analysis Code: A-FW-0K-DO-06 Target Analyte List: All Analytes	A-FW-0K-DO	90-0		·			Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Digest, Precipitate, Separation SR-89 BY GFPC EPA 905.0 MOD DOE QSAS TestAmerica St. Louis	ipitate, Separati 05.0 MOD	uoj
<b>Syn</b> 4109	Analyte List Syn Compound 4109 Strontium 89	3 RL	Detection Limits Units MDL pCi/g	Limits MDL	Units	Run Date 0	Check List 6	Check List 6886 t Units LCL	1886 LCL UCL RPD T A 75 125 25 C Y	Amt	oike List ( Units	5887 LCL UCL RPD 60 140 25

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Structured Analysis Code: I-FX-ZO-01-06 Target Analyte List: All Analytes	I-FX-ZO-01-06 All Analytes					а ОС	Matrix: Extraction: Method: QC Program: Location:	WATER Precipitate, Separation SR-90 BY GFPC EPA-905 MOD STANDARD TEST SET TestAmerica St. Louis
Analyte List Syn Compound 4111 Strontium 90	R 3	Detection Limits Units MDL pCI/L	Limits MDL	Units	Run Date	Check List TAAmt Units CY	Check List 6502 t Units LCL 78	Spike List 6549  LCL UCL RPD T A Amt Units LCL UCL RPD  78 146 40 C Y 37 150 40

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Structured Analysis Code: A-FW-ZO-01-06 Target Analyte List: All Analytes	A-FW-ZO-01 All Analytes	90					<sup>ш</sup> 8	Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Digest, Precipitate, Separation SR-90 BY GFPC EPA-905 MOD STANDARD TEST SET TestAmerica St. Louis	RD-0403
Analyte List Syn Compound	ೱ	Detection Limits Units MDL	Limits MDL	Units	Run Date	<b>∀</b> ∀	Checi	Check List 6502 t Units LCL	. UCL RPD T A An	Check List 6502 Spike List 6549  T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD	
4111 Strontium 90	ო	pCi/g			0	≻ O		74	74 140 40 C Y	70 130 40	

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Structured Analysis Code: A-HL-ZO-01-06 Target Analyte List: All Analytes	A-HL-ZO-01-(	90					Matrix: Extraction: Method: QC Program: Location:	x: SOLID n: PREP RC-0003, RC-0004, SEP RC-0050, CT SL-13021 d: SR-90 BY GFPC EPA-905 MOD n: STANDARD TEST SET n: TestAmerica St. Louis	50 , CT SL-13021 0-
Analyte List Syn Compound 4111 Strontium 90	R 3	Detection Limits Units MDL pCi/g	Limits	Units	Run Date	T A Amt	Check List 6502 t Units LCL 74	Spike List 6 .UCL RPD T A Amt Units 140 40 C Y	549 LCL UCL RPD 70 130 40

Structured Analysis Code: I-FX-ZK-01-06  Target Analyte List: All Analytes			1000	ı
	:		Extraction:  Method:  QC Program:  Location:	WATEK Precipitate, Separation Strontium-90 by GFPC DOE SR-03-RC MOD STANDARD TEST SET TestAmerica St. Louis
Analyte List Detection Limits	-imits		Check List 6502	)2 Spike List 6549
Syn Compound RL Units MDL	MDL Units	Run Date	T A Amt Units LO	LCL UCL RPD T A Amt Units LCL UCL RPD
4111 Strontium 90 3 pCi/L		0	C Y 78	78 146 40 C Y 37 150 40

Structured Analysis Code: A-FW-ZK-01-06 Target Analyte List: All Analytes	A-FW-ZK-01. All Analytes	90-			;		Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Digest, Precipitate, Separation Strontium-90 by GFPC DOE SR-03-RC MOD STANDARD TEST SET TestAmerica St. Louis	, Precipitate, FPC DOE SF SET outs	, Separal R-03-RC	tion : MOD
Analyte List Syn Compound 4111 Strontlum 90	3 E	Detection Limits Units MDL pCl/g	n Limits MDL	Units	Run Date 0	<b>→ A</b>	Check List 6502 nt Units LCL 74	t 6502 LCL UC 74 14	C Y C Y Spike List 6549  Spike List 6549  C A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD  C Y 74 140 40 C Y 70 130 40	Spike L Amt Unit	Spike List 6549 Units LCL	549 LCL UCL RPD 70 130 40

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Structured Analysis Code: A-HL-ZK-01-06 Target Analyte List: All Analytes	A-HL-ZK-01-0 All Analytes	90					Matrix: Extraction: Method: QC Program: Location:		SOLID PREP RC-0003, RC-0004, SEP RC-0050, ( Strontium-90 by GFPC DOE SR-03-RC MOD STANDARD TEST SET TestAmerica St. Louis	SOLID PREP RC-0003 , RC-0004 , SEP RC-0050 , CT SL-13021 Strontium-90 by GFPC DOE SR-03-RC MOD STANDARD TEST SET TestAmerica St. Louis
Analyte List		Detection Limits	Limits			o	Check List 6502	\$502	Spike	Spike List 6549
Syn Compound	RL	Units	MDL	Units	Run Date	T A Amt Units		LCL UCL RPD	T A Amt Uni	LCL UCL RPD T A Amt Units LCL UCL RPD
4111 Strontium 90	က	pCi/g			0	<b>≻</b> 0		74 140 40 C Y	> 0	70 130 40

Structured Analysis Code: A-K3-ZK-01-06 Target Analyte List: All Analytes	A-K3-ZK-01-(	90	:		i		Matrix: Extraction: Method: QC Program: Location:	l	SOLID As Receive trontium-{ TANDARI	SOLID As Received, Digest, Precipitate, Seperation Strontium-90 by GFPC DOE SR-03-RC MOD STANDARD TEST SET TestAmerica St. Louis	ecipitate, 9	Seperatio 3-RC MOI	
Analyte List		<b>Detection Limits</b>	Limits			J	Check List 6502	: 6502		w	Spike List 6549	6549	
Syn Compound	RL	Units	MDF	Units	Run Date	T A Amt	Units	in ToT	CL RPD	T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD	Units	רכר חכ	L RPD
4111 Strontium 90	ო	pCi/g			0	<b>≻</b>		74 14	74 140 40 C Y	C ≺		70 130 40	40

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Structured Analysis Code: I-FX-ZO-DO-06 Target Analyte List: All Analytes	I-FX-ZO-DO-(	90					Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation SR-90 BY GFPC EPA-905 MOD DOE QSAS TestAmerica St. Louis	n v-905 MOD	
Analyte List		Detection Limits	n Limits				Check List 6888	6888		Spike List 6889	6889
Syn Compound	RL	Units	MDL	Units	Run Date	T A Amt	Units	רכר תכו	T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD	t Units	LCL UCL RPD
4111 Strontium 90	ო	pCi/L			0	<b>≻</b> O		75 125	75 125 25 C Y		60 140 25

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Structured Analysis Code: A-FW-ZO-DO-06 Target Analyte List: All Analytes	<b>A-FW-ZO-D</b> C All Analytes	90-(				AC P	Matrix: Extraction: Method: QC Program: Location:	SOLID Dry, Grind, Digest, Precipitate, Separation SR-90 BY GFPC EPA-905 MOD DOE QSAS TestAmerica St. Louis	ecipitate, Separation 905 MOD
Analyte List Syn Compound 4111 Strontium 90	3 RL	Detection Limits Units MDL pCi/g	ı Limits MDL	Units	Run Date 0	Check T A Amt Unit	Check List 6888 t Units LCL 75	.888 LCLUCL RPD T A Ami 75 125 25 C Y	C Y C Y Spike List 6889 Spike List 6889 Spike List 6889 C L UCL RPD T A Amt Units LCL UCL RPD C Y 60 140 25

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Structured Analysis Code: I-FX-ZK-DO-06 Target Analyte List: All Analytes	I-FX-ZK-DO-( All Analytes	90					Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation Strontium-90 by GFPC DOE SR-03-RC MOD DOE QSAS TestAmerica St. Louis	Separation by GFPC E St. Louis	00E SR-03.	-RC MOD
Analyte List Syn Compound 4111 Strontium 90	R 3	Detection Limits Units MDL pCi/L	Limits	Units	Run Date 0	<b>∀</b>	Check List 6888 TAAmt Units LCL	st 6888 LCL 1	Spike List 6889 LCL UCL RPD T A Amt Units LCL UCL 75 125 25 C Y 60 140	S A Amt	Spike List 6889 Units LCI 60	889 LCL UCL RPD 60 140 25

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Structured Analysis Code: A-FW-ZK-DO-06 Target Analyte List: All Analytes	A-FW-ZK-D(	90-0					00	Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Digest, Precipitate, Separation Strontium-90 by GFPC DOE SR-03-RC MOD DOE QSAS TestAmerica St. Louis	recipitate, S C DOE SR- is	eparation 03-RC MOD
Analyte List		Detection Limits	ı Limits				Check	Check List 6888	88		Spike List 6889	t 6889
Syn Compound	RL	Units	MDL	Units	Run Date	T A	Amt Un	its L	CL UCL RP	DTAA	nt Units	T A Amt Units LCLUCL RPD T A Amt Units LCLUCL RPD
4111 Strontium 90	ო	pCi/g			0	≻ C		7,	75 125 25 C Y	≻ C		60 140 25

Matrix: WATER Extraction: Precipitate, Separation  Method: Total SR BY GFPC EPA-905 MOD  AC Program: STANDARD TEST SET  Location: TestAmerica St. Louis				-	Τ/
Extraction: Method: QC Program: Location:			Matrix:	WATER	<u> </u>
Method: QC Program: Location:	Structured Analysis Code:	1.EY.7M.01.06	Extraction:	Precipitate, Separation	U
QC Program: Location:	onacialea Aliaiysis code.		Method:	Total SR BY GFPC EPA-905 MOD	T
Location: TestAmerica St. Louis	Target Analyte List:	All Analytes	QC Program:	STANDARD TEST SET	<i>.</i> .
			Location:	TestAmerica St. Louis	

l Strontium Total 3 pCi/L 0 C Y 70 130 40 C Y 70 130 40	Analyte List Detection Limits Check List 6525 Spike List 6593

	Matrix:	SOLID
Strictured Applyais Codo: A.EW.7M.01.08	Extraction:	Dry, Grind, Digest, Precipitate, Separation
of actual and size of the mind of the control of th	Method:	Method: Total SR BY GFPC EPA-905 MOD
Target Analyte List: All Analytes	QC Program:	STANDARD TEST SET
	Location:	Location: TestAmerica St. Louis

Analyte List		Detection Limits	Limits				Check List 6525	st 6525	Spike	Spike List 6593	93
Syn Compound	됩	Units	MDL	Units	Run Date	T A An	nt Units	T A Amt Units LCL UCL RPD T A Amt I	Amt	Jits L	Units LCL UCL RPD
4154 Strontium Total	ო	pCi/g			0	<b>≻</b>		70 130 40 C Y		7	70 130 40

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Structured Analysis Code: I-FX-ZM-DO-06 Target Analyte List: All Analytes				
			Matrix: Extraction: Method: QC Program: Location:	WATER Precipitate, Separation Total SR BY GFPC EPA-905 MOD DOE QSAS TestAmerica St. Louis
Analyte List Detection	Detection Limits		Check List 6892	Spike List 6893
Syn Compound RL Units	ts MDL Units	Run Date	T A Amt Units LC	LCL UCL RPD T A Amt Units LCL UCL RPD
4154 Strontium Total 3 pCi/L	لي	0	C Y 75	75 125 25 C Y 60 140 25

		STL	STL Refe		rence Data Summary	ta Sı	mmr	ary		
Structured Analysis Code: A-FW-ZM-DO-06 Target Analyte List: All Analytes	A-FW-ZM-D	90-O					Matrix: Extraction: Method: QC Program: Location:		SOLID Dry, Grind, Digest, Precipitate, Separation Total SR BY GFPC EPA-905 MOD DOE QSAS TestAmerica St. Louis	ipitate, Separation A-905 MOD
Analyte List	i	<b>Detection Limits</b>	Limits				Check List 6892	t 6892	<i>s</i>	Spike List 6893
Syn Compound	R	Units	MOL	Onits	Run Date	<b>∀</b> ∀ ⊢	mt Units	20 727	. RPD T A Amt	T A Amt Units LCLUCL RPD T A Amt Units LCLUCL RPD
4154 Strontium Total	က	pCi/g			0	≻ O		75 126	75 125 25 C Y	60 140 25

·K	<u>D.</u>	<u> </u>	4	<u>U 3</u>	<u> </u>
	Matrix: WATER	xtraction: Precipitate, Separation	Total Strontium by GFPC DOE SR-03-RC MOD	STANDARD TEST SET	Location: TestAmerica St. Louis
	Matrix:	xtraction:	Method:	QC Program:	Location:
		Ш		၁၀	
		Structured Anglusis Code: 1-EX-71-04-08	Structured Atlantaio Code: 1-1 A-Ec-01-00	Target Analyte List: All Analytes	

	Analyte List		<b>Detection Limits</b>	Limits				Check	Check List 6525	25	S	Spike List 6593	6593	
Syn	Syn Compound	R	Units	MDL	Units	Run Date	T A An	nt Unit	Ņ.	T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD	A Amt	Units	דכר חכו	. RPD
4154	4154 Strontium Total	ဇ	pCi/L			0	<b>≻</b>		7	70 130 40 C Y	>		70 130	40

	STL Reference Data Summary	ummary		ST-
Structured Analysis Code: A-FW-ZJ-01-06 Target Analyte List: All Analytes	: <b>A-FW-ZJ-01-06</b> All Analytes	Matrix: Extraction: Method: QC Program: Location:	SOLID Dry, Grind, Digest, Precipitate, Separation Total Strontium by GFPC DOE SR-03-RC MOD STANDARD TEST SET TestAmerica St. Louis	RD-0403

	Matrix: SOLID	SOLID
throughton Analysis Codo: A-K3-71-04-06	Extraction:	As Received, Digest, Precipitate, Seperation
cluied Alialysis Code. A-NS-23-01-00	Method:	Method: Total Strontium by GFPC DOE SR-03-RC MOD
Target Analyte List: All Analytes	QC Program:	STANDARD TEST SET
	Location:	Location: TestAmerica St. Louis

	Analyte List		<b>Detection Limits</b>	imits				Che	Check List 6525	3525		o)	spike List 6593	t 6593		
Syn (	Syn Compound	R	Units	MDL	Units	Run Date	T A Amt	\mt C	Inits	Units LCL UCL RPD T A Amt Units	PD T	A Amt	Units	걸	LCL UCL RPD	
4154	4154 Strontium Total	10	pCi/sampl			0	≻ C			70 130 4	130 40 C Y	<b>&gt;</b>		70 130	130 40	

Structured Analysis Code: I-FX-ZJ-DO-06 Target Analyte List: All Analytes	I-FX-ZJ-DO-0 All Analytes	90		:			Matrix: Extraction: Method: QC Program: Location:		WATER Precipitate, Separation Total Strontium by GFPC DOE SR-03-RC MOD DOE QSAS TestAmerica St. Louis	n PC DOE SR	-03-RC MOD
Analyte List		Detection Limits	n Limits				Check List 6892	t 6892		Spike List 6893	6893
Syn Compound	RL	Units	MDL	Units	Run Date	T A A	T A Amt Units	רכר תנ	LCL UCL RPD T A Amt Units LCL UCL RPD	t Units	LCL UCL RPD
4154 Strontium Total	ო	pCi/L			0	≻ C		75 12	75 125 25 C Y		60 140 25

	  -  )		)		}					ST-1
Structured Analysis Code: A-FW-ZJ-DO-06 Target Analyte List: All Analytes	90-0Q-1s					9 o	Matrix: Extraction: Method: QC Program: Location:	SOLID Dry, Grind, Digest, Precipitate, Separation Total Strontium by GFPC DOE SR-03-RC MOD DOE QSAS TestAmerica St. Louis	oitate, Separation DOE SR-03-RC MOD	RD-0403
Analyte List Syn Compound 4154 Strontium Total	Detection Limits Units MDL pCi/g	n Limits MDL	Units	Run Date	<b>∀</b> ≻ <b>⊢</b> O	Check List TAAmt Units CY	<b>.</b>	.UCL RPD T A Amt	Spike List 6893 Units LCL UCL RPD 60 140 25	

ST-RD-0403

Str	Structured Analysis Code: I-JE-3S-01-06 Target Analyte List: All Analytes	<b>I-JE-3S-01-06</b> All Analytes						Matrix: Extraction: Method: QC Program: Location:		WATER Dry, Grind, Leach/Dige Phosphorus-32/33 by ( STANDARD TEST SE' TestAmerica St. Louis	WATER Dry, Grind, Leach/Digestion, Phosphorus-32/33 by GFPC STANDARD TEST SET TestAmerica St. Louis	ion, phos p PC	WATER Dry, Grind, Leach/Digestion, phos ppt> amm. ppt. Phosphorus-32/33 by GFPC STANDARD TEST SET TestAmerica St. Louis	
Syn 5481	Analyte List Syn Compound 5481 Phosphorus-32/33	R S	Detection Limits Units MDL pCi/L	Limits	Units	Run Date	T A Am	Check List 6544 it Units LCL	16544 LCL U 70 13	544 LCL UCL RPD 70 130 5	Si T A Amt C Y	Spike List 6568 Units LCL	C Y Spike List 6544 Spike List 6568  Spike List 6568  Spike List 6568  C Y Spike List 6568  C Y Spike List 6568  C Y Spike List 6568	

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S	STL Reference Data Summary		ST-
	Matrix: S	SOLID	RD
Structured Analysis Code: A-JE-3S-01-06		Dry, Grind, Leach/Digestion, phos ppt> amm. ppt.	- 0
	Method: Pi	Phosphorus-32/33 by GFPC	4
Target Analyte List: All Analytes	QC Program: S'	STANDARD TEST SET	0,3
	Location: Te	Location: TestAmerica St. Louis	3

Analyte List		Detection	_			O	Sheck List 6544	6544	Spil	spike List 6568	568
Syn Compound	몺	Units	MDL	Units	Run Date	T A Amt	Units	Units LCL UCL RPD T A Amt	Amt	Units	Units LCL UCL RPD
5481 Phosphorus-32/33	ĸ	pCi/g			0	<b>≻</b> O		70 130 40 C Y			50 150 40

Target Analyte List: All Analytes	Structured Analysis Code: I-JG-3U-01-06 Target Analyte List: All Analytes					Matrix: Extraction: Method: QC Program: Location:		WATER Dry, Grind, Leach/diges Sulfur-32/35 by GFPC STANDARD TEST SET TestAmerica St. Louis	ition,separat	WATER Dry, Grind, Leach/digestion, separation -> BaSO4 ppt. Sulfur-32/35 by GFPC STANDARD TEST SET TestAmerica St. Louis
Analyte List	Detection Limits	ı Limits			_	Check List 6546	546		Spike List 6570	570
Syn Compound RL	Units	MDL	Units	Run Date	T A Amt Units	Units	LCL UCL RF	LCL UCL RPD T A Amt Units LCL UCL RPD	Units	LCL UCL RPD
5485 Sulfur-32/35 5	pCI/L			0	≻ C		70 130 40 C Y	≻ C		50 150 40

					Γ-F
Structured Analysis Code: A-JG-3U-01-06 Target Analyte List: All Analytes			Matrix: Extraction: Method: QC Program: Location:	:: SOLID :: Dry, Grind, Leach/digestion,separation -> BaSO4 ppt. :: Sulfur-32/35 by GFPC :: STANDARD TEST SET :: TestAmerica St. Louis	RD-0403
Analyte List Detection Limits Syn Compound RL Units MDL 5485 Sulfur-32/35 5 pCi/g	on Limits MDL Units	Run Date	Check List 6546 T A Amt Units LCL	Spike List 6570   Spike List 6570   CL UCL RPD T A Amt Units LCL UCL RPD 70 130 40 C Y 50 150 40	

Structured Analysis Code: I-JF-3T-01-06 Target Analyte List: All Analytes	-JF-3T-01-06						o l	Matrix: Extraction: Method: QC Program: Location:		WATER Dry, Grind, Leach/digestion,separation -> AgCl ppt. Chlorine-36 by GFPC STANDARD TEST SET TestAmerica St. Louis	stion,separ. T	ation -> AgCl	td:
Analyte List		Detection Limits	ı Limits				ີ້ວ	Check List 6545	45		Spike List 6569	6969	
Syn Compound	R	Units	MDL	Units	Run Date	<b>∀</b> ⊢	T A Amt Units		CL UCL R	LCL UCL RPD T A Amt Units LCL UCL RPD	Units	LCL UCL F	0
5483 Chlorine-36	တ	pCi/L			0	≻ 0		7	70 130 40 C Y	≻ C ≺		70 130 40	0

		Matrix: SOLID	SOLID
		Extraction:	Dry, Grind, Leach/digestion, separation -> AgCl ppt.
offuctured Affalysis code: A-31-01-00		Method:	Method: Chlorine-36 by GFPC
Target Analyte List:	All Analytes QC	C Program:	QC Program: STANDARD TEST SET
		Location:	Location: TestAmerica St. Louis

T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD Spike List 6569 Check List 6545

68 120 40

**≻** 

Run Date

Units

MDL

Units pCi/g

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Analyte List Syn Compound

5483 Chlorine-36

**Detection Limits** 

70 130 40

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ge number 1