SECTION A - ADMINISTRATION

A1 TITLE AND APPROVAL PAGE

EPA BROWNFIELDS HAZARDOUS SUBSTANCES CLEANUP GRANT - REGION 7
CITY OF DUBUQUE, DUBUQUE COUNTY, IOWA

TITLE: PROJECT PLAN— West Blum Cleanup Project
Data Quality Objectives and Generic Quality Assurance Project Plan Version 1.0, December 9, 2019
EPA ID No. CAG#s BF 97762001 and BF-97764401

PREPARED BY:

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12-9-2019
(Signature) Steve Sampson Brown (Date)
Project Director/Manager

BLACKSTONE ENVIRONMENTAL, INC.REVIEWER:

12/9/2019
(Signature) Emily Smart (Date)
Project Manager

12/9/2019
(Signature) Scott Mattes (Date)
QA/QC Reviewer

U.S. EPA PROJECT MANAGER APPROVAL:

SUSAN KLEIN Digitally signed by SUSAN KLEIN Date: 2020.03.17 10:41:10 -05'00'
(Signature) ~~Deborah Kennedy~~ (Date)
Brownfields Project Officer

U.S. EPA QA OFFICERS APPROVAL:

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A Project Location Map

B QAPP Amendment Template

C Iowa Land Recycling Program & Statewide Response Action Standards (IAC 137) Iowa Administrative Code (455H) Chapter 137

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E Laboratory Quality Assurance / Quality Control Manual

F Standard Operating Procedures

G EPA Method 6200

H EPA §61.145 Standard for Demolition and Renovation – Asbestos

A3 DISTRIBUTION LIST

Digital copies of *Project Plan: West Blum Cleanup Project Data Quality Objectives and Quality Assurance Project Plan, Version 1.0, December 14, 2018* will be distributed as follows. These persons will also receive copies of routine report distributions as set forth in section C2.

A3.1 United States Environmental Protection Agency, Region 7
11201 Renner Blvd.
Lenexa, KS 66219
Deborah Kennedy, Brownfields Project Officer

A3.2 City of Dubuque
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Steve Sampson-Brown, Engineering Department

A3.3 Project File
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A3.4 TestAmerica Incorporated
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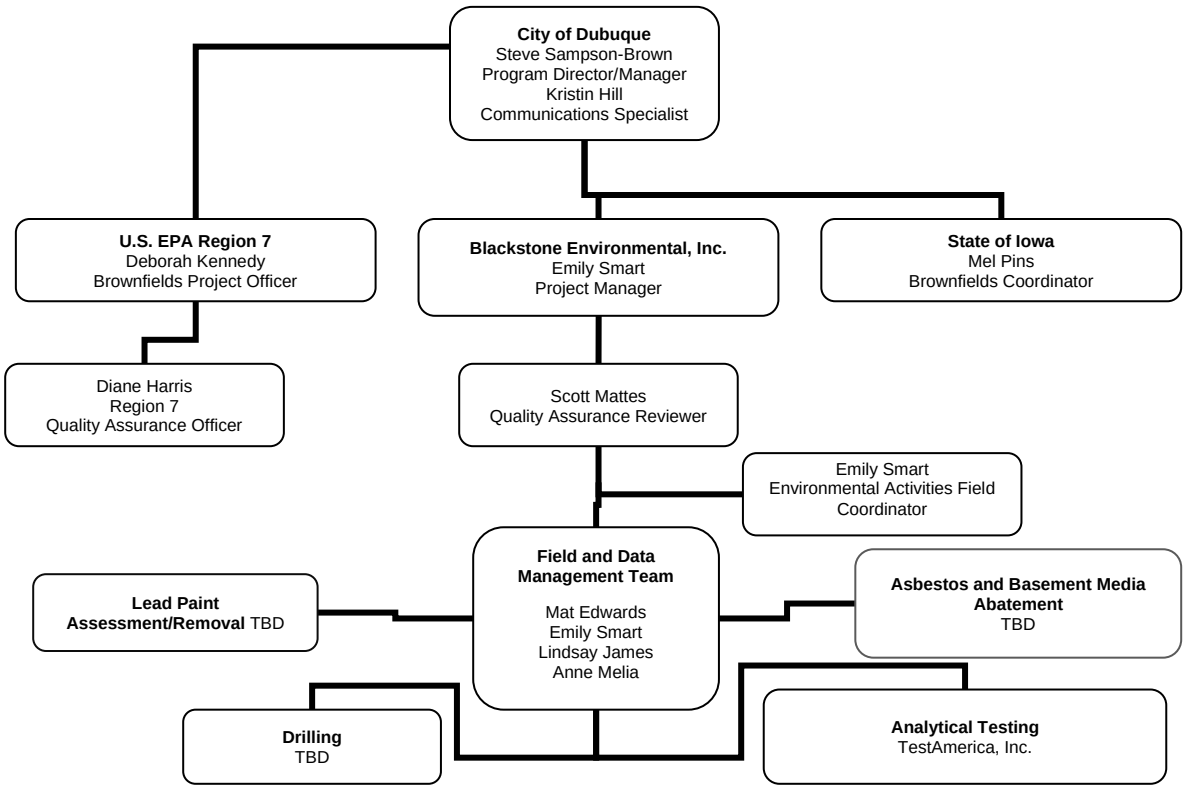
Christopher A. Deimerly, Quality Assurance Coordinator

A3.5 Iowa Department of Natural Resources
Contaminated Sites Section / Iowa Land Recycling Program
Henry A. Wallace Building
502 E. 9th Street
Des Moines, IA 50319-0034
Mel Pins, Iowa Brownfields Coordinator

A4 PROJECT ORGANIZATION



Representing the City of Dubuque (City) as issuing agency is Mr. Steve Sampson-Brown, Program Director/Manager. Mr. Sampson-Brown is charged with directing project activities, approving final documents, and coordinating efforts between the consultant, state, and federal reviewers.

The Environmental Protection Agency (EPA) Brownfields Project Officer is the point of contact for the Cleanup Grant. The Brownfields Project Officer is responsible for reviewing and providing comments on the Quality Assurance Project Plan (QAPP), responding to questions regarding the grant process, and other project submittals.

The Project Manager for Blackstone, Inc. (Blackstone) is Ms. Emily Smart. Ms. Smart oversees the consultant activities with the Project including planning, monitoring, and evaluating project field activities; resolving technical issues and providing guidance; and reviewing reports and documents. Ms. Smart reports to the City's Project Director/Manager.

The Quality Assurance Reviewer for Blackstone is Mr. Scott Mattes. Mr. Mattes will perform quality assurance/quality control (QA/QC) audits, check and assist in document and project reviews relative to the project plan, and review the QAPP annually for updates.

Site investigation, cleanup activities, and evaluation are overseen by the Blackstone Environmental Activities Field Coordinator, Mr. Mat Edwards. Mr. Edwards will conduct investigations and coordinate other Blackstone employees who perform the intrusive investigation and cleanup portion of the Project. Mr. Edwards will be working with TestAmerica analytical laboratory services and a certified drilling service to be determined at the time of the investigation. Ms. Smart will coordinate cleanup activities involving asbestos and lead, analytical laboratories, and drilling professionals who hold all necessary state and federal certifications and licenses to complete work planned on the Project.

The Iowa Department of Natural Resources (IDNR) Brownfields Coordinator oversees the Land Recycling Program (LRP). Mr. Mel Pins will provide project oversight and guidance activities as a result of enrolling the Site into the LRP.

A5 PROBLEM DEFINITION AND BACKGROUND

The primary objective of the Project is to design and implement the cleanup of the West Blum site located at 411 West 15th Street (site) in Dubuque, Iowa (Appendix A). To meet the primary objective, activities to be conducted pursuant to this generic QAPP and its amendments, are, abatement of site hazardous materials and delineation of extent of contamination for the purposes of completing the design of the cleanup and cleanup confirmation/verification sampling in accordance with IDNR LRP requirements. The City needs to fully evaluate the extent of identified contamination at the site (Table 1) as required by the IDNR LRP. This project plan is intended to provide an overview of EPA Brownfields Cleanup activities performed in support of the City's EPA Brownfields Redevelopment Initiative and to help ensure the reliability of data generated from those activities.

The City acquired the site on December 20, 2016 after completing their due diligence and following All Appropriate Inquiry requirements. Subsequent Phase II Environmental Site Assessment (ESA) results indicated that the parcel was not suitable for future residential, commercial, or industrial uses without remediation of shallow soil. Levels of polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs) and Resource Conservation and Recovery Act (RCRA) metals in shallow soil presented an unacceptable cancer and non-cancer risk. Additionally, cleanup activities will be required prior to structure demolition including asbestos containing material (ACM) abatement and characterization and disposal of the material which has accumulated on the basement floor of the main office building and the underlying concrete (including building footers) according to local state and federal regulations.

Table 1

Phase II ESA Range 1 Soil Analytical Results – PCBs, TEHs, PAHs, VOCs, and RCRA Metals (mg/kg)

Parameter	SWS / Tier 1	SB1 @ 0-2	SB2 @ 0-2	SB3 @ 0-2	OC	BC	CC	Duplicate
PCBs								(CC)
PCB-1016	1.1*	<2.90	NS	NS	<0.0560	<0.0573	<0.0592	<0.0566
PCB-1221		<2.90	NS	NS	<0.0560	<0.0573	<0.0592	<0.0566
PCB-1232		<2.90	NS	NS	<0.0560	<0.0573	<0.0592	<0.0566
PCB-1242		16.3	NS	NS	40.2	1.93	7.53	5.35
PCB-1248		<2.90	NS	NS	<0.0560	<0.0573	<0.0592	<0.0566
PCB-1254		<2.90	NS	NS	<0.0560	<0.0573	<0.0592	*<1.13*
PCB-1260		<2.90	NS	NS	<0.0560	<0.0573	<0.0592	<0.0566
PCB-1268		<2.90	NS	NS	<0.0560	<0.0573	<0.0592	<0.0566
Total PCB**		36.6	NS	NS	40.2	1.93	7.53	6.49
TEHs								SB1
Waste Oil	9400	58500	NS	NS	NS	103000	93800	50700
PAHs								(CC)
Acenaphthene	3400	0.403 J	<0.371	<0.0346	<0.350	<1.73	<0.181	<0.179
Acenaphthylene	1700	<0.233	0.274 J	0.126	<0.220	<1.09	0.206 J	0.144 J
Anthracene	17000	0.603 J	<0.357	0.0686 J	<0.337	<1.67	<0.174	<0.172
Benzo[a]anthracene	3.1	0.952 J	0.853 J	0.171	0.338 J	<1.19	0.437 J	0.271 J
Benzo[a]pyrene	0.31	0.965 J	0.963 J	0.311	0.573 J	<0.727	0.649	0.435 J
Benzo[b]fluoranthene	3.1	1.32	1.60	0.510	1.02 J	0.908 J	1.41	1.03
Benzo[g,h,i]perylene	170	0.592 J	0.837 J	0.337	0.623 J	<0.803	0.825	0.674
Benzo[k]fluoranthene	31	0.613 J	0.584 J	0.219	0.250 J	<0.688	0.562 J	0.408 J
Chrysene	310	1.23	1.07 J	0.239	0.478 J	<0.732	0.800	0.473 J
Dibenz(a,h)anthracene	0.31	<0.161	0.206 J	0.0877 J	<0.152	<0.754	0.170 J	0.154 J
Fluoranthene	2300	2.73	2.11	0.259	0.660 J	<1.61	1.49	0.855
Fluorene	2300	0.515 J	<0.443	<0.0413	<0.419	<2.07	<0.216	<0.214
Indeno[1,2,3-cd]pyrene	3.1	0.480 J	0.676 J	0.279	0.499 J	<0.787	0.694	0.556 J
2-Methylnaphthalene	230	1.01 J	<0.405	0.0379 J	<0.382	<1.89	0.423 J	0.363 J
Phenanthrene	1700	2.21	1.56	0.121	0.413 J	<1.43	0.869	0.480 J
Pyrene	1700	2.99	1.85	0.267	0.675 J	1.61 J	1.47	0.836
Naphthalene	1100	0.642 J	<0.508	0.0492 J	<0.479	<2.37	<0.248	<0.245
VOCs								(SB1)
Acetone	68000	0.117	NS	NS	NS	<0.575	NS	<0.532
Benzene	56 / 3800	0.0277	NS	NS	NS	<0.115	NS	1.23
n-Butylbenzene	3800	0.0093	NS	NS	NS	<0.115	NS	0.332
Chlorobenzene	1500	<0.00688	NS	NS	NS	<0.115	NS	0.433
1,2-Dichlorobenzene	5500	0.0158	NS	NS	NS	<0.115	NS	2.63
1,4-Dichlorobenzene	760	<0.00688	NS	NS	NS	<0.115	NS	0.111
Ethylbenzene	7600 / 15	0.0144	NS	NS	NS	<0.115	NS	0.908
Hexane	4600	<0.0344	NS	NS	NS	<0.115	NS	0.409
Isopropylbenzene	7600	<0.00688	NS	NS	NS	<0.115	NS	0.158
p-Isopropyltoluene	NA	<0.00688	NS	NS	NS	<0.115	NS	0.157
Naphthalene	1100	<0.0344	NS	NS	NS	<0.287	NS	0.893
N-Propylbenzene	7600	0.00759	NS	NS	NS	<0.115	NS	0.413
Tetrachloroethene	1500	<0.00688	NS	NS	NS	<0.115	NS	0.24
Toluene	6100 / 3.2	0.0447	NS	NS	NS	<0.115	NS	1.7
Trichloroethene	67	<0.00688	NS	NS	NS	<0.115	NS	1.29
1,2,3-Trichloropropane	0.1	<0.00688	NS	NS	NS	*<0.115*	NS	*<0.106*
1,2,4-Trimethylbenzene	3800	0.0548	NS	NS	NS	<0.115	NS	2.17
1,3,5-Trimethylbenzene	760	0.0197	NS	NS	NS	<0.115	NS	0.618
Xylenes, Total	15000 / 52	0.0699	NS	NS	NS	<0.172	NS	3.09
RCRA Metals								(SB1)
Arsenic	17	102	*<24.8*	13.8	<15.0	37.2	33.1 J	42.5
Barium	15000	401	575	118	183	111	962	822
Cadmium	NA	13.4	23.5	4.57	10.7	14.3	53.6	39.0
Chromium	190	35.8	112	20.3	51.3	109	247	109
Lead	400	896	1700	246	737	29500	2720	3350
Silver	370	<2.88	<3.35	<2.03	<3.74	23.7	<3.87	<8.79
Mercury	23	1.59	4.65	0.298	2.42	2.56	7.95	4.48

Bold indicates concentration reported above laboratory reporting limits. Shaded indicates concentration above SWS. NS: Not sampled. NA: Not calculated. J indicates the result is less than the reporting limit but greater than or equal to the method detection limit and the concentration is an approximate value. *Italics* indicate a non-detect concentration above an applicable SWS. *SWS is 1.1 mg/kg for Total PCBs. **Non-detect values in excess of 1.1 mg/kg are included in the total PCB summation.

This generic QAPP covers all activities associated with the cleanup of the site performed by the City and its consultants pursuant to the Small Business Liability Relief and Brownfields Revitalization Act of 2002. The cleanup activities addressed in this generic QAPP will be pursuant to:

- Iowa Land Recycling Program and Statewide Response Action Standards Iowa Administrative Code (IAC) Chapter 137 (Appendix C)

The State of Iowa has programs for environmental impairment assessments in place through the Iowa Administrative Code (IAC) and rules. These programs include risk-based corrective action programs and a voluntary LRP administered by the IDNR. The appropriate programs overlap in some instances regarding regulation of environmental impairment and releases to soil, groundwater, and air. The Project involves a number of properties having potential environmental issues that overlap regulatory programs. For this Project, soil evaluation completed on the site will be conducted according to Iowa Administrative Code (455H) Chapter 137: *Iowa Land Recycling Program and Statewide Response Action Standards* (IAC 137). An overview of the Iowa LRP is included in Appendix C.

It is the purpose of the generic QAPP to provide a program of decision that produces data of sufficient quantity and quality, balancing between the requirements of the EPA Grant and state programs (IAC 137). This must be done with limited funds and still provide sufficient value to the City with regard to its redevelopment initiative. The following strategies are to be implemented to achieve this balance:

- Use data collected during previous Phase II ESAs when possible;
- Minimize the number of sample locations where feasible;
- Minimize the number of site visits; and
- Collect only the data needed to evaluate and remediate the site appropriately.

These goals will be attained while meeting IAC 137 (Appendix C) requirements.

This generic QAPP is in effect for the duration of the Cleanup Grant project period as indicated on the signature page. The Blackstone QA Reviewer will review the generic QAPP periodically during this time for applicability. Further, if an extension of the Cleanup Grant is necessary and subsequently approved by EPA, or if site conditions change, this generic QAPP will be internally reviewed and amended as required. The amendments will be submitted to the EPA for concurrence and approval.

This generic QAPP distinguishes between West Blum 1 and West Blum 2 to ensure that all activities conducted under the QAPP are associated with the correct cooperative agreement (CA) and are eligible and allowable under that CA. The brownfield properties, West Blum 1 and 2, are distinguished per the definitions below.

- **West Blum 1 Brownfield Site** consists of Buildings 1 (the western building) and 2 (the southeastern building) on the West Blum property located at 411 E 15th St., Dubuque, IA 52001. Cleanup activities include asbestos-containing materials (ACM) abatement and removal, and the characterization and

appropriate disposal (prior to demolition) of an approximately 6''-thick layer of media, as well as the underlying concrete, that has accumulated on the basement floor of the main office building (Building 1) and contains exceedances of PCBs, waste oil, arsenic, and lead. The West Blum 1 brownfield site is being addressed under EPA cleanup CA No. BF97762001.

○ **West Blum 2 Brownfield Site** consists of the soils and subsurface, excluding Buildings 1 and 2, on the West Blum property located at 411 E 15th St., Dubuque, IA 52001. The cleanup addresses unacceptable levels of PCBs, waste oil, arsenic, lead, and chromium concentrations in the soil. The West Blum 2 brownfield site is being addressed under EPA cleanup CA No. BF97764401.

Project-specific activities will be covered by individual QAPP Amendments and any project outside the scope of this generic QAPP will require a separate, stand-alone QAPP.

All future amendments to the generic West Blum QAPP, as well as other CA activities, must be associated with the appropriate site/CA based on these definitions. For amendments to the Generic West Blum Quality Assurance Project Plan, this may be easily accomplished by circling the relevant cooperative agreement number on the QAPP Amendment Template.

A6 PROJECT DESCRIPTION

The Project intends to make use of this Data Quality Objectives (DQO) and QAPP to expand upon contamination identified in the previous Phase II ESA through further delineation, and by designing and implementing a cleanup approach to meet the standards identified in IAC 137: *Iowa Land Recycling Program and Response Action Standards* (Appendix C). Contaminants of concern (COCs) are listed in Table 1 of section A5.

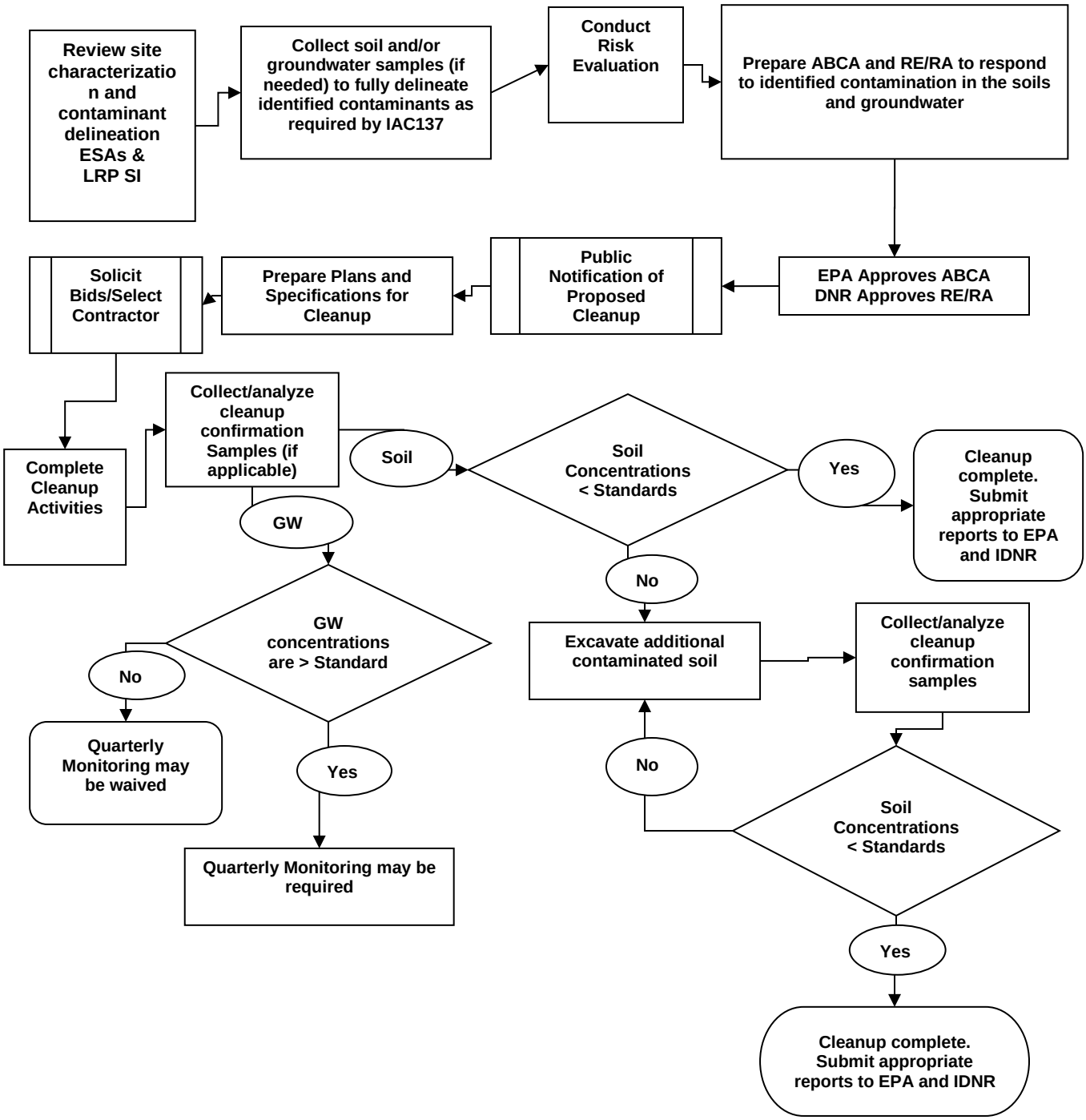
Additional sampling will be completed to fully delineate the horizontal and vertical extent of soil contamination identified during previous ESA activities (See Section B). Additional sampling for complete delineation will include collecting soil samples to be analyzed for PAHs, PCBs, and RCRA metals. Contaminant delineation sampling activities will be presented as part of the LRP Site Assessment Report and will include all soil data. This data will then be utilized for evaluating exposure risks and the final response action plan discussed below.

The Analysis of Brownfields Cleanup Alternatives (ABCA) update will be completed in conjunction with the Risk Evaluation and Response Action (RE/RA) Plan. The ABCA and RE/RA will evaluate contaminant exposure risks and corrective remedies to determine the most efficient and effective method to manage the soil contamination at the site with respect to proposed future land use and will be submitted to EPA and IDNR for approval. After approval of the cleanup approach proposed in the ABCA and RE/RA, a public notice summarizing the ABCA and RE/RA will be issued by the IDNR to all adjoining property owners. Upon completion of the public notice process, the RA plan will be implemented. If implementation of the RA plan includes removal of contaminated soils, confirmation samples will be field screened using XRF, and PID.. Samples selected for fixed-base lab confirmation testing will be collected and analyzed following excavation activities to determine the effectiveness of the remedy. The results of the confirmation sampling and the effectiveness of the selected remedy will be summarized in the RA Implementation Report and submitted to IDNR for review and concurrence. A second public

notification will be issued to present the results of the RA and allow for public comment. At the completion of the Cleanup Project, a Final Report will be submitted to IDNR for approval with a copy to EPA.

A project work schedule will be incorporated into each QAPP Amendment.

Figure 1 EPA Brownfields Cleanup Process



A7 QUALITY OBJECTIVE AND CRITERIA FOR MEASUREMENT DATA

DQOs for activities performed under this project should ensure environmental data obtained meet the needs of the study and can be used with confidence to support specific decisions (both administrative and regulatory) pertaining to the site. DQOs specify the quality of data required from a particular activity to support specific decisions. Specific DQOs used from the list of those outlined under this QAPP will be specified in the QAPP Amendment. A QAPP Amendment template is attached in Appendix B.

In IAC 137 one of three sets of standards may be used to evaluate contaminant concentrations:

- **Background standards** represent concentrations of contaminants that are naturally occurring or generally present and not related to a readily identifiable release. Background standards provide a baseline for assessing impacts of contaminant releases from within the affected area.
- **Statewide standards** address what are considered to be the most likely, normal exposure situations. Statewide Standards (SWS) for soil address direct exposure to soil via ingestion and dermal contact.
- **Site-specific standards** may involve development of target levels for contaminants of concern based on site-specific exposure assumptions for use in lieu of background or statewide standards. Site-specific standards may also include consideration of the actual or potential location where exposure to contaminants occurs or may occur, the likelihood of an exposure occurring, and the overall magnitude and extent of contamination. Site-specific standards may involve use of site-specific target levels for contaminants of concern alone or in conjunction with other site-specific criteria, such as the location where the standard is applied.

For site characterization and contaminant delineation, different types of data may be necessary depending upon the findings in the Phase I and Phase II ESAs. For example, petroleum contamination from an underground storage tank (UST) evaluated according to IAC 135 requires soil samples to be analyzed for benzene, toluene, ethyl benzene, and xylenes (BTEX) by Iowa Method OA-1 and for total extractable hydrocarbons (TEH) by Iowa Method OA-2. The evaluation is not dependent upon the depth at which the contamination is found. For contaminated sites evaluated according to IAC 137, samples may be analyzed for VOCs by EPA SW-846-8260B and RCRA metals by EPA-SW-846-7000/6010. PAHs may be analyzed by EPA Method 8270D SIM and PCBs may be analyzed by EPA method 8082A. Evaluation of the data for IAC 137 may be dependent upon the depth at which the sample was collected. Confirmation/verification sampling will also bee conducted as a part of the LRP process.

Chemical parameters will be measured in both a field and laboratory setting. Field measurements will be made using XRF and PID to help direct the selection of samples for fixed-base laboratory testing. These field measurements will not be used except as qualitative indicators in the evaluation with the possible exception of using field portable X-ray fluorescence (XRF) using EPA Method SW-846-6200 (Appendix G) in lieu of some laboratory analytical testing.

Specific data to be assessed and obtained during site assessment activities based on the applicable state programs are summarized in Tables 2 and 3.

Off-site sampling is not included in Phase II ESAs but may be necessary to determine the extent of contamination and would be completed during site characterization and contaminant delineation if a source of identified contamination appears to be located on the site. This generic QAPP will be used to define contamination sufficiently to begin cleanup planning and will allow for site characterization and contaminant delineation to be conducted in order to prepare sites for enrollment into state voluntary cleanup programs. The specific sampling design information details and rationale will be included in the QAPP Amendment for each additional assessment.

Table 2
EPA Brownfields Hazardous Materials Delineation and Site Assessment

Samples	Approach	Rationale
Source	IAC 137—Identify hazardous materials at the site through composite or discrete samples.	Assess risk to human health and the environment.
Soil samples with other indicators	Key indicators for sampling areas include the following: stressed or absent vegetation; lagoons or pits; drums; tanks; abandoned or leaking containers; container labels with corrosive, explosive, flammable, radioactive, toxic or biologically pathogenic material; stained or damaged buildings or concrete; stained soil; dead animals or birds; sumps; septic systems; etc.	Assess risk to human health and the environment. Sample source and target areas (indicating possible exposure to surficial contamination) within two feet of the surface.
QC Samples	Trip blank (VOC), Duplicate, Rinsate Blank, Field Blank	

*Not all possibilities could be practically conceived or listed. Any additional indicators or deviations from this list would be cited in the field records and the trip report.

Table 3 - Soil

Samples	Approach	Rationale
Residential soil samples	IAC 137—On-site samples collected if proposed future use is residential.	Residential samples collected off-site are outside the scope of the Project.
Background samples	IAC 137—One for each matrix with an identified upgradient REC.	Sample to determine relative concentration of petroleum and hazardous substances potentially migrating onto the site.
Sources	IAC 137—Identify hazardous substances present at the site through composite or discrete samples.	Identified contamination exists. Source identification allows for comparison with any off-site contamination.
QC Samples	Trip blank (VOC), Duplicate*, Rinsate Blank, Field Blank	

*There is routinely a high degree of heterogeneity of soil samples and variability of soil types in an area. Take extra care to collect duplicates from the same soil type as the original sample. Duplicate sampling needed for statistical purposes would be beyond the scope of this generic QAPP.

Previous sample collection identified ACM and material that has accumulated in the basement of the main office building that will require cleanup prior to demolition of the structure. This work will be completed by properly certified professionals, as needed. A QAPP Amendment will be completed and submitted for EPA approval that will provide sampling design information for screening and sampling procedures and SOPs for this component of the Project.

QAPP Amendments for site characterization and contaminant delineation and confirmation/verification sampling will also be and submitted for EPA approval that will provide screening and sampling procedures and SOPs for this component of the Project.

A7.1 Data Categories

Two categories of data will be collected during this Cleanup Project: 1) screening data with or without definitive confirmation results, and 2) definitive data. These categories segregate environmentally related measurement data into two groups which are based primarily on increasing levels of confidence in the precision and accuracy of the analytical results. Screening data with definitive confirmation results comprise data of known quality that are quantitatively "verified" and for which the analyte identification is "definitively" confirmed. Definitive data include all measurements performed using analyte-specific, EPA-approved laboratory methodologies that definitively identify and quantify the analytes of interest. Screening results will be used to select the type and location of fixed-base laboratory analytical samples, including those to be used for definitive confirmation. The data quality available from current field screening technologies is acceptable for this purpose.

For cleanup activities, either of the data categories may be used to determine the necessity for further action at the site. However, only definitive data will be used to demonstrate that cleanup activities are complete and meet the standards outlined in IAC 137.

A7.1.1 Screening Data With and Without Confirmation Results

The screening category is a broad classification including non-quantitative to semi-quantitative measurements, or involving only probable identification of a compound class, such as total VOCs with a flame or photoionization detector. This category will be appropriate for data collection activities involving rapid, non-rigorous measurement or analytical procedures and limited QA/QC requirements. The screening methods will be used to make quick evaluations of the types and concentration of pollutants. Screening will often be employed during cleanup activities and may be used for a preliminary evaluation of cleanup success.

Definitive confirmation refers to the analysis of samples by a technique that can unequivocally detect the specific analyte in question and can produce verifiable documentation that the analyte identification is correct. Quantification of a parameter of interest is considered to be valid if the precision and accuracy of the data is determined to be within the control limits established in this document. For the cleanup activities, flame or photoionization detector. If screening indicates that the excavation extent has satisfactorily removed the impacted media, then the actual number of samples required by IAC 137 will be collected. The number of samples collected will not be related to the number of samples screened.

A7.1.2 Definitive Data

The most exhaustive category is definitive data, which is appropriate when rigorous, EPA-approved methods of analysis and comprehensive QA/QC procedures are necessary. This category will be applied when a highly significant cost or risk is associated with an incorrect decision. Definitive data are analyte-specific with confirmation of analyte identities and concentrations. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either

analytical or total measurement error must be determined. See Section B5 for quantitative DQOs.

A8 SPECIAL TRAINING REQUIREMENTS AND CERTIFICATIONS

All site sampling personnel will have successfully completed the 40-hour health and safety (Hazardous Waste Operations and Emergency Response [HAZWOPER]) training course and annual refreshers. Familiarity with sampling equipment and procedures will also be necessary for the sampling team.

IDNR maintains a list of registered drillers who drill water supply and monitoring wells into groundwater in the State of Iowa. This registration requires that the driller demonstrate proficiency in drilling and sampling wells and the methods necessary under Iowa Code for abandoning wells. This project requires drilling and sampling groundwater through monitoring wells and will utilize state-registered drillers to complete this work.

A9 DOCUMENTATION AND RECORDS

Field personnel will maintain a field logbook to record all pertinent activities, including any difficulties encountered in the field, associated with the sampling event. Appropriate documentation pertaining to photographs taken by field personnel will also be recorded in the field logbook. Information pertaining to samples (i.e., sampling dates and times, locations, etc.) collected during this event will be recorded on sample field sheets. Sample labels will be affixed to sample containers, identifying sample numbers, dates collected, and requested analyses.

TestAmerica, Inc. have prepared the related fixed-base laboratory quality assurance portions of the generic DQO/QAPP.

- Sample data for the laboratory as discussed in Appendix E: TestAmerica, Inc. Quality Assurance Manual (July 12, 2018).
- Sample management records and documentation for the laboratory as discussed in Appendix E: TestAmerica, Inc. Quality Assurance Manual (July 12, 2018).
- Test methods for the laboratory as discussed in Appendix E: TestAmerica, Inc. Quality Assurance Manual (July 12, 2018).
- Quality assurance and control reports for the laboratory as discussed in Appendix E: TestAmerica, Inc. Quality Assurance Manual (July 12, 2018).
- Data handling records for the laboratory as discussed in Appendix E: TestAmerica, Inc. Quality Assurance Manual (July 12, 2018).

The final product (i.e., deliverable report) for the Cleanup Project will be a report describing the implementation of the ABCA and RE/RA as required in IAC 137 (Appendix C). The report will provide:

- A narrative of field activities

- A Site map
- Laboratory Analytical Reports and summaries of analytical data
- Manifests for hauling of contaminated soil, if any
- Documentation of any completed proper disposal of contaminated soil, if any
- Recommendations

The Blackstone Project Manager, in conjunction with the TestAmerica QA Manager, has the primary responsibility for defining site-specific data reporting requirements and relating them to the Environmental Activities Field Coordinator. These requirements, the turnaround time for receipt of deliverables specified, and any site-specific requirements for retention of samples and laboratory records, should be clearly defined in requests for analytical services. The TestAmerica QA Manager is responsible for ensuring that all laboratory data reporting requirement in the QAPP Amendment and the QAPP are met. It is also the responsibility of the Blackstone Project Manager to provide the Environmental Activities Field Coordinator with the most recent version of the EPA-approved QAPP.

SECTION B - MEASUREMENT AND DATA ACQUISITION

B1 SAMPLING PROCESS DESIGN

B1.1 Sampling Methodologies

Procedures for the sampling process design are part of an overall DQO process completed during project planning. The Blackstone Project Manager is familiar with the previous documentation of contamination at the site. The factors to be considered in the design include possible sources, migration pathways, potential receptors, contaminants of concern, state programs likely involved, future land use, and the consequences of the use of false negative or false positive data points.

For EPA Brownfields Cleanup activities, a non-probabilistic sampling (judgmental) approach may be used to provide subjective information to fully delineate the horizontal and vertical extent of soil contamination identified in previous assessment activities. In addition, the non-probabilistic sampling approach will be used to determine the project decision of whether the site has been sufficiently cleaned up for future land use. A brief definition of the basic sampling approaches is provided below.

- Judgmental Sampling—Judgmental sampling is the subjective selection of sampling locations based on historical information, visual inspection, and the best professional judgment of the sampler.

The above approach can be combined with composite sampling in which two or more sample aliquots are mixed together. Compositing is useful when the degree of variability in a concentration is not a concern or when the samples are collected in a phased approach.

B1.2 Management of Investigation-Derived Waste and Decontamination Procedures

Investigation-derived waste (IDW) may consist of decontamination fluids, drill cuttings, purge/development water, excess sampled media (e.g., soil, sediment, water, etc.), disposable sampling supplies, and personal protective equipment (PPE) (e.g., gloves, respirators, etc.). As outlined in the EPA/540/G-91/009 document titled "Management of Investigation-Derived Wastes During Site Inspections", water containing contaminants, but not free product, may be disposed onto the ground provided the contaminated water is contained on the property, the discharge does not endanger public health or safety, and the discharge does not enter any surface water or tributary. Only IDW water will be disposed of in this manner. Any water accumulating in bermed areas where contaminated soil is stockpiled will be containerized, tested, and properly disposed of. The following goals pertain to IDW management:

- Leave the site in no worse condition than it existed prior to the site activity
- Remove wastes that pose an immediate threat to human health or the environment
- Leave wastes on site that do not require off-site disposal or extended containerization
- Comply with state and federal requirements to the extent practicable
- Minimize the quantity of wastes generated

Decontamination of personnel and equipment will be conducted in accordance with the site-specific health and safety plan and the Investigative Derived Waste SOP (Appendix F).

B2 SAMPLING METHOD REQUIREMENTS

Samples will be collected and handled in accordance with the following SOPs provided in Appendix F which were developed from state and national guidance:

- Standard Operating Procedure for Soil Sampling
- Standard Operating Procedure for Soil Screening with the Portable X-Ray Fluorescence (XRF) Device)
- Standard Operating Procedure for Soil Screening with a Photoionization Detector (PID),
- Standard Operating Procedure for Chain of Custody
- Standard Operating Procedure for Equipment Decontamination
- Standard Operating Procedure for Investigative Derived Waste
- Standard Operating Procedure for Lead-Based Paint Testing

Additional SOPs that address specialized sample collection and screening techniques may also be incorporated, if approved by the EPA Brownfields Project Officer. An amendment to this QAPP will specify any sampling methodologies used that are not listed above.

The Blackstone Project Manager will be responsible for identifying and taking corrective actions in accordance with Section C1.2 of this QAPP.

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

The samples will be handled in accordance with the Blackstone Chain of Custody (COC) SOP, (Appendix F) which was developed following state and federal guidance. Deviations from COC procedures will be noted in the report.

B4 ANALYTICAL METHODS REQUIREMENTS

Field measurements will be obtained in accordance with applicable SOPs and/or manufacturers' guidance and user manuals for the parameters being measured.

TestAmerica, Inc. has provided the QA Manuals for the fixed-base laboratory, dated July 12, 2018 found in Appendix E. The preparation and analysis of fixed-base laboratory samples is described in general in Appendix E, Section 19 of TestAmerica, Inc.'s QA manual. Specific method protocols will be within recommended procedures of standard methods. All samples will be analyzed within standard turnaround time unless the Blackstone Project Manager deems "rush" analyses are necessary to meet project goals.

B5 QUALITY CONTROL REQUIREMENTS

QC samples will be required to verify the validity of analytical results and, where QC issues arise, to assess whether samples were contaminated as a result of improper decontamination procedures, use of contaminated containers or preservatives, and/or introduction of contaminants during transportation of the samples to the laboratory. Field QC samples may include trip blanks, rinsate samples, and duplicates as appropriate. Duplicate samples may be collected to assess the reproducibility of the sampling procedures and analytical methods. Temperature blanks are included to verify sample preservation. Required field QC samples will be identified in the QAPP Amendment.

Trip blanks will be prepared by TestAmerica, Inc. and will be taken into the field by the sampling team to determine whether any field-related activities resulted in the introduction of VOCs that would jeopardize the validity of analytical results. Field blanks are samples prepared in the field to assess whether any contaminants were introduced by sample containers and/or preservatives. Rinsate samples are used to determine if decontamination procedures are being performed adequately to prevent cross-contamination between samples. The Blackstone Project Manager, in conjunction with the QA Reviewer, will evaluate the results of the trip, rinsate, and field blanks to determine if they are acceptable. If sample results indicate contamination of blank samples (detections above method reporting limits), sampling and analysis may be performed again for the associated target analytes. The Project Manager, in conjunction with the QA Reviewer, will make this decision.

Laboratory QC samples include duplicates, spikes, laboratory blanks, and performance evaluation (PE) samples as appropriate. All pertinent SOPs and guidance documents referenced in this QAPP will be followed to ensure QA objectives are met. Fixed-base laboratory QC procedures will be performed in accordance with the SOPs for the applicable analytical methods, TestAmerica, Inc. Quality Assurance Manual (July 12, 2018), (Appendix E).

The following QA/QC guidance documents will be implemented as appropriate to ensure all QA/QC elements are adequately addressed:

- *Guidance for the Data Quality Objectives Process* - EPA QA/G-4, OSWER. USEPA, February 2006.
- *EPA Requirements for Quality Assurance Project Plans* - EPA QA/R-5, March 2001.
- *Guidance for Quality Assurance Project Plans* - EPA QA/G-5, OSWER, USEPA, December 2002.

B5.1 Representativeness

All samples will be collected in such a manner (and at suitable locations) to accurately reflect the contaminant concentrations in the media from which they were taken at the time of sampling. Sample or measurement locations may be biased (judgmental) or unbiased (random or systematic), depending on the desired data use. The biased sampling will be performed for LRP delineation activities per IAC137, while unbiased sampling will represent the extent of contamination throughout the site.

Representativeness of the data is partially ensured by avoiding cross-contamination, adherence to standard sample handling and analysis procedures, and use of proper COC and documentation procedures. Representativeness may be assessed by comparing repeated analysis from the same sampling point over a period of time.

B5.2 Comparability

In order for one set of data to be compared with another, all analyses will be performed by accepted EPA or state methods and all analytical results will be reported in similar concentration units and format. Further, to ensure that subsequent samples are collected from the previous sampled locations for comparison purposes, specific sampling points will be documented using recognizable descriptions and identifiers or by Global Positioning System (GPS) methodologies.

B5.3 Completeness

In order for a set of data to be used with confidence to make a decision, the data must be complete (i.e., there must be enough valid data from analyses to support the decision). An integral part of obtaining adequate valid data will be to design the sampling network in such a manner that enough data are obtained to enable site decisions to be made, even if some of the data are determined to be invalid or cannot be collected due to unexpected field conditions. If an adequate degree of completeness is represented by the data set allowing site decisions to be made, as determined by the Blackstone Project Manager, the data will be considered complete. The Blackstone Project Manager will determine if invalid or missing data is critical to the decisions being made about the site, or if a decision can be made without the data. The Blackstone Project Manager will determine this on a case-by-case basis according to specific data missing, rather than determining completeness based on percentage of proposed samples collected. If an inadequate degree of completeness is represented by the data set, corrective actions (including resampling) may be necessary before an appropriate decision can be made.

B5.4 Sensitivity

Detection and quantification limits for sample data must be below the action levels specified in the nonresidential SWS as calculated according to IAC 137 (Appendix C). When the list contains more than one action level, the lowest level is chosen. Sensitivity can be affected by contamination as reflected in the method blank results. High method blank results are cause for reruns in sample preparation or sample analysis. Method detection limits for laboratory analyses are specified in TestAmerica, Inc.'s Quality Assurance Manual (July 12, 2018).

It is possible the action level for certain constituents, such as certain PAHs (e.g. benzo(a)pyrene, dibenzo(a,h)anthracene) may still be lower than the method detection limit. In these instances, the method detection limit for the analyte may be used as the action level.

B5.5 Precision

Precision describes the variability of a measurement system. Precision is typically an estimate by means of duplicate and replicate measurements and is expressed in terms of RPD. For field sampling, precision is increased by following SOPs and by collecting all samples using the same sampling procedures. Field QC samples collected to measure precision include field duplicate samples (i.e. transport and field handling bias) and include collocated samples (i.e. sampling and measurement precision). Field measurement precision is monitored by taking duplicate measurements at a frequency of 10% of the samples collected and is increased through proper operation and maintenance of field equipment.

Precision for VOCs is evaluated using the RPD between the results of the matrix spike (MS) and matrix spike duplicate (MSD) samples. This precision evaluation can also be performed using the RPD between a blank spike (BS) and blank spike duplicate (BSD). The spiked samples are laboratory samples that have been fortified. Precision for laboratory analysis will be measured as described in TestAmerica, Inc.'s Quality Assurance/Quality Control Manual (July 12, 2018) (Appendix E).

Precision for field work is evaluated by calculating the RPD between the results for the field duplicate samples. RPDs will only be calculated for results which are detected at a value greater than 5x the reporting limit. A RPD goal of +/- 50% for soils and +/- 35% for aqueous samples will be used for both field and lab analyses and will be included in the task assignment. Precision determined using RPD would be calculated as follows:

$$RPD = \left[\frac{2x(X_1 - X_2)}{(X_1 + X_2)}\right] x100$$

where: X_1 = analyzed concentration in the samples

X_2 = analyzed concentration in the duplicate

If RPDs greater than 50% for soils and 35% for aqueous samples are encountered, corrective action procedures will be implemented. Corrective actions would include evaluation of the sampling procedures, inspection of the sample matrix, and review of field screening results. Laboratory quality control statistics will be calculated per methods specified in Appendix E.

B5.6 Accuracy

Field blanks may be used to evaluate the purity of sample containers and chemical preservatives. In most cases, one field blank per sampling event will be sufficient. No other measures will be taken to evaluate accuracy that are directly associated with sampling and field procedures. For samples analyzed by TestAmerica, Inc. accuracy will be assessed and evaluated by laboratory personnel in accordance with TestAmerica, Inc.'s Quality Assurance Manual (July 12, 2018) (Appendix E).

B6 INSTRUMENT AND EQUIPMENT TESTING, INSPECTION AND MAINTENANCE REQUIREMENTS

Field equipment will be calibrated and maintained in accordance with the manufacturers' specifications and applicable Blackstone SOPs. Calibration and maintenance documents will be stored in the case alongside the associated field equipment or in a field log book as appropriate. Laboratory equipment will also be calibrated and maintained in accordance with the manufacturers' specifications and applicable analytical SOPs and applicable TestAmerica SOPs. Depending on the type of field equipment, critical spare parts such as tape, paper, pH probes, electrodes, batteries, and battery chargers will be kept within the associated field equipment case to minimize equipment downtime. Backup instruments, equipment, and additional spare parts will be available on site or within a 1-day shipping period to avoid delays in the field schedule.

B7 INSPECTION AND ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

Blackstone maintains centralized control of field sampling expendables, supplies, and materials for conducting environmental sampling through the position of Environmental Activities Field Coordinator. Only supplies and consumables that are of adequate quality to sustain confidence in the sample collection, processing, and laboratory analysis will be used. Purchased supplies and consumables will not be used until they have been inspected, calibrated, or otherwise verified to be in compliance with any standard specifications relevant to all calibrations or tests being performed and will be dedicated to that project. When possible, certified contaminant free sampling supplies and consumables (e.g. Voss® disposable bailers, Best® nitrile gloves) will be used and dedicated for one use at one location.

TestAmerica Inc. offers pre-cleaned sampling containers for use by field sampling personnel. Cleaning is verified by the companies' QA Managers. These containers are obtained from reputable container manufacturers and are cleaned to EPA specifications (Specifications and Guidance for Contaminant-Free Sample Containers OSWER Directive #9240.0-05A Dec 92). In addition, the Blackstone Environmental Activities Field Coordinator will visually inspect containers for gross contamination, necessary preservatives, appropriate size, number, and material for the required analyses.

B8 DATA ACQUISITION REQUIREMENTS

No data from other sources, except the EPA Brownfields ESAs and prior Blackstone initiated Hazardous Materials Surveys will be used for decision-making purposes. Any secondary (non-EPA Brownfields) information, including other analytical data, reports, photographs, maps, etc.

from additional sources referenced in reports compiled by Blackstone, may not have been verified by Blackstone. This information is mentioned for informational purposes only, and will be addressed as supplemental information. It is not to be used for decision-making purposes without verification by an independent professional who is qualified to verify such data or information.

B9 DATA MANAGEMENT

Data management will include data format preservation, allowing the City to compare information to IAC 137. Blackstone's Project Manager will be responsible for supervising the administrative support personnel in maintaining the project files for the duration of the project and shall not exceed five years with out resubmission of a QAPP and approval thereof by EPA. The project files will be kept in Blackstone's Iowa City office while the project remains active. Upon completion of the project, Blackstone will archive the project files until the completion of the project. After completion of the project, project files will be transferred to the City.

Blackstone will use desktop and portable laptop computers along with data loggers to record, process, and manage project data. The following software potentially will be used to process data: Access®, ArcGIS®, Aqtesolv®, AutoCad®, AutoDesk®, DQO/DEFT®, Excel®, Surfer®, VSP®, Word®, and IDNR's Tier 1 and Tier 2 software.

Laboratory data management will focus on a level requisite of EPA protocols and the standard methods. These procedures are set forth in Appendix E.

SECTION C - ASSESSMENT AND OVERSIGHT

C1 ASSESSMENT AND RESPONSE ACTIONS

C1.1 Performance and System Audits

Both internal performance and system audits may be conducted during field operations. Performance audits include verification that field sampling activities and measurements and laboratory analyses of performance evaluation samples are being conducted in accordance with the requirements of this generic QAPP and any QAPP Amendment. System audits involve a qualitative examination of an environmental data collection system including records, personnel, and QA management activities.

This section describes the selection of audit personnel, the scope of field and laboratory audits, audit frequencies, and typical audit reports for internal audits initiated by the Blackstone QA Reviewer.

The fixed-base laboratory performance and system audits will be as outlined in TestAmerica, Inc.'s Quality Assurance Manual (July 12, 2018) (Appendix E).

C1.1.1 Audit Personnel

The QA/QC Reviewer has the lead role in directing and executing all internal audit activities during an investigation. The QA/QC Reviewer is responsible for preparing an audit plan; coordinating and scheduling the audit with the project team or subcontractor; participating in the audit; coordinating the preparation and issuance of audit reports and corrective action request forms; and evaluating audit responses and resulting corrective actions.

C1.1.2 Audit Scope of Work

Performance audits of field activities will be conducted to evaluate compliance with the requirements of the generic QAPP and QAPP Amendment. Field system audits may include an examination of the following items:

- Sample collection records.
- Sample collection, handling, preservation, packaging, shipping, and custody records.
- Equipment operation, maintenance, and calibration records.

The laboratory performance and system audits by TestAmerica Inc. will be completed as outlined in TestAmerica, Inc.'s Quality Assurance Manual (July 12, 2018) (Appendix E).

C1.1.3 Audit Frequencies

As necessary, the generic QAPP and QAPP Amendment will provide a schedule for all planned audits to be conducted during the investigation. These audits may be required by

EPA or planned by QA/QC Reviewer. Audit frequency will depend on several factors. In selecting investigations for auditing, the QA/QC Reviewer will consider investigations with a large volume of work or those on which EPA has placed a high level of importance. The QA/QC Reviewer may also randomly select investigations for auditing.

Unscheduled follow-up audits may occur if any deficiencies are discovered during an audit or review. Follow-up audits serve to verify that all necessary corrective actions have been properly implemented to address deficiencies.

C1.1.4 Audit Reports

Audit reports will be prepared for performance and system audits of field and laboratory activities and all laboratory evaluation studies conducted under these Cooperative Agreements. Reports will be prepared by the QA/QC Reviewer. Audit reports will identify participants, describe the activity audited, summarize audit findings, and detail any deficiencies or deviations from protocol discovered during the audits, as well any corrective actions proposed. Any field or laboratory analytical data generated during performance evaluation must be validated. Validated dates will be included in the audit reports.

Audit reports are distributed to the Blackstone Project Manager and the Field Captain or laboratory QA manager, as appropriate. The QA/QC Reviewer has primary responsibility for ensuring audits are conducted thoroughly and properly. The Blackstone Project Manager and Field Captain or laboratory QA Manager is responsible for implementing corrective actions resulting from the audit. The QA/QC Reviewer is responsible for verifying recommended corrective actions have been implemented.

C1.2 Corrective Action

Corrective actions will be taken whenever problems appear to be adversely affecting data quality and/or when the resulting decisions may affect future response actions pertaining to the site. When such conditions are identified, the following corrective actions will be taken:

- Document that suspect data have been obtained.
- Review the system in question to ensure procedures were properly performed.
    - If procedures were not carried out properly, then document errors and repeat the procedures in accordance with proper methodologies, including all applicable quality control checks.
    - If any control checks gave out-of-control results, advise the project supervisor, and do not continue until the problem has been resolved.
    - If all of the control checks gave satisfactory results after corrective actions have been taken, document the corrective actions and continue.

C2 REPORTS TO MANAGEMENT

Reports describing the project activities, status, results of audits, corrective actions, needs for resolution among participating parties, and schedule changes will be distributed electronically and

in writing. Quality Assurance problems will be noted in the reports as needed. These are summarized below in the following table.

Table 4 - Routine Reports

Document	Party	Preparer	Distribute	Frequency
Grant Reports	City	Project Manager	Section A3.1-3	Quarterly throughout Grant and as determined by the City
Daily Job Reports	Blackstone	Environmental Activities Field Coordinator	Project Manager	Daily when field work in progress with logbook copies
Project Status Meetings	Blackstone	Project Manager	Section A3.1-3	Weekly during field activities otherwise monthly
Website for Community Outreach	City	Communications Specialist	Section A3.1-3, 5 Public PC	Updated at final report for project activities or as desired by the City
Project Closeout Report	Blackstone / City	Project Manager	Section A3.1-3	End of Project

SECTION D - DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VERIFICATION, AND VALIDATION

Data review and verification will be performed by a qualified laboratory analyst and the laboratory's operations manager as described in Section 19.14 of TestAmerica, Inc.'s Quality Assurance Manual (July 12, 2018) (Appendix E).

Verification of the data shall be the responsibility of the Blackstone Project Manager, who will review the data for completeness and obvious discrepancies. Field notes, COCs, activity summary forms, and soil boring logs will be compared for consistency and any anomalies documented by the Blackstone Project Manager. The Blackstone Project Manager will also inspect the data to provide final review and approval to ensure that the data meet the sampling requirements.

D2 VALIDATION AND VERIFICATION METHODS

The Brownfields cleanup demonstration of compliance with IAC 137 is the final step prior to redevelopment. The data will engage in an IAC 137 comparison with either one or a combination of background, statewide, or site-specific cleanup standards to be determined during the cancer and non-cancer health risk evaluation for the site and developed in the RE/RA Plan.

The Blackstone Project Manager will be responsible for validation of project implementation, conducting a direct comparison of the project records to the QAPP for the cleanup prior to writing the cleanup implementation report required by IAC 137. This will be initiated immediately upon completion of the field sampling activities on the property to be cleaned up. The cleanup implementation report will contain a section designated for data validation and verification. The data users will be provided with copies of the report.

Due to the limited nature of the validation, the QA Reviewer will conduct a minimum of one review per year, or as needed, of the Blackstone Project Manager's validation of project implementation. The QA Reviewer will evaluate the implementation of the following relative to field and management procedures as they apply to the Brownfields Cleanup. Data resulting from cleanup activities will be reviewed to evaluate conformance with the quality criteria set forth in the Cleanup DQO/QAPP. These evaluations will include, but are not limited to:

- Conformance to the QAPP's data quality objectives
- Conformance of the proposed sampling plan as detailed in Appendix B
- Conformance with sample handling protocols and holding times
- Results of quality control checks as they relate to field influences on data quality
- Results of calibration of instruments at bench mobilization and in the field from instrument records and field logbooks specific to the property enrolled and assessed

The Blackstone Project Manager will rely on standard methods conformance according to the TestAmerica laboratory SOP and their system of flagging data in the laboratory data packages to support valid analytical data.

D2.1 Field and Management

The review will specifically evaluate implementation of the sampling plan in Appendix B relative to field and management procedures as they apply to the Brownfields Cleanup Project. The quality of the resultant data will be evaluated in accordance with of the following:

- IAC 137
- Generic conformance to design parameters of the QAPP and DQOs
- Sampling design as detailed in Appendix B
- Sample collection procedures as prescribed in soil and groundwater sampling protocols of Appendix F and compared to field documentation and corrective audits of Section C1.2.
    - Sampling will be considered complete only if an adequate degree of completeness is represented by the data set allowing site decisions to be made, as determined by the Blackstone Project Manager
    - Sampling will be considered accurate if ninety-five percent (95%) of the soil and groundwater sampling protocols stipulated were used and documentation supports proper use
    - Sampling will be considered representative if seventy percent (70%) of the sample interval for soil was recovered and submitted.
- Sample handling protocols and chain-of-custody will be reviewed. Holding and transport times must be met for the sample to be considered valid
- Quality control checks conducted as they relate to field influences on data quality
- Calibration of instruments at bench mobilization and in the field from instrument records and field logbooks specific to the property enrolled and assessed

D3 RECONCILIATION WITH USER REQUIREMENTS

The Blackstone Project Manager will evaluate data for the completeness needed to achieve the project's goal. If the data quality indicators do not meet the project requirements outlined in the QAPP, the data may be discarded and re-sampling may occur. In case of a failure, the project team will evaluate the cause. If the failure is due to laboratory procedures or equipment, necessary corrective measures will be taken by the TestAmerica QA Manager and Blackstone Project Manager. If failure is associated with sampling, field procedures will be re-evaluated with any changes documented by the Blackstone Project Manager and included in the Assessment Report.

The primary purpose of the QA system is to define a process for collecting data that is of known quality, is scientifically valid, is legally defensible, and fully supports the decisions that will be

based on the data. To achieve this purpose, this QAPP requires the DQOs be fully defined in Section A7. All other parts of the QA system must then be planned and implemented in a manner consistent with the DQOs. The QA system components that follow directly from the DQOs include documentation and reporting requirements (Section A9); sample network design and sampling methods (Sections B1, B2, and B3); analytical methods requirements (Section B4); QC requirements (Section B5); and data reduction, validation, and reporting methods (Sections D1 and D2).

Once environmental data have been collected, reviewed, and validated, the data must be further evaluated to determine whether the DQOs identified in the QAPP Amendments and the QAPP have been met. Blackstone will follow EPA's data quality assessment (DQA) process to verify that the type, quality, and quantity of data collected are appropriate for their intended use. The DQA process involves first verifying that the assumptions under which the data collection design and DQOs were developed have been met, or taking appropriate corrective action if the assumptions have not been met. The DQA process then evaluates how well the data collected support the decision that must be made so scientifically valid and meaningful conclusions can be drawn from the data. These conclusions may be based upon a statistical evaluation, as allowed in IAC 137, of the data collected. To the extent possible, Blackstone will follow DQA methods and procedures outlined in EPA's *Guidance for Data Quality Assessment: A Reviewer's Guide* (EPA QA/G-9R) (February 2006) and *Data Quality Assessment: Statistical Methods for Practitioners* (EPA QA/G-9S) (February 2006).

If data quality indicators do not meet the Project's requirements as outlined in the QAPP, the data may be discarded and re-sampling and/or re-analysis may be required.

APPENDIX A

PROJECT LOCATION MAP

PROJECT LOCATION MAP

APPENDIX B

QAPP AMENDMENT TEMPLATE

QAPP Amendment
For
Quality Assurance Project Plan 411 E 15 Street as adapted from September 19, 2017 West Blum Cleanup Grant QAPP

EPA Region 7
BROWNFIELDS HAZARDOUS SUBSTANCE CLEANUP GRANTS
CITY OF DUBUQUE, IA
BF 97762001 and BF 97764401

DATE

I. APPROVALS

Ia.	**BLACKSTONE PROJECT MANAGER**	**Emily Smart**	**Date**
Ib.	**BLACKSTONE. QA/QC REVIEWER**	**Scott Mattes**	**Date**
Ic.	**CITY OF DUBUQUE PROJECT DIRECTOR/MANAGER**	**Steve Sampson Brown**	**Date**
Id.	**USEPA REGION 7 PROJECT OFFICER**	**Deborah Kennedy**	**Date**
Ie.	**USEPA REGION 7 QUALITY ASSURANCE MANAGER**	**Diane Harris**	**Date**

II. THIS QAPP AMENDMENT TO BE USED WITH:

Project Plan: West Blum Cleanup Project

Data Quality Objectives and Generic Quality Assurance Project Plan
EPA Brownfield Hazardous Substance Cleanup Grant

City of Dubuque

Dubuque, Iowa

December 9, 2019

Attachments to the Site Characterization and Contaminant Delineation Sampling Plan:

1. Contractor Certifications
2. Contractor HASP
3. Contractor SOP – identify and INSERT per specific Amendment design
4. Planned Disposal Facility
5. Anticipated Schedule of Activities

III. IN-PROCESS ADJUSTMENTS, CLARIFICATIONS & CORRECTIVE ACTIONS

		Checklist Modification Location		Adjuster		Approval	
Date	QA/QC Notation No.	Section	Page	Initials	Date	Initials	Date
	1						
	2						

IV. PROPERTY IDENTIFICATION

1. Facility (Property) Name: 4 1 1 East 15th Street
2. Parcel Numbers: 1024283001, 1024283002
3. Common Address: 411 East 15th Street
4. Project ID Number: 10140049
5. Access Agreement Signed By Owner(s) and Attached: ____No ____Pending ____Yes
6. Have Property Conditions changed since Phase I ESA? ____No ____Yes, discuss and attach

Primary Land Use Categories: The subject property is currently owned by Blum Properties and consists of a junkyard/scrap metal recycling center.

V. PREVIOUS ACTIVITIES

Multiple investigations have been performed on the subject property with as a part of the current EPA Brownfields Hazardous Materials Grant. A summary of previous investigations of the subject property is provided in the following list:

- HR Green, Inc., Phase I Environmental Site Assessment, June 13, 2016.
- HR Green, Inc., Phase II Environmental Site Assessment, October 4, 2016.
- HR Green, Inc., Analysis of Brownfields Cleanup Alternatives (ABCA), December 2, 2016.
- Advanced Environmental Testing and Abatement, Asbestos Inspection Report, July 30, 2015. •

HR Green, Inc., Hazardous Materials Inventory, September 18, 2016.

- Advanced Environmental Testing and Abatement, Asbestos Inspection Report – Aluminum Furnace, August 15, 2016.

VI. PROPERTY-SPECIFIC SAMPLING DESIGN(S)

SOP – identify and INSERT per specific Amendment design

VII. CHEMICAL ANALYSES SAMPLE PARAMETERS/BOTTLES

SOP – identify and INSERT per specific Amendment design

VIII.SAMPLE PRESERVATION AND HOLDING TIMES

SOP – identify and INSERT per specific Amendment design

IX. EQUIPMENT LIST

SOP – identify and INSERT per specific Amendment design T

.

IX. HEALTH AND SAFETY (A8.2.5, Default Approval Limited to D & D Modified Levels)

See attachment INSERT

X. UNANTICIPATED DEVIATIONS FROM DQO/QAPP REFERENCED

Variance: None.

Necessity To Brownfields Study: The proposed efforts are consistent with the approved Data Quality Objectives and Quality Assurance Project Plan (DQO/QAPP) objectives to evaluate the risk and feasibility of redevelopment options for the target property.

XI. FIELD OPERATIONS

SOP – identify and INSERT per specific Amendment design

XIII. QUALITY CONTROL CHECKS

SOP – identify and INSERT per specific Amendment design

ATTACHMENT 1

CONTRACTOR CERTIFICATIONS

ATTACHMENT 2

CONTRACTOR HASP

ATTACHMENT 3

CONTRACTOR SOP

ATTACHMENT 4

PLANNED DISPOSAL FACILITY

ATTACHMENT 5

ANTICIPATED SCHEDULE OF ACTIVITIES

APPENDIX C

IOWA LAND RECYCLING PROGRAM &
STATEWIDE RESPONSE ACTION STANDARDS (IAC 137)
IOWA ADMINISTRATION CODE (455H) CHAPTER 137

CHAPTER 137
IOWA LAND RECYCLING PROGRAM AND RESPONSE ACTION STANDARDS

567—137.1(455H) Authority, purpose and applicability.

137.1(1) *Authority.* This chapter is adopted under the authority of Iowa Code Supplement chapter 455H. These rules establish the policy and procedures for the voluntary enrollment of contaminated property in the "land recycling program" established under chapter 455H. These rules also establish the response action standards which participants must meet in order to qualify for a no further action certificate and the statutory protections and immunities which follow from it.

137.1(2) *Purpose.* Consistent with the declaration of policy stated in Iowa Code Supplement section 455H.104, these rules are intended to achieve the dual objective of addressing the current and future risks associated with contaminated property and thereby enhancing the market conditions which can lead to development of these properties into their highest productive use. These objectives can in part be met through a program which encourages voluntary participation by persons who may have a legal duty to address, in whole or in part, the contamination within an affected area as well as persons who might not have a legal obligation but who have an interest in development of enrolled sites. These rules attempt to provide a degree of certainty in the response action process as an incentive to participants and as a means of assisting participants in quantifying their financial investment. The following statement of principles is intended as a guide both in the interpretation of these rules and as a statement of the department's regulatory philosophy.

a. It is the objective of the department and these rules to establish a collaborative process between the participant(s) and department staff as the most effective means of achieving consensus and resolving disputes on issues which are not or cannot be fully defined and anticipated by rule.

b. Although participation in this program is voluntary, these rules establish basic standards which must be met in order to obtain regulatory closure from the department through issuance of a no further action certificate.

c. Although the scope of the response actions addressed under these rules may not in every case address all known or unknown releases within an affected area, it should be the objective of both the department and the participants to work together and to use all resources available to address all known releases within an affected area in the interest of protecting public health, safety and the environment as well as achieving regulatory finality.

137.1(3) *Applicability.* These rules shall apply only to releases of contaminants which are being addressed at enrolled sites. The department may in its discretion apply the response action rules in 137.4(455H) through 137.10(455H) to releases of contaminants at sites which are not enrolled. These rules do not in any way limit the statutory liabilities of participants or nonparticipants except as expressly provided within the context of enrollment and Iowa Code Supplement chapter 455H. Consistent with Iowa Code Supplement section 455H.505, these rules do not limit the authority of the department or the responsibility of statutorily responsible persons to provide notice of hazardous conditions under 567—Chapter 131 or to respond to new releases and undertake emergency response actions under 567—Chapter 133. For sites which are not enrolled, 567—Chapter 133 rules will remain in effect and for enrolled sites 567—Chapter 133 shall apply to the extent it is not inconsistent with this chapter.

567—137.2(455H) Definitions.

"Affected area" means any real property affected, suspected of being affected, or modeled to be likely affected by a release occurring at an enrolled site.

"Affiliate" means a corporate parent, subsidiary, or predecessor of a participant, a co-owner or co-operator of a participant, a spouse, parent, or child of a participant, an affiliated corporation or enterprise of a participant, or any other person substantially involved in the legal affairs or management of a participant as defined by the department.

"Background standard" means a standard which represents concentrations of contaminants which are naturally occurring or are generally present and not related to a readily identifiable release.

"Carcinogenic health risk" means the incremental risk of a person developing cancer over a lifetime (70 years) as a result of exposure to a hazardous substance, expressed as a probability such as one in a million (10^{-6}). The contaminant level for the probability value is derived from application of certain designated exposure assumptions and a slope factor.

"Contaminant" means any hazardous substance found in the various media of the environment.

"Contaminant of concern" means specific hazardous substances that are identified for evaluation in the risk assessment process. Identification can be based on their historical and current use at the site, detected concentrations in environmental media and their mobility, toxicity, and persistence in the environment.

"Cumulative risk" means a summation of cancer and noncancer risks, determined separately, based on exposure to multiple contaminants from the same medium and exposure of the same individual to contaminants in multiple media.

"Enrolled site" means any property which has been or is suspected to be the site of or affected by a release and which has been enrolled pursuant to this chapter by a participant.

"Environmental protection easement" means an institutional control created under Iowa Code Supplement section 455H.206 which is a statutorily authorized restriction on land use.

"Exposure pathway" means the course a contaminant of concern may take from its source area to an exposed organism. Each exposure pathway includes a source or release from a source, a point of exposure, and an exposure route.

"Exposure route" means the manner in which a contaminant of concern comes in contact with an organism (e.g., ingestion, inhalation, dermal contact).

"Free product" means a hazardous substance that is present as a nonaqueous phase liquid (e.g., liquid not dissolved in water) or is present as a solid in its original form as a product or waste material.

"Gross contamination" means contamination present at concentrations in an amount sufficient to reasonably expect that institutional or technological controls will not be adequately protective of human health or the environment.

"Group A, B, C, D and E chemicals" means hazardous substances which have been classified based on the weight of evidence of human carcinogenicity. Group A substances are carcinogenic to humans. Group B substances are likely to be carcinogenic to humans. Group C substances have suggestive evidence of human carcinogenicity, but not sufficient evidence to assess human carcinogenic potential. Data are inadequate to assess human carcinogenic potential for Group D substances. Group E substances are not likely to be carcinogenic to humans.

"Hazardous substance" means any substance or mixture of substances that presents a danger to the public health or safety and includes, but is not limited to, a substance that is toxic, corrosive, or flammable, or that is an irritant or that generates pressure through decomposition, heat, or other means. "Hazardous substance" may include any hazardous waste identified or listed by the administrator of the United States Environmental Protection Agency under the Solid Waste Disposal Act as amended by the Resource Conservation and Recovery Act of 1976, or any toxic pollutant listed under Section 307 of the federal Water Pollution Control Act as amended to January 1, 1977, or any hazardous substance designated under Section 311 of the federal Water Pollution Control Act as amended to January 1, 1997, or any hazardous material designated by the Secretary of Transportation under the Hazardous Materials Transportation Act.

"Hydraulic conductivity" means a measure of the capacity of a porous medium (rock or soil) to transmit water. It is expressed as the volume of water that will flow through a unit length of a unit cross-sectional area of the porous medium in a unit time with a unit head loss.

"Institutional controls" means a nonphysical action which restricts land use to reduce or eliminate exposure to the contaminants of an affected area.

"Lifetime health advisory level (HAL)" means an advisory level established by the United States Environmental Protection Agency which represents the concentration of a single contaminant in drinking water which is not expected to cause adverse health effects over lifetime exposure.

"Maximum contaminant level (MCL)" means a standard for drinking water established by the United States Environmental Protection Agency under the Safe Drinking Water Act which is the maximum permissible level of a contaminant in water which is delivered to any user of a public water supply.

"No further action certificate" means the same as no further action letter in Iowa Code Supplement section 455H.301. It is a document issued by the department to the participant certifying no further response action is required at an enrolled site for those conditions classified as no further action except the monitoring or the maintenance of institutional or technological controls when required.

"No further action certification" means the department has determined an enrolled site has met all standards applicable for the identified hazardous substances and no further response action is required except the monitoring or the maintenance of institutional or technological controls when required.

"Noncancer health risk" means the potential for adverse systemic or toxic effects caused by exposure to noncarcinogenic hazardous substances expressed as the hazard quotient for a hazardous substance. A hazard quotient is the ratio of the level of exposure of a hazardous substance over a specified time period to a reference dose derived for a similar time period.

"Nonresidential land-use area" means any area that is not a residential land-use area.

"Participant" means any person who enrolls property pursuant to this chapter. A participant is a participant only to the extent the participant complies with the requirements of this chapter.

"Point of compliance" means a location selected within the affected area where the concentration of contaminants of concern must be at or below the target levels established for that point.

"Point of exposure" means the location at which an individual or population may come in contact with a contaminant of concern from the enrolled site.

"Protected groundwater source" means a saturated bed, formation, or group of formations which has a hydraulic conductivity of at least 0.44 meters per day (m/d) and a total dissolved solids concentration of less than 2,500 milligrams per liter (mg/l).

"Receptor" means an individual or population that is or may be affected by a release from the enrolled site.

"Reference dose," expressed in units of milligrams per day exposure to the contaminant per kilogram of body weight of the exposed individual, means the amount of contaminant that an individual can ingest on a daily basis for a lifetime that is not likely to result in adverse noncancer health effects. A reference dose is protective of the entire human population, including sensitive subpopulations.

"Release" means any spilling, leaking, pouring, emitting, emptying, discharging, injecting, escaping, leaching, dumping, or disposing into the environment of a hazardous substance, including the abandonment or discarding of barrels, containers, and other closed receptacles containing any hazardous substance, but excludes all of the following:

1. Any release which results in exposure to persons solely within a workplace, with respect to a claim which such persons may assert against the employer of such persons.
2. Emission from the engine exhaust of a motor vehicle, rolling stock, aircraft, vessel, or pipeline pumping station engine.
3. The release of source, by-product, or special nuclear material from a nuclear incident, as those terms are defined in the federal Atomic Energy Act of 1954, if such release is subject to requirements with respect to financial protection established by the Nuclear Regulatory Commission under 42 U.S.C. § 2210 or, for the purposes of 42 U.S.C. § 9604 or any other response action, any release of source, by-product, or special nuclear material from any processing site designated under 42 U.S.C. § 7912(a)(1) or § 7942(a).
4. The use of pesticides in accordance with the product label.

"Residential land-use area" means an area zoned for residential use or an area where residential use currently exists, is planned, or is not otherwise precluded. In addition, a residential land-use area includes other areas where frequent, long-term, close contact with soils is likely to occur (e.g., playgrounds, sport fields, gardens, child care facilities).

"Response action" means an action taken to reduce, minimize, eliminate, clean up, control, assess, or monitor a release to protect the public health and safety or the environment. "Response action" includes, but is not limited to, investigation, excavation, removal, disposal, cleaning of groundwaters or

surface waters, natural biodegradation, institutional controls, technological controls, or site management practices.

"Risk evaluation/response action document" means a document based on the site assessment for the enrolled site which includes a risk evaluation, proposed response action, and proposed compliance verification strategy for the enrolled site.

"Site assessment plan" means the optional plan submitted to the department which lays out the rationale and the steps to be followed in the conduct of a site assessment for the enrolled site.

"Site assessment report" means the report of the site assessment which defines the nature and extent of contamination, identifies likely exposure pathways, and allows for characterizing potential and current exposure risks posed by the enrolled site.

"Site-specific standard" means a standard for a specific site which represents a concentration of a contaminant in a media of an affected area at which exposure through a specific pathway is considered unlikely to pose a threat to human health, safety, or the environment given site-specific factors related to contaminant transport and likely exposure.

"Slope factor" means an upper bound estimate that approximates a 95 percent confidence limit of the increased cancer risk from a lifetime exposure to a contaminant. This estimate is expressed in units of the proportion of a population that is affected per milligram per day exposure to the contaminant per kilogram of body weight of the exposed individual.

"Statewide standard" means a standard which represents a concentration of a contaminant in a specific media of an affected area at which normal, unrestricted exposure through a specific exposure pathway is considered unlikely to pose a threat to human health, safety, or the environment.

"Surface water" means general use segments as provided in 567—paragraph 61.3(1)*"a"* and designated use segments of water bodies as provided in 567—paragraph 61.3(1)*"b"* and 567—subrule 61.3(5).

"Target level" means a concentration of a contaminant of concern required to establish compliance with background, statewide or site-specific standards.

"Target organ" means the biological organ(s) most adversely affected from exposure to the contaminant of concern. A "reference dose" used to calculate noncancer health risk is normally established based on adverse impact to a target organ or organs from exposure to the contaminant of concern.

"Technological control" means a physical action whose main purpose is to reduce or eliminate exposure to the contaminants of an affected area.

567—137.3(455H) Enrollment in land recycling program.

137.3(1) *Property eligible for enrollment.* Unless excluded by statute or this rule and subject to eligibility conditions specified in this chapter, property which has been or is suspected to be the site of or affected by a release of a hazardous substance as defined in Iowa Code Supplement section 455H.103 is eligible for enrollment beginning October 27, 1998. The following sites shall not be enrolled in the land recycling program:

a. Property with petroleum releases associated with underground storage tanks subject to regulation under Iowa Code chapter 455B, division IV, part 8; and department rules under 567—Chapter 135. (However, property affected by releases of "regulated substances" from underground storage tanks other than petroleum as defined in rule 567—135.2(455B) subject to regulation under 567—Chapter 135 may be enrolled under this chapter.) Property enrolled and affected by a release from underground storage tanks of regulated substances other than petroleum will be subject to the response action standards in this chapter rather than those in 567—135.8(455B) through 135.12(455B). See also 567—paragraph 135.1(3)*"e."*

b. Property which has been placed or is proposed to be included on the national priorities list established pursuant to the federal Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 U.S.C. Section 9601 et seq. A property will be considered proposed at the time that a public notice of intent to list the property on the national priorities list is published in the Federal Register in accordance with 40 CFR 300.425.

c. An animal feeding operation structure as defined in Iowa Code section 455B.161.

d. Properties subject to administrative or judicial enforcement action by the department or the Environmental Protection Agency or subject to an administrative or judicial consent order addressing environmental conditions. These properties may be eligible for enrollment only with the written approval of and under such terms as determined by the enforcing agency.

e. Eligible properties which are or may be affected by or commingled with ineligible releases or conditions will be evaluated on a case-by-case basis to determine their appropriateness for enrollment. Only the eligible property and participant(s) will be afforded the benefits and immunities available under Iowa Code Supplement chapter 455H. Any protections provided by issuance of a no further action certificate will be limited by and may be subject to reopening due to future conditions associated with the ineligible release. Considerations for enrollment or exclusion include but are not limited to the following:

(1) The extent to which eligible releases and site conditions can be assessed and response action(s) designed and implemented independent of the ineligible releases and property.

(2) The extent to which the liability and other protections offered by Iowa Code Supplement chapter 455H and the conditions of a no further action certificate can reasonably be defined to apply to the eligible site without consideration of or dependence on future conditions associated with the ineligible release and property.

(3) The extent to which a participant is willing to conduct all response action(s) necessary to address the health, safety and environmental conditions implicated by both eligible and ineligible releases and conditions. The extent to which a nonparticipant responsible for the ineligible release and property can establish an intention and ability to cooperatively address and share costs associated with the commingled conditions and satisfy both the standards in this chapter and any other regulatory standards applicable to the ineligible release or condition.

137.3(2) *Enrollment policy and procedures.* Prior to enrollment, the applicant/participant(s) should have conducted sufficient preliminary site investigation and project planning to be prepared to show that a site is eligible for enrollment and the participant(s) is ready and capable of initiating and completing a response action in accordance with these rules. The applicant/participant(s) must submit a completed program application and participation agreement form as supplied by the department. The program application shall contain at least the following information.

a. An acknowledgment of access/control of the site signed by the participant if that person is a fee titleholder in the affected property; if the applicant/participant(s) is not a fee titleholder, then an acknowledgment by the fee titleholder of the affected property. If acknowledgment of access cannot be obtained, the participant must describe efforts to obtain access and reasons why it has been refused.

b. The name, address and other relevant information of each current and anticipated participant(s). The description should include a brief statement of the reasons for each person's participation including but not limited to that person's interest in and legal relationship to the property enrolled and the expected role and scope of any participation. Other persons who are not participants but who may have an interest in the project should be identified, such as state and local development agencies, community groups, and financing sources.

c. The applicant/participant(s) must demonstrate the presence of hazardous substances at concentrations that warrant response action(s) under the standards in this chapter. At a minimum the environmental condition to be addressed must be documented by the submission of a report which includes the following:

(1) Soil and groundwater samples of hazardous substances which have been analyzed by a laboratory certified under 567—Chapter 83 for the analytes being tested. The laboratory analysis should establish the presence of hazardous substances under conditions which exceed or are likely to exceed a statewide standard, if a statewide standard is available. Copies of the laboratory analytical report, boring logs and a site diagram showing the location of the sampling points in relation to the site should be included.

(2) A description of the current and historical uses of the property based on a reasonable and diligent inquiry. This must include a description of the following: known sources and probable locations of hazardous substances and probable location of the sources at the property which the participant proposes

to address as part of the project; a general description of the historical uses of the property and probable hazardous substances which could reasonably be associated with past land use; and a general description of the surface characteristics of the property and surrounding areas such as current zoning, residential, commercial and industrial uses, and current uses of adjoining properties.

d. Any assessments or other reports relating to contamination at the property in excess of a statewide standard or reportable under 567—Chapter 131 which are known to and within the control of the applicant/participant shall be submitted. If the applicant/participant intends to claim that information constitutes a privileged environmental audit as provided in 1998 Iowa Acts, House File 681, the applicant must notify the department of the claim and resolve the issue of privilege prior to submittal. The applicant shall not submit to the department a report or any part of a report which it claims to be privileged and any information submitted under this paragraph shall be deemed a nonprivileged submittal as provided in section 6, paragraph (1)*"a,"* of the Act. This provision does not relieve the applicant/participant of any obligation to notify the department of a hazardous condition as provided in Iowa Code section 455B.386 and rules under 567—Chapter 131.

e. A statement of the project objectives which includes the current use of the property, proposed development activities, and an expected time frame for meeting these objectives. The statement should include a general description of the scope of the proposed environmental condition to be addressed and a proposed schedule for initiation and submittal of site assessment activities pursuant to rule 137.8(455H). The statement should describe any foreseeable barriers toward achieving project objectives such as access to property, financing uncertainties, legal actions, allocation of responsibility amongst parties.

f. A list of all known permits and regulatory actions and directives associated with an environmental condition at the site. If any parcel of the proposed enrolled site is subject to any federal regulatory corrective action directives, administrative orders or judicial actions, these must be explained. The applicant must submit written proof that the appropriate federal regulatory agency has been notified of the applicant's desire to participate in the Iowa land recycling program. Objections, concerns or issues which could lead to disputes regarding dual or conflicting jurisdiction should be resolved prior to application, if possible, and before admission.

g. The department will respond in writing within 60 days of receipt of the enrollment application. The department will notify the applicant/participant(s) whether the site has been accepted and an expected time line for assignment of the project to a manager. If the site is not accepted, the department will notify the applicant of the reason(s). Upon notification of admission, the property shall be considered enrolled. Once the department has assigned the enrolled site to a project manager, the department will enter into a participation agreement with the participant(s).

137.3(3) *Enrollment fees and oversight costs.* A nonrefundable enrollment fee of $750 must be submitted with the program application. This fee is intended to cover the department's cost of reviewing the program application and a minimum amount of subsequent oversight costs. Subsequent fees in excess of the minimum $750 may be assessed for actual oversight costs incurred by the department as provided in this chapter. Department oversight activities may include, but are not limited to: review of documents, meetings with the participant(s), site visits, sampling, and laboratory costs related to verification of submitted materials. The total fees for oversight costs shall not exceed $7,500 per enrolled site. Fees shall be assessed and collected as follows:

a. Hourly billing rate. Project oversight fees shall be based on an hourly rate to cover wages and overhead costs of personnel employed by the department in the land recycling program. The department shall calculate and publish on an annual basis an hourly billing rate at which oversight fees shall be calculated.

b. Quarterly payments. The department shall bill the participant(s) on a quarterly basis for additional oversight costs beyond the review of the application incurred by the department. The participant(s) shall pay the department within 30 days after receiving the department's quarterly fee statement. If there is more than one participant, each shall be jointly and severally responsible for payment. The department will provide split billings if provided with an enforceable written contract allocating the fees amongst the participants.

c. Failure to pay required fees. If the participant(s) fails to pay department oversight fees that are required under this subrule, the department shall cease to provide oversight to the participant(s) and terminate enrollment of the site as described in subrule 137.3(7).

137.3(4) *Participation agreement.* All participants shall enter into a participation agreement. This agreement shall be executed at the time the project is assigned to a project manager. At a minimum, the agreement shall establish the following:

a. A requirement that the participant(s) agree and provide necessary documentation to ensure reasonable access to the affected property by department staff and other authorized representatives of the department.

b. A requirement that the participant(s) reimburse the department for the actual costs assessed as provided in 567—subrule 137.3(3).

c. A requirement that the participant(s) certify that the participant(s) has the financial means to complete the project based on an initial estimate of completion costs. The department may require modification and amendment of the financial certification at any stage in the project and may require the participant(s) to provide financial documentation as necessary to support the certification.

d. A requirement that the participation agreement include a general description of the scope of the project and the goals to be achieved, a general time frame for submission and review of documents in accordance with this chapter, allocation of responsibility amongst multiple participants and other appropriate milestones. Either the participant(s) or the department may request a meeting to develop a statement describing the scope, goals, and time frames for the project.

137.3(5) *Prioritization.* Eligible sites will be enrolled in the order in which they are received. The department reserves the right to elevate the priority of a given site if it determines the threat to the public health or environment or environmental conditions in combination with the development objectives consistent with Iowa Code Supplement section 455H.104 is significantly greater than those of sites with an earlier enrollment date.

137.3(6) *Withdrawal procedures.* Enrollment and continued participation in the program are voluntary. The participant(s) may withdraw the enrolled site and individual participants may withdraw from further participation in the land recycling program at any time upon written notice to the department. Any participant who withdraws an enrolled site from further participation in the program shall not be entitled to any refund or credit for the $750 enrollment fee and shall be liable for any oversight costs actually incurred by the department up to the cap of $7,500 per enrolled site. A participant who withdraws a site prior to completion of all response action(s) required by this chapter and issuance of a no further action certificate in accordance with rule 137.11(455H) forfeits all benefits and immunities provided by this chapter and Iowa Code chapter 455H. Prior to withdrawal, the participant(s) shall submit a plan, which must be approved by the department, for stabilization of conditions at the site or a justification for why further action to stabilize the site is not necessary. Participants shall be required to take such actions as the department determines necessary to stabilize conditions at the site, including, but not limited to, securing or properly abandoning monitoring wells, removing or otherwise properly disposing of all contaminated soil excavations, removing or properly disposing of exposed or exhumed contaminants, filling or properly fencing open excavations, and posting safety notices.

137.3(7) *Termination of enrollment.* Enrollment of the participant(s) may be terminated based on a finding of material noncompliance with department rules and statutory requirements including but not limited to the following:

a. Significant failure, after written notice, to comply with schedules for completion and submission of reports and implementation of response action(s) required by these rules or otherwise agreed upon in writing by the department and participants. Written requests for reasonable schedule extensions may be granted upon a showing of extenuating circumstances beyond the control of the participant(s) and the participant(s) agent/contractor.

b. Failure to proceed in a timely manner after written notice in performing the additional response action required due to a failure of technological and institutional controls pursuant to rule 137.7(455H).

c. Material misstatement or omission of fact in reports submitted to the department by the participant or agents of the participant.

d. Evidence that the site falls under one of the exclusion categories in subrule 137.3(1).

e. Failure to pay required fees to the department as required in subrule 137.3(3).

137.3(8) *Appeal rights.* The department will notify participant(s) of a denial of enrollment or of an intent to terminate enrollment and provide a statement of reasons. The participant(s) shall have a right to appeal the decision to deny enrollment or to terminate enrollment. Upon timely appeal, contested case procedures shall be initiated pursuant to 561—Chapter 7.

567—137.4(455H) Background standards.

137.4(1) *Purpose.* This rule defines the basis and procedure for establishing background standards in groundwater, soil, surface water, and air. Background standards represent concentrations of contaminants that are naturally occurring or generally present and not related to a readily identifiable release. Background standards provide a baseline for assessing impacts of contaminant releases from within the affected area.

137.4(2) *Determination of background standards.* Background standards shall be based on sampling at appropriate site-specific background locations. Background sampling locations shall be outside the influence of any possible contamination associated with releases occurring on the property in which the enrolled site is located. Sufficient supporting information shall be provided to demonstrate the appropriateness of background sampling locations. Appropriateness for background sampling locations has two aspects which shall be addressed:

a. Background samples shall be collected from a location which represents a true background condition with respect to the enrolled site. For example, a background groundwater sample will be collected from an upgradient location relative to groundwater movement.

b. Background samples will represent conditions which are comparable to the contaminated media being addressed. In the case of soils, samples from the affected area and the background areas will be comparable in physical, chemical, and biological attributes.

Sampling conducted for the purpose of establishing a background standard shall meet quality criteria specified for the site assessment, rule 137.8(455H). The minimum number of samples to be collected from the medium of concern for which a background standard is being established shall be consistent with rule 137.10(455H), regarding demonstration of compliance.

567—137.5(455H) Statewide standards.

137.5(1) *Purpose.* This rule defines the basis and procedure for establishing statewide standards for contaminants in groundwater, soil, and surface water. Statewide standards for groundwater and soil represent concentrations of contaminants in these media at which normal exposure via ingestion and dermal contact with soil is considered unlikely to pose a threat to human health. Statewide standards for surface water are based on protection of aquatic life and protection of human health. This rule also describes how air standards are to be addressed.

137.5(2) *Scope.* Statewide standards described herein address what are considered to be the most likely, normal exposure situations. Statewide standards for groundwater address direct exposure via ingestion to individual contaminants in the media of concern only. Statewide standards for soil address direct exposure to individual contaminants via ingestion and dermal contact. In the event exposure to multiple contaminants may occur or exposure from more than one medium may occur, statewide standards alone may not be protective of human health; therefore, cumulative risk standards must be met in accordance with subrule 137.10(7). In addition, the department may deny the use of the statewide standards prescribed herein and require the use of site-specific standards based on site-specific conditions pursuant to subrule 137.6(10).

Examples of exposure concerns not anticipated by the statewide standards might include, but are not limited to:

- Significant plant uptake of contaminants from soil or groundwater;
- Contaminants entering drinking water lines from contact with soil or groundwater;

- Ecological concerns, other than for surface water;
- Groundwater in a nonprotected groundwater source that is used or likely to be used for drinking water or other use.

137.5(3) *Establishment of risk-based contaminant concentrations.*

a. Risk-based concentration formula. Risk-based contaminant concentrations for soil and groundwater, except lead, shall be computed using the following formula, where appropriate:

(Formula I)

$$C = \left| \frac{RF \times AT \times 365 \text{ days/year}}{Abs \times [(ER_c \times EF_c \times ED_c) \div BW_c + (ER_a \times EF_a \times ED_a) \div BW_a] \times CF} \right|$$

NOTE: When a risk-based concentration is computed for two routes of exposure to the same medium (e.g., soil oral exposure and soil dermal exposure), the composite risk-based concentration equals the multiple of the risk-based concentration for each route of exposure divided by the sum of the risk-based concentration for each route of exposure.

Where: C = Concentration of contaminant (soil: mg/kg, water: mg/l)
RF = Risk factor

For protection from cancer health risks:

$RF = TR \div SF$

Where: TR = Target cancer risk (unitless)
SF = Slope factor $[(mg/kg)/day]^{-1}$ for a route of exposure; see paragraph *"c"* for source.

For protection from noncancer health risks:

$RF = THQ \times RfD$

Where: THQ = Target hazard quotient (unitless)
RfD = Reference dose (mg/kg)/day for a route of exposure; see paragraph *"c"* for source.

AT = Averaging time (years); time over which exposure is averaged and potential adverse effects may occur

Abs = Absorption factor (unitless); portion of exposed contaminant absorbed by the body

ER_c = Exposure rate by a child (soil: mg/day, water: l/day)

EF_c = Exposure frequency by a child (days/year)

ED_c = Exposure duration by a child (years)

BW_c = Body weight of exposed child (kg)

ER_a = Exposure rate by an adult (soil: mg/day, water: l/day)

EF_a = Exposure frequency by an adult (days/year)

ED_a = Exposure duration by an adult (years)

BW_a = Body weight of exposed adult (kg)

CF = Conversion factor: 10^{-6} kg/mg for soils; 1 (unitless) for water

b. Carcinogenic classification of chemicals. The potential carcinogenicity of chemicals will be based on the weight-of-evidence classification system utilized by the U.S. Environmental Protection Agency (EPA). Risk-based concentrations will be based on cancer health effects for individual chemicals that are classified as Group A or Group B. The risk-based concentration for an individual chemical will be based on noncancer health effects for chemicals that are classified as Group C, Group D or Group E. In the absence of such classification for a chemical, the Group D classification will be assumed. Noncancer risks for a Group A or Group B chemical will be included in the determination of cumulative noncancer risk in accordance with subrule 137.10(7), if a reference dose exists for that chemical. Cancer

risk associated with a Group C chemical shall be included in the determination of cumulative cancer risk in accordance with subrule 137.10(7), if a cancer slope factor exists for that chemical.

c. Source of toxicity values. EPA's Integrated Risk Information System (IRIS) shall be the primary source of information on toxicity factors (e.g., oral reference doses and oral slope factors), carcinogenic classification for chemicals, and the target organs. Such information that is not available on IRIS shall be obtained from other sources consistent with current EPA guidelines. The Iowa department of public health shall be consulted regarding toxicity values not available on IRIS. Absorption factors for dermal soil exposure shall be based on best available information, which will usually be obtained from EPA guidance documents.

137.5(4) *Statewide standards for groundwater.*

a. Protected groundwater source. Statewide standards for groundwater in a protected groundwater source will be the enforceable Maximum Contaminant Level (MCL) established by the EPA pursuant to the Safe Drinking Water Act, if an MCL exists. If no enforceable MCL exists, the statewide standard for chemicals will be the lifetime health advisory level (HAL) as provided in the latest "Drinking Water Regulations and Health Advisories" by the EPA's Office of Water or equivalent. If no MCL or HAL exists, the statewide standard for a chemical will be calculated using Formula I and input variables for groundwater ingestion in accordance with Table I.

b. Groundwater in a nonprotected groundwater source. The statewide standard for a chemical in groundwater in a nonprotected groundwater source will be five times the statewide standard for the chemical in a protected groundwater source or a risk-based concentration using Formula I with input variables specified in Table I, whichever is larger. The statewide standards for groundwater in a nonprotected groundwater source are based on groundwater ingestion only.

Table I

Input Variables for Risk-Based Statewide Standards for Groundwater from Protected and Nonprotected Groundwater Sources

Parameter	Units	Cancer Group	Protected	Nonprotected
TR	unitless	A, B	5×10^{-6}	1×10^{-4}
SF	$[(mg/kg)/day]^{-1}$	A, B, C	Chem.-spec.	Chem.-spec.*
THQ	unitless	C	0.02	0.1/1*
		D, E	0.2	1
RfD	(mg/kg)/day	C, D, E	Chem.-spec.	Chem.-spec.
AT	years	A - E	70	70
Abs	unitless	A - E	1	1
ER_c	l/day	A - E	1	1
EF_c	days/yr	A - E	0	0
ED_c	years	A - E	6	6
BW_c	kg	A - E	15	15
ER_a	l/day	A - E	2	2
EF_a	days/yr	A - E	365	365
ED_a	years	A - E	70	70
BW_a	kg	A - E	70	70
CF	unitless	A - E	1	1

The risk-based concentration using Formula I for Cancer Group C chemicals that have an SF value established per paragraph 137.5(3)"c"* will be the larger of a value based on the risk factor for protection from noncancer health risks with a THQ = 0.1 or the risk factor for protection from cancer health risks. Risk-based concentrations using Formula I for Cancer Group C chemicals that do not have an SF value

established per paragraph 137.5(3)*"c"* will be a value based on the risk factor for protection from noncancer health risks with a THQ = 1.

137.5(5) *Statewide standards for soil.* Statewide standards for chemicals in soil, except lead, will be calculated using Formula I based on incidental ingestion of soil and dust and dermal contact with soil with input variables in accordance with Table II. The statewide standard for lead in soil shall be 400 mg/kg.

Table II

Input Variables for Statewide Soil Standards

			Route of Exposure	
Parameter	Units	Cancer Group	Oral	Dermal
TR	unitless	A, B	5×10^{-6}	5×10^{-6}
SF	$[(mg/kg)/day]^{-1}$	A, B, C*	Chem.-spec.	Chem.-spec.
THQ	unitless	C*	0.1/1	0.1/1
		D, E	1	1
RfD	(mg/kg)/day	C, D, E	Chem.-spec.	Chem.-spec.
AT	years	A, B	70	70
		C, D, E	6	6
Abs	unitless	A - E	1	Chem.-spec.
ER_c	mg/day	A - E	200	560**
EF_c	days/yr	A - E	350	350
ED_c	years	A - E	6	6
BW_c	kg	A - E	15	15
ER_a	mg/day	A - E	100	400**
EF_a	days/yr	A - E	350	350
ED_a	years	A, B	24	24
		C, D, E	0	0
BW_a	kg	A - E	70	70
CF	kg/mg	A - E	10^{-6}	10^{-6}

The risk-based concentration using Formula I for Cancer Group C chemicals that have an SF value established per paragraph 137.5(3)"c"* will be the larger of a value based on the risk factor for protection from noncancer health risks with a THQ = 0.1 or the risk factor for protection from cancer health risks. Risk-based concentrations using Formula I for Cancer Group C chemicals that do not have an SF value established per paragraph 137.5(3)*"c"* will be a value based on the risk factor for protection from noncancer health risks with a THQ = 1.

**Dermal exposure rate is based on 2,800 cm^2 of exposed skin on a child with 0.2 mg/cm^2 of soil adhering to the child's skin and 5,700 cm^2 of exposed skin on an adult with 0.07 mg/cm^2 of soil adhering to the adult's skin per each dermal exposure event. A dermal exposure event is assumed to be one event per day of exposure.

137.5(6) *Statewide standards for surface water.* Water quality standards pursuant to 567—Chapter 61 shall be considered statewide standards for surface water. If a promulgated water quality standard does not exist for a contaminant of concern, the department may establish an appropriate standard in a manner consistent with 567—Chapter 61.

137.5(7) *Statewide standards for air.* Ambient air quality standards pursuant to 567—Chapter 28 constitute statewide standards for air. Air emission sources must meet air quality emission standards as set forth in 567—Chapters 20 through 31 inclusively, as applicable. Any relevant air quality standard that is subsequently promulgated by statute or rule shall become a statewide standard for air upon the effective date of adoption by the state. In the absence of applicable, adopted standards, site-specific air

standards must be met, in accordance with subrule 137.6(9), when air quality issues are addressed at a site.

137.5(8) *Point of exposure for statewide standards.* The point of exposure associated with the use of only statewide standards in the determination of compliance will be assumed to be anywhere and everywhere, except for surface water. The point of exposure associated with the use of statewide standards for surface water will be assumed to be the point of groundwater or other site runoff immediately before it discharges to the surface water body.

137.5(9) *Practical quantification limits.* In no case will the statewide standard be less than the practical quantification limit, as determined by the department.

137.5(10) *Maintenance of statewide standards.* The toxicity values, absorption factors for dermal exposure to soils, and promulgated standards that are a basis for statewide standards are subject to periodic revision due to actions not governed under this rule. The department in conjunction with the Iowa department of public health will maintain a guidance document that contains a current list of toxicity values, absorption factors for dermal exposure to soils, target organs for cumulative noncarcinogenic health risks, promulgated standards, and the resultant statewide standards that will be readily available to the public. This guidance document will reference all the sources of the information. In the absence of a dermal slope factor or a dermal reference dose for a chemical, the oral slope factor or oral reference dose will be used with adjustments made to account for differences in oral and dermal absorption rates in accordance with current EPA guidance. Statewide standards for individual sites will be locked-in at the beginning of the site assessment process (rule 137.8(455H)). If a statewide standard does not exist for a chemical, it will be the department's responsibility to establish a statewide standard, pursuant to subrules 137.5(4) and 137.5(5), for groundwater and soil, and to add the newly established statewide standard to the comprehensive list of statewide standards in the guidance document maintained by the department.

567—137.6(455H) Site-specific standards.

137.6(1) *Purpose.* As opposed to statewide standards, site-specific standards are derived by applying exposure and risk assumptions applicable to the conditions at a particular site. Like statewide standards, site-specific standards must always be shown to be protective of public health and safety and the environment. Statewide standards may be used in combination with site-specific standards to address different exposure pathways. Site-specific standards may be required to address exposure pathways which the department determines must be evaluated to be protective of human health, safety and the environment and for which statewide standards have not been established under rule 137.5(455H). Site-specific standards may involve development of target levels for contaminants of concern based on site-specific exposure assumptions for use in lieu of background or statewide standards. Site-specific standards may also include consideration of the actual or potential location where exposure to contaminants occurs or may occur, the likelihood of an exposure occurring, and the overall magnitude and extent of contamination. Site-specific standards may involve use of site-specific target levels for contaminants of concern alone or in conjunction with other site-specific criteria, such as the location where the standard is applied.

137.6(2) *General provisions.*

a. This rule establishes a minimum protocol that must be met at all enrolled sites which have not established compliance by application of background or statewide standards. Groundwater ingestion and soil ingestion pathway standards under this rule must be evaluated. Surface water and air quality standards under subrules 137.6(8) and 137.6(9) must be met whenever exposure concerns are evident and the participant or the department determines these pathways may present an unacceptable risk for current or future exposures. This rule is not intended to preclude the department or the participant from addressing other exposure pathways, and the department expressly reserves the right to require evaluation of other exposure pathways and compliance with site-specific standards developed for them, such as dermal contact, ingestion of vegetables containing contaminants from soil or irrigation water, migration of contaminants from groundwater or soil into water distribution lines or into air in a confined space, migration of contaminants from soil to groundwater, and migration of contaminants in a nonprotected groundwater source to a protected groundwater source. Participants must establish compliance with

standards applicable to all exposure pathways required by the department under this rule in order to qualify for no further action classification under rule 137.11(455H) unless granted a variance as provided in Iowa Code section 455H.205.

b. Site-specific standards are subject to the approval of the department. Assurances in the form of technological or institutional controls (rule 137.7(455H)) will be required, as needed, to ensure continued protectiveness of site-specific standards.

c. The following subrules provide options for the site-specific standards. The participant may select any of these options, or combinations thereof, for use as site-specific standards.

137.6(3) *Site-specific groundwater point of exposure.* A site-specific groundwater standard may be an appropriate target level applied at groundwater points of exposure that are limited by technological or institutional controls.

a. A point of exposure for groundwater is a location within the affected area where a well exists or could be placed (potential point of exposure). Where technological or institutional controls are determined to effectively restrict the placement of groundwater wells, the points of exposure apply outside the area of restriction. A sufficient number of points of exposure may be established for determining compliance such that compliance with appropriate target levels at these points will ensure compliance at all points of exposure. Normally a compliance point of exposure will be a location at the boundary of the area restricted by an institutional control where a groundwater well could be installed that would have the highest contaminant concentration. Generally more than one compliance point of exposure must be established due to uncertainties, such as spatial and temporal variabilities in groundwater flow and contaminant occurrence.

b. Target levels. The point of exposure target level for drinking water wells is the statewide standard applicable to groundwater ingestion or an alternative site-specific target level approved under subrule 137.6(10) or 137.6(11). The point of exposure target level for non-drinking water wells is the statewide standard applicable to nonprotected groundwater or an alternative site-specific target level approved under subrule 137.6(10) or 137.6(11). The point of exposure target level for nonused groundwater meeting the conditions in subrule 137.6(5) is the statewide standard for a nonprotected groundwater source.

c. Nonprotected groundwater sources. A nonprotected groundwater source which is affecting or likely to affect an existing drinking water well shall be required to meet the same site-specific standards, including point of exposure target level(s), as applied to a protected groundwater source.

d. Unless conditions can be demonstrated to be stable, predictive techniques in accordance with subrule 137.9(4) must be used to determine the future effects of groundwater contamination on existing drinking and non-drinking water wells and to determine the area predicted to exceed the point of exposure target level(s) where wells could be installed. When using predictive techniques, determining the location(s) where the applicable point of exposure target level is expected to be exceeded may involve comparison of the appropriate numerical standard to the predicted contaminant concentration at a passive monitoring well at the groundwater point of exposure. Alternatively, predictive techniques using site-specific models (paragraph 137.9(4)*"b"*) may involve simulation of pumping at a well located at the point of exposure, in which case the pumping rate used in the simulation shall be the rate that is reasonably possible for the area that yields water with the highest contaminant concentration. In absence of site-specific justification for doing otherwise, long-term pumping will be assumed to be at a rate of 100 gallons per day; the sustainable yield, if less than 100 gallons per day; or a reasonable, higher rate, if such a rate results in higher contaminant concentration.

e. Institutional controls. For a protected groundwater source or a nonprotected groundwater source as described in paragraph *"b"* above, institutional controls must be shown to effectively prohibit the installation of wells for the period of time in which contaminant concentrations might otherwise be expected to result in an exceedance of the appropriate target levels. For a nonprotected groundwater not described as in paragraph *"b"* above, a less stringent standard of effectiveness as well as the type of future well installation to be restricted may be utilized for those areas of potential concern. Unless there is a history of usage of what might otherwise be considered nonprotected groundwater or there is uncertainty as to the uniformity in the hydraulic characteristics of the nonprotected groundwater source,

notice to the authority responsible for permitting private wells under 567—Chapters 39 and 49 may be adequate especially if combined with a municipal or county ordinance prohibiting installation of private wells based on the availability of a public water supply.

137.6(4) *Site-specific groundwater point of compliance.* A site-specific standard may be established for a site-specific groundwater point of compliance that is different from a compliance point of exposure. A site-specific groundwater point of compliance must be used in conjunction with all groundwater compliance points of exposure pursuant to subrule 137.6(3) to provide an alternative monitoring location. Target levels for contaminants of concern at a site-specific groundwater point of compliance must be established using predictive techniques as specified in subrule 137.9(4). A target level established for a groundwater point of compliance must ensure that the appropriate target level at the groundwater compliance points of exposure will be achieved. A groundwater point of compliance shall be located on the contaminant migration path from the contaminant source to the point of exposure to the maximum extent practicable.

137.6(5) *Nonused groundwater in a protected water source.* Statewide standards for groundwater in a nonprotected groundwater source, pursuant to paragraph 137.5(4)*"b,"* may be used as target levels for contaminants in an otherwise protected groundwater source when groundwater in the affected area is not used and is not likely to be used in the future in accordance with the following. It must be demonstrated to the satisfaction of the department that contaminants from the enrolled site do not currently, and likely will not in the future, have an impact on any existing water supply well. Any detection, or predicted detection above the practical quantification limit, of a chemical that can be attributed to a release from the enrolled site will be considered to constitute an impact. In addition, it must be demonstrated to the satisfaction of the department that the impacted or potentially impacted aquifer is not a locally significant water resource. Factors that will go into this determination may include, but are not limited to:

- Existence of a nonimpacted public water supply in the potentially affected area;
- General availability of other water resources in the vicinity;
- Plans for development of public water supplies in the vicinity;
- Potential for use of the impacted aquifer as a water supply (e.g., yield, natural water quality); and
- Identification of the aquifer(s) commonly used for water supply in the vicinity.

A local ordinance prohibiting installation of private drinking water wells or notification to the local water utility and water permitting authority, or both, may constitute acceptable institutional controls for site-specific standards under this subrule.

The target levels that may be used in accordance with this subrule are based solely on groundwater ingestion. Compliance with this site-specific standard will not guarantee that contaminants in groundwater may not cause unacceptable exposure via other pathways (e.g., groundwater to air in a confined space, groundwater to surface water, or groundwater to a water distribution line).

137.6(6) *Site-specific soil standards based on land use and soil depth.* Site-specific soil standards based on land use and soil depth in conjunction with institutional controls may be used. Predetermined site-specific soil exposures based on land use and soil depth are provided in the following paragraphs. Lists of resulting site-specific soil standards for individual contaminants for these land-use and soil-depth categories will be maintained by the department in a guidance document and made readily available to the public. Use of these site-specific soil standards must be supported by appropriate institutional controls. Site-specific soil standards based on land use and soil depth, as described herein, address ingestion of and dermal contact with soil. Compliance with these standards will not guarantee that contaminants in soils may not cause unacceptable exposure via other pathways (e.g., ecological exposure, soil to groundwater, subsurface movement of vapors from soil to indoor air). In addition, the risk factors that form the bases for site-specific soil standards for individual contaminants, with the exception of some Group C chemicals, are the same as acceptable cumulative risk factors allowed for exposure to multiple contaminants in the same medium and multiple media. Therefore, compliance with site-specific soil standards for individual contaminants may not result in compliance with cumulative risk requirements pursuant to rule 137.10(455H).

a. Deep soil in a residential land-use area. Site-specific soil standards for deep soils equaling ten times the statewide standard for soils, except for lead, may be used. The site-specific standard for lead in deep soil in a residential land-use area shall be calculated using the most current version of EPA's Exposure Model for Assessing Risk Associated with Adult Exposures to Lead in Soil. Soils at a depth of ten feet and greater will normally be classified as deep soils. The department may deny the use of a deep soil standard associated with a residential land use or require a modification to the standard due to site-specific considerations including topography, development potential, and actual development plans. The use of a site-specific standard for deep soil in a residential land-use area shall be supported by an institutional control that permanently records the existence of contaminants above statewide standards in deep soils and restricts excavation resulting in deep soils being placed on the surface.

b. Nonresidential land use. The nonresidential land-use designation will be applicable to areas that are not classified as residential. Site-specific soil standards, except for lead, for nonresidential areas may be based on Formula I using the risk and exposure factors shown in Table III. A value of 1,100 mg/kg may be used as a site-specific soil standard for lead in soils less than 2 feet deep in a nonresidential land-use area. In lieu of this default site-specific lead standard, a site-specific standard for lead in soil less than 2 feet deep may be calculated using the most current version of EPA's Exposure Model for Assessing Risk Associated with Adult Exposures to Lead in Soil. The site-specific standard for lead in soils greater than 2 feet deep in a nonresidential land-use area shall be calculated using the most current version of EPA's Exposure Model for Assessing Risk Associated with Adult Exposures to Lead in Soil. The use of a nonresidential land-use classification must be supported by an environmental protection easement that prevents a change in land use to residential.

Table III

Input Variables for Site-Specific Soil Standards for Individual Contaminants for Nonresidential Area Land-Use Designation

Parameter	Units	Cancer Group	Soil Depth (ft.)	
			<2	>2
TR	unitless	A, B	1×10^{-4}	1×10^{-4}
SF (oral)	$[(mg/kg)/day]^{-1}$	A, B, C*	Chem.-spec.	Chem.-spec.
SF (dermal)	$[(mg/kg)/day]^{-1}$	A, B, C*	Chem.-spec.	Chem.-spec.
THQ	unitless	C*	0.1/1	0.1/1
		D, E	1	1
RfD (oral)	(mg/kg)/day	C, D, E	Chem.-spec.	Chem.-spec.
RfD (dermal)	(mg/kg)/day	C, D, E	Chem.-spec.	Chem.-spec.
AT	years	A, B	70	70
		C, D, E	1	1
Abs (oral)	unitless	A - E	1	1
Abs (dermal)	unitless	A - E	Chem.-spec.	Chem.-spec.
ER_c	mg/day	A - E	0	0
EF_c	days/yr	A - E	0	0
ED_c	years	A - E	0	0
BW_c	kg	A - E	15	15
ER_a (oral)	mg/day	A, B	100	330
		C, D, E	330	330
ER_a (dermal)	mg/day	A, B	660**	990**
		C, D, E	660**	990**
EF_a	days/yr	A, B	225	200
		C, D, E	200	200

Parameter	Units	Cancer Group	Soil Depth (ft.)	
			<2	>2
ED_a	years	A, B	25	1
		C, D, E	1	1
BW_a	kg	A - E	70	70
CF	kg/mg	A - E	10^{-6}	10^{-6}

NOTE: Oral and dermal factors are the same unless otherwise noted.

The risk-based concentration using Formula I for Cancer Group C chemicals that have an SF value established per paragraph 137.5(3)"c"* will be the larger of a value based on the risk factor for protection from noncancer health risks with a THQ = 0.1 or the risk factor for protection from cancer health risks. Risk-based concentrations using Formula I for Cancer Group C chemicals that do not have an SF value established per paragraph 137.5(3)*"c"* will be a value based on the risk factor for protection from noncancer health risks with a THQ = 1.

**Dermal exposure rate is based on 3,300 cm^2 of exposed skin on an adult with 0.2 mg/cm^2 of shallow soil adhering to the skin and 0.3 mg/cm^2 of deep soil adhering to the skin per each dermal exposure event. A dermal exposure event is assumed to be one event per day of exposure.

c. Restricted access land use. Rescinded IAB 7/21/04, effective 8/25/04.

137.6(7) *Site-specific cumulative risk for residential exposures to soil.* A cumulative risk standard may be used as a site-specific standard for soil in lieu of statewide standards that are provided in subrule 137.5(5) for individual chemicals and soil. Cumulative risk will be determined using the toxicity values and exposure factors (i.e., the input variables less TR and THQ) from Table II in subrule 137.5(5). Criteria for compliance with the cumulative risk standard are specified in subrule 137.10(7). No institutional control will be required with the use of this site-specific standard.

137.6(8) *Site-specific surface water standards.* The department will establish site-specific surface water standards at the request of the participant. The participant shall provide the department with information necessary to make this determination upon request from the department. Site-specific surface water standards will be generally equivalent to effluent limitations under a National Pollutant Discharge Elimination System (NPDES) permit pursuant to 567—Chapter 62. Mixing zones and allocation of contaminant loads in a surface water body will be considerations in attainment of in-stream water quality standards. If the site-specific surface water quality standards are met, best practical control technology currently available will not be imposed.

137.6(9) *Site-specific air standards.* If there are air quality concerns at a site, they will normally be addressed with site-specific standards until such time as ambient air quality or source-specific standards are adopted for hazardous air pollutants.

a. Explosivity. In no case shall contaminants from the enrolled site cause an explosivity level in a confined space of greater than 10 percent of the lower explosivity limit.

b. Background. In addition to the establishment of a background standard pursuant to rule 137.4(455H), a site-specific air standard may be set at twice the typical background level based on published information for a comparable setting, if approved by the department.

c. Health risk. The U.S. Department of Labor Occupational Safety and Health Administration (OSHA) limits for air contaminants pursuant to 29 CFR 1910, Subpart Z, may be utilized for site-specific standards in workplace settings where the OSHA standards are applicable and the contaminant of concern is used. For locations where OSHA standards are not applicable, site-specific standards for air in a confined space shall be risk-based using the chemical-specific toxicity values of inhalation unit risk (UR) and inhalation reference concentration (RfC) determined in accordance with paragraph 137.5(3)*"c."* Formulas II and III shall be used to calculate risk-based, site-specific air standards based on carcinogenic and noncarcinogenic effects, respectively, where C is the risk-based contaminant concentration in air. If a value for both RfC and UR exists for a compound, the risk-based site-specific standard will be the smaller of C resulting from Formulas II and III.

(Formula II)

$$C = AF \times TR \div UR$$

(Formula III)

$$C = AF \times RfC$$

The UR and RfC toxicity values are based on a continuous exposure of 20 cubic meters per day by a 70 kg adult. The adjustment factor (AF) in Formulas II and III may be used to adjust for site-specific exposure conditions. A target cancer risk (TR) of 10^{-4} shall be used unless another value is approved by the department.

d. Institutional or technological controls. Institutional or technological controls may be used to prevent future exposure to contaminants in air in confined spaces and will be required to prevent residential use of the affected area when a nonresidential air standard is used.

137.6(10) *Site-specific standards based on site-specific factors.* Numerical site-specific standards (i.e., target levels) for groundwater or soil may be established using site-specific exposure factors in Formula I. Site-specific pumping rates greater than specified in paragraph 137.6(3)*"d"* herein may be used when approved by the department. Site-specific exposure factors must be approved by the department. For the department to approve any such site-specific factor there must be well documented rationale for doing so and appropriate institutional or technological controls must be provided.

137.6(11) *Site-specific standards or approaches not anticipated by this rule.* Nothing in this rule precludes the use of site-specific standards derived in some way not anticipated by this rule, provided that the rationale is adequately presented and the approach is both approved by the department and provides a level of protection comparable to standards set forth under this rule.

567—137.7(455H) Institutional and technological controls.

137.7(1) *Technological controls.* The purpose of a technological control is to effectively sever a pathway by use of technologies such that an applicable receptor could not be exposed to hazardous substances above an applicable target risk level. Subject to limitations in this chapter, technological controls are an acceptable response action either alone or in combination with other remediation systems and institutional controls. The purpose of technological controls may be to control plume migration through use of containment technologies, barriers, or other methods, as an interim or permanent response action or to permanently sever a pathway to a receptor. Technological controls may also be appropriate to treat or control contamination at the point of exposure. Any technological control proposed as a permanent response action option without meeting the reduction in contaminant concentrations objectives must establish that the pathway to a receptor will be permanently severed or controlled. The effectiveness of a technological control must be monitored under a department-approved plan. The department may require reasonable proof of financial assurance when necessary to ensure that a technological control remain effective.

137.7(2) *Institutional controls.* The purpose of an institutional control is to restrict access to or use of an affected area such that an existing or future receptor could not be exposed to hazardous substances addressed by the controls for as long as the target level is exceeded at applicable points of exposure and compliance. Single or multiple institutional controls may be used alone or in combination and may also be employed with technological controls and response action to effectively achieve, maintain and enforce an approved level of risk reduction and risk management. The following enumeration of types of institutional and technological controls is not a finding that each is per se an effective control. The effectiveness of any institutional or technological control or combination of controls must be evaluated on a case-by-case basis and in accordance with specified conditions in this chapter. Institutional and technological controls include:

a. A state or federal law or regulation which can be shown to effectively achieve, maintain and enforce the required land-use restrictions and controls.

b. An ordinance of any political subdivision of the state which can be shown to effectively achieve, maintain and enforce the required land-use restrictions and controls.

c. A contractual obligation recorded and executed in a manner satisfying Iowa Code chapter 558. Recorded notices and affidavits, including a no further action letter as provided in rule 137.11(455H),

which do not create rights or obligations or restrict land use but serve to put current and future property owners on notice of present or future conditions within the affected area.

d. A control which the participant demonstrates to the department reduces or manages the risk from a release through the period necessary to comply with the applicable standards, including but not limited to informational devices such as public notices, informational registries, notices to regulatory authorities and continuing site activities such as periodic inspections, equipment repair and maintenance, and soil and groundwater monitoring.

e. An environmental covenant established in accordance with 2005 Iowa Code Supplement chapter 455I, 2005 Iowa Code Supplement section 455H.206, and 567—Chapter 14.

137.7(3) *Environmental covenants.* Participants may submit a draft environmental covenant to the department for review and approval in accordance with 567—Chapter 14.

137.7(4) *Public notification.* The department shall prepare a public notice prior to approval of any no further action classification which is conditioned upon use of institutional or technological control(s). The public notice will describe the results of the risk assessment conducted in the affected area, any proposed or completed response action, the vertical and horizontal extent and concentrations of existing soil and groundwater contamination in the affected area, and the actual and potential pathways of exposure the controls are intended to address. The notice will describe the purpose of the institutional and technological control(s) being proposed and the predicted period of coverage. The notice will provide the opportunity for members of the public to review department files, make written comments and request a public hearing. The department may schedule a public hearing on the basis of requests from the public and when it determines the particular remedial options proposed for a site warrant public consideration, for example, when issues of whether and to what concentrations gross contamination should be allowed to remain within the affected area given the relative effectiveness of institutional controls and other community concerns and development plans.

a. The notice will be served by certified mail on all property owners that the actual or modeled data indicates are or may be affected by the present or future conditions addressed by the control. The notice will be published in a newspaper of general circulation most likely to reach persons in the immediate locality.

b. If the controls are intended to restrict surface or subsurface future land use, the notice shall be sent to each local regulatory body having jurisdiction and control over or a direct interest in regulation of these activities. These may include but are not limited to municipal or county zoning boards, municipal building authorities, public utilities and economic development agencies. If the controls are intended to restrict groundwater use, the notice shall be sent to the county or city board of health responsible for private well permitting.

c. Failure to provide notice to an interested party shall not constitute a basis for invalidating a subsequently approved no further action classification.

137.7(5) *No further action certificates.* Any no further action certificate shall contain a specific reference to any applicable institutional and technological control and shall meet the requirements in rule 137.11(455H). The reference must identify the location of any recorded instrument, contractual agreement or other documents applicable to the control, provide a brief description of the terms of the control and, where appropriate, site diagrams.

137.7(6) *Enforcement of institutional and technological controls.* Institutional and technological controls which have been incorporated into a no further action certificate pursuant to rule 137.10(455H), or have been approved prior to issuance of a no further action certificate, may be enforced in Iowa district court by the department, a political subdivision of this state, the participant or any successor in interest to the participant as provided in Iowa Code section 455H.206(4). Enforcement of the terms of an environmental covenant shall be in accordance with 2005 Iowa Code Supplement chapter 455I, 567—Chapter 14, and the terms of the environmental covenant.

137.7(7) *Failure of an institutional and technological control(s).* The effectiveness of institutional and technological controls may be jeopardized for several reasons including situations where the technological controls are no longer effective in achieving their technical objectives, the validity of technological or institutional control is challenged due to a pending or final administrative or judicial

action or legislative action changing its regulatory effect (e.g., change in an ordinance), or persons fail to comply with the terms of the institutional or technological control. The effect of the failure of a technological or institutional control to achieve its intended purpose is to remove the no further action classification and put all interested parties in the same position had the no further action classification not been made. When the department has reason to believe technological or institutional control(s) is jeopardized or determines that the control is no longer effective, the following policy and procedure shall apply:

a. The department shall make reasonable efforts to provide notice of the failure or noncompliance to the participant(s), protected parties, persons having legal standing to enforce the terms of the controls, other persons who may be legally responsible for contamination at the site and persons legally obligated to comply with the terms of the controls. The notice shall inform these parties of the consequences of failure of the controls and provide the opportunity for one or more of them to correct the deficiency by taking further response action or undertaking enforcement action to obtain compliance with the terms of the controls.

b. The participant(s) and other persons legally responsible for contamination at the site shall have primary responsibility to correct deficiencies or seek enforcement of the terms of controls, if they wish to maintain a no further action classification and any attendant statutory protections. The department may in its discretion seek enforcement of controls where persons fail to comply with the terms when it determines there is a strong likelihood of success, other participant(s) or legally responsible persons are unable or unwilling to undertake enforcement, and utilization of the controls remains consistent with these rules and site conditions currently in effect at the site. However, the department is not obligated to seek enforcement of the terms of any technological or institutional controls nor does the election not to undertake enforcement constitute a defense to further action by responsible parties or a basis for challenging the rescission of the no further action classification.

c. The department may also elect to require statutorily responsible parties to correct the deficiency as an alternative to rescinding the no further action classification.

d. Failure of a participant to timely undertake additional response action and response may result in termination of enrollment and loss of benefits under these rules and Iowa Code Supplement chapter 455H. Any person found to have intentionally violated an environmental protection easement or other institutional or technological control, whether included in a no further action letter or as part of an approved response action, may lose any of the benefits under these rules or Iowa Code Supplement chapter 455H.

137.7(8) *Modification and termination of institutional and technological controls.* A participant or successor in interest to a participant, or an owner of property subject to an institutional or technological control, may seek approval from the department for the removal, discontinuance, modification or termination of an institutional or technological control. The person must demonstrate that the control in its present form is no longer required to ensure compliance with applicable standards. The person seeking revision must undertake sufficient risk assessment and provide sufficient assessment data to establish that the applicable compliance standards can be met based on the proposed modification. The department may also determine based on a revised assessment that the applicable controls are no longer effective to meet compliance standards and may require other response action. The department shall issue an amendment to any previously issued no further action letter specifying the approved modification of the institutional or technological controls. Modification and termination of an environmental covenant shall be consistent with these rules and shall conform with 2005 Iowa Code Supplement chapter 455I and 567—Chapter 14.

567—137.8(455H) Site assessment.

137.8(1) *Purpose.* The purpose of the site assessment is to define the nature and extent of contamination, along with identifying likely exposure pathways, with the aim of characterizing potential, current and future risks and making an informed decision concerning an appropriate response in the context of probable future land uses at the site and in the surrounding area. Assessment is to be conducted with the recognition that contaminant fate and transport may alter the current areal extent

and depth of contamination. It is recognized that the scope of such an assessment may be appropriately varied dependent upon interrelated factors including the nature and severity of the contamination, the complexity of specific details of the site and its setting, and the nature of the chosen response, if known.

137.8(2) *Site assessment plan.* The participant is encouraged, but not required, to submit for department review a site assessment plan, prior to proceeding with the site assessment. Participants choosing to initiate site assessment without department review and approval of a work plan shall notify the department in writing of their intentions. Likewise, participants choosing to proceed to the risk evaluation/response action phase in accordance with rule 137.9(455H) without seeking review of the site assessment report shall give prior notice to the department of their intentions. The notice shall include a schedule for implementation and completion, a description of the area to be assessed and the scope of the proposed assessment to be undertaken, any planned construction activities in the affected area and a proposed date for submission of the site assessment report for department review. If the notice includes an intention to go directly to the risk evaluation/response action phase, it shall also include a general description of the site assessment results, a schedule for submission of the risk evaluation/response action document and the reasons for not requesting department review and approval of the site assessment report.

The plan is intended to lay out the rationale to be followed in the conduct of the site assessment. The purpose for this optional stage is to provide an opportunity for the participant and the department to reach a consensus regarding the appropriate scope of the site assessment. The development of a consensus should serve to diminish the likelihood that the department will find the final site assessment to be deficient and, for the benefit of the participant, to avoid the expenditures and time associated with the collection of what may ultimately prove to be unnecessary data.

In order to accomplish this, it is suggested that the plan should address relevant, known characteristics related to the site and its history as well as plans for addressing pertinent details spelled out in the subsequent sections on the site assessment and the site assessment report. Departmental review may result in suggestions from the department regarding perceived shortcomings or proposed activities which are deemed to be unnecessary.

The participant may find it desirable to conduct some preliminary investigation in order to develop a site assessment plan.

137.8(3) *Site assessment details.* In order to meet the stated purpose of the site assessment, it will be necessary to characterize numerous attributes related to the enrolled site and its setting. The following objectives are intended to provide a framework in which to accomplish this purpose. It is recognized that these objectives may exceed the appropriate scope of some site assessments and that there may be situations in which it may be necessary to define additional objectives. Any such deviation would preferably be addressed in a site assessment plan. The department may also develop guidance documents that recommend more specific procedures for accomplishing site assessment objectives. Such guidance documents will be readily available to the public. In general, an acceptable site assessment should address the following items.

a. Identify and address the medium or media of concern associated with the contamination situation for which the site is enrolled. The regulatory classification or jurisdiction of contaminants shall be indicated if applicable and, if known, e.g., the compound is regulated under the Resource Conservation and Recovery Act (RCRA), Toxic Substances Control Act (TSCA), or Federal Insecticide, Fungicide and Rodenticide Act (FIFRA).

b. Characterize the nature, extent, and degree of contamination in both horizontal and vertical dimensions. This should involve appropriate sample numbers and locations within the contaminated area and beyond the area contaminated in excess of the background or statewide standard. Analyses should be conducted for the contaminants of concern, breakdown products, and other contaminants likely to be present at significant levels. The department may also require analyses for additional contaminants which are not the focus of enrollment in the program, but which may be of special concern. Special concerns might include waste handling or treatment problems posed by the additional contaminants, or unacceptable risks remaining unaddressed within the affected area, due to the presence of the additional contaminants. In the case of groundwater, attention should also be given to the possibility of contaminant

accumulation in strata overlying confining layers and to the possible presence of non-aqueous phase liquids (NAPL). In the case of groundwater, more than one round of sampling shall be incorporated, appropriately separated in time. In the case of soils, particular attention should be given to characterizing shallow soil contamination, from zero to six inches in depth.

c. Characterize the nature of the source of contamination or propose a conceptual model explaining the presence of the contamination of concern.

d. Characterize local contamination maxima or hot spots for the purposes of evaluation against relevant standards and to identify handling or treatment concerns that they may pose.

e. Characterize the stratigraphy. This should be done to a depth extending to the first significant confining layer below the deepest contamination. Descriptions should rely primarily on results gathered in the site assessment, but relevant reference materials or geologic logs from other sources may be incorporated as a supplement.

f. Characterize the hydrologic properties of the site and its vicinity to a distance appropriate to the fate, transport and exposure concerns associated with the site. This characterization should consider both horizontal and vertical components of groundwater movement as well as other influences on groundwater hydrology such as pumping wells, injection wells, surface water bodies, effects of seasonal or precipitation-driven variability, and possible aquifer interconnections, including those related to existing or abandoned wells. Water level measurements, related to a common datum, screening of appropriate depth intervals, and determination of hydraulic conductivity will generally be considered as necessary.

g. Characterize physical and chemical properties of the site and its environs associated with contaminant fate and transport, e.g., percent organic matter, redox potential, soil bulk density, and transmissivity.

h. Characterize topographic and cultural features of the site and its immediate vicinity. Cultural features may include, but not be limited to, buildings, basements, paved areas, roadways, utilities, storage tanks and associated piping, piles, impoundments, wells, and waste disposal systems.

i. Evaluate concerns related to whether the contamination situation is dynamic or stable; if dynamic, address fate and transport and breakdown products appropriately.

j. Identify and characterize receptor or exposure concerns. This most clearly involves concerns for drinking water and exposures to contaminated soils, as suggested by the statewide standards, but additional concerns should be identified and addressed by the participant or the department, as the situation warrants, e.g., vapors to basements, threats to water supply lines, threats to surface waters, or environmental threats.

k. Characterize current and probable future uses of the site and its surroundings. If probable future uses differ significantly from current uses, then characterize them separately and conduct the assessment in a fashion which addresses concerns arising from the possible change in use.

l. Evaluate the potential for contaminants to migrate from one medium to another. The following subparagraphs prescribe requirements for assessing potential migration of contamination from one medium to another. Requirements in the following subparagraphs may be waived if it can be demonstrated in accordance with procedures established in 567—Chapter 135 or the latest version of ASTM Standards related to the Phase II environmental site assessment process that migration of contamination from one medium to another will not cause a violation of the applicable standard in the receiving medium. The assessment activities prescribed in the following subparagraphs are intended to determine if significant migration of contamination from one medium to another has occurred. If evidence of significant migration of contamination from one medium to another (i.e., generally a contaminant concentration in the receiving medium in excess of the statewide standard) is discovered, full-scale characterization of the receiving medium may be required.

(1) The water from any pond or lake on the site or within 300 feet of the site shall be sampled and analyzed for the contaminants of concern, if it is reasonably possible that contaminants from the site could impact the pond or lake. Any surface stream that runs through the site or within 300 feet of the site should be sampled at a location downstream of any potential impact from the site and analyzed

for the contaminants of concern. Depending on the characteristics of the contaminants (e.g., solubility), associated sampling and analysis of sediments may be required.

(2) Groundwater at the location most likely to be impacted by each known substantial area of soil contamination shall be sampled and analyzed for the contaminants of concern. If the area of soil contamination exceeds 10,000 square feet, additional groundwater samples may be required.

(3) Soil vapors in each area that is most likely to be impacted by known groundwater or soil contamination shall be sampled and analyzed for the volatile organic contaminants of concern. If the area of soil or groundwater contamination exceeds 10,000 square feet, additional soil vapor samples may be required. If vapors may be impacting an existing enclosed space, a soil vapor sample shall be collected from a location that is most likely to have vapor contamination adjacent to the enclosed space.

If the potential for the existence of problematic concentrations of the vapors in the enclosed space cannot be dismissed based on soil vapor sampling, sampling and analysis of vapors inside the enclosed space may be conducted to determine whether or not a problem exists. Appropriate measures for distinguishing between contaminant vapors originating from within the enclosed space versus those from the external sources that are under investigation may be made with the approval of the department.

Ambient air sampling may be required if a very large area or extremely high concentrations of highly volatile contaminants exist in shallow soil or evidence of vapor contamination exists, such as odors or a high vapor reading on a vapor-screening instrument.

(4) If a water line exists within the zone of known organic contamination of soil, groundwater or soil vapor and the potential for significant diffusion of contaminants into the water line cannot otherwise be dismissed, a sample from the water line shall be collected at the nearest location where any impact may exist and that sample shall be analyzed for the organic contaminants of concern. All such samples should be collected at times following minimum movement within the water line (e.g., early morning following a weekend).

137.8(4) *Site assessment report.* The site assessment report shall include the presentation of all information gathered relative to the foregoing description of the site assessment, arranged in appropriate sections of the report. It shall include a summary of preliminary information on which the site assessment is based, e.g., background and site history. The report shall discuss the sampling strategy and methods used in the assessment. The department encourages the use of innovative or screening techniques to expedite investigations and to control costs, provided that such techniques are approved by the department and are supported through verification by accepted scientific practices. The report shall also include a description of the quality assurance/quality control (QA/QC) protocols followed during the investigation. QA/QC protocols shall be consistent with accepted scientific practices, including those set forth in appropriate EPA or ASTM guidance or otherwise approved by the department.

The presentation should be organized so as to facilitate the assimilation of information by the reader. Maps to be presented, as appropriate, might include maps illustrating the location of the site in a larger geographical context; maps showing cultural features associated with the site and its environs; maps illustrating the contamination extent and concentration in three dimensions; maps illustrating the site hydrology in three dimensions; and maps illustrating receptors, potential receptors, and relevant pathways of exposure. Cross-sectional diagrams should be included to illustrate stratigraphy, geological boring information, and hydrologic and contaminant factors with depth. Tables and graphs should be designed for the purpose of summarizing data in a meaningful fashion, including information about successive rounds of sampling. Appendices should include well logs, copies of laboratory analytical reports, and raw data used to calculate parameters presented elsewhere in the report. Appended material shall be labeled in a fashion permitting the cross-referencing of appended materials and the body of the report.

137.8(5) *Approval of site assessment report.* The department suggests, but does not require, that the site assessment report be approved prior to proceeding with the subsequent risk evaluation/response action phase. Unless notice has already been given prior to initiation of the site assessment, participants choosing to proceed to the risk evaluation/response action phase without department review and approval of the site assessment report must notify the department in advance as provided in subrule 137.8(2).

137.8(6) *Public notification.* Before or upon completion of the site assessment, the participant shall provide the department with the names and addresses of the owners and occupants of all property adjacent to the site enrolled in the land recycling program and any additional properties where contaminants from the enrolled site have migrated or are likely to migrate in the future. The department shall notify by direct mailing all such property owners and occupants, the city or county in which the property is located, and officials of any potentially impacted public water supply of the site's enrollment in the land recycling program and of the scope of work described in the participation agreement. The department shall give the notified parties the opportunity to obtain updates regarding the status of activities relating to the site that is enrolled in the land recycling program. The department may also require the participant of a site enrolled in the land recycling program to publish public notice in a local newspaper if the department determines that widespread interest in the site exists or is likely to exist. The department may provide additional opportunities for public participation if, after consultation with the participant, the department determines such opportunities are warranted.

567—137.9(455H) Risk evaluation/response action.

137.9(1) *Purpose.* The purpose of risk evaluation/response action is to utilize information from the site assessment as a basis for:

a. Determining whether current exposures result in risks deemed to be excessive, based on evaluation against appropriate background, statewide, or site-specific standards.

b. Determining whether future exposures may result in risks deemed to be excessive, based on evaluation against appropriate background, statewide, or site-specific standards. This will likely include:

(1) Evaluation of potential changes in usage, e.g., installation of a new well, change in land use, or other activities, which result in unacceptable, potential exposures not evaluated as current exposures, and

(2) Evaluation of exposure concerns related to the movement of contamination such that potential exposures might arise which are not considered under current exposure assumptions, e.g., groundwater plume migration creating a potential for future contamination of existing wells or creating newly contaminated areas in which new well installation may result in unacceptable exposures.

c. Proposing an appropriate and acceptable response action or strategy to address the identified, unacceptable exposures or potential exposures.

d. Establishing the test criteria to be applied under rule 137.10(455H) for determining final compliance with the selected standard. In some cases this may consist of proving that standards are currently met; in other cases it may result in an assessment of whether the response action succeeds in bringing about compliance with a selected standard.

The risk evaluation/response action is intended only for application to the specific contaminants and situations for which the site is enrolled.

137.9(2) *Risk evaluation.* The risk evaluation/response action document shall identify all locations or areas, and associated exposure pathways, where exposure currently exceeds a statewide standard or where a statewide standard may be exceeded in the future, due to either a change in exposure-related usage or contaminant migration. Current and future exposure pathways shall be evaluated and presented separately. This evaluation shall not be limited to exposure pathways for which the department has formulated risk-based values in rule 137.5(455H) (the statewide standard) or 137.6(455H) (the site-specific standard) but should include any pathway related to the situation for which the site is enrolled, for which a no further action certificate is sought, or for which an unacceptable risk may now or in the future exist, e.g., high concentrations of volatile compounds in proximity to a confined space, high concentrations of solvents in proximity to a water distribution line, or environmental concerns unrelated to human health.

In a case where a background standard is to be applied and there is no violation of a statewide standard, it will be necessary to identify only locations or areas where the background standard is exceeded.

In some instances it is anticipated that the risk evaluation may be appropriately abbreviated from the preceding description, based on the specific details of the contamination and the proposed response

action. Participants are strongly urged to discuss the appropriate scope of their risk evaluation with the department.

137.9(3) *Establishing cleanup standards.* The risk evaluation/response action document shall identify the cleanup standards to be applied in accordance with rule 137.4(455H), 137.5(455H), or 137.6(455H) of this chapter, outlining respectively the background, statewide, or site-specific standards. These standards may be applied in any combination to address specific components of the contamination problem for which the site is enrolled. If cleanup standards other than those specifically formulated under the statewide standard (rule 137.5(455H)) are to be applied, then the rationale behind the determination of such standards shall be justified, in the document, to the department's satisfaction.

137.9(4) *The use of models.* The department recognizes that the use of numerical models will likely be necessary in order to evaluate potential future exposures or that models may be used to develop target levels.

a. Standard models. Standard models may be used to predict future contaminant concentrations at potential points of exposure to contaminants or at other locations used for determining compliance when such models are appropriate, as determined by the department. Applicable Tier 2 models approved for use in accordance with 567—Chapter 135 for underground storage tanks (USTs) and applicable Tier 2 models provided in American Society for Testing of Materials (ASTM) standards are acceptable standard models. Models which provide a two-dimensional representation of groundwater flow will not be considered to be appropriate when significant three-dimensional components to groundwater flow are anticipated. Default values for input parameters for ASTM and UST Tier 2 models, as provided in applicable ASTM standards and approved for use in accordance with 567—Chapter 135, may be utilized without approval by the department. The department will maintain a guidance document which includes a list of other chemical-specific default values for all chemicals having statewide standards. The use of other, site-specific input parameters is addressed under site-specific modeling in paragraph *"b"* below.

b. Site-specific models. Site-specific models may be used to predict future contaminant concentrations at potential points of exposure to contaminants or at other locations used for determining compliance when such models are appropriate, as determined by the department. Site-specific models may include standard models with site-specific input parameters or models utilizing more sophisticated analytical techniques. The department will utilize versions of A Modular Three-Dimension Finite-Difference Ground-Water Flow Model (MODFLOW) as developed by the United States Geological Survey in conjunction with A Modular Three-Dimensional Transport Model (MT3D) by S.S. Papadopulos & Associates, Inc. as a site-specific model for assessment of potential future exposures to contaminants in groundwater. MODFLOW and MT3D will be considered to be appropriate site-specific groundwater and contaminant transport models for any situation. Other site-specific groundwater and contaminant transport models may be utilized with the approval of the department. In general, a site-specific groundwater model shall have proven reliability and be able to simulate, as needed:

- A fixed contaminant source,
- Groundwater and contaminant flow in three dimensions,
- Groundwater and contaminant flow through as many distinct geologic layers as necessary for the site in question,
- Effects of pumping,
- Effects of groundwater recharge and discharge,
- Impacts of hydrologic boundaries,
- Contaminant advection, dispersion and chemical reactions, as appropriate for the site in question, and
- Other site-specific variables as appropriate.

Default values for input parameters approved for standard models will be approved for use in site-specific models. Otherwise, input parameters used in site-specific models are subject to the department's approval.

137.9(5) *Response action.* The risk evaluation/response action document shall include a proposal for a response action or strategy to achieve and maintain compliance with the selected standard(s). This may consist of activities designed to remove or treat contaminants, prevention of exposure to unacceptable

levels of contamination through technological/institutional controls or monitoring, or it may consist of a combination thereof. If the response action involves the use of a standard which is less stringent than the statewide standard, it will generally be necessary to implement institutional controls to prevent the type of exposure on which the statewide standard is based. It is the intent of the department to permit the participant to identify and carry out those options by which this may be accomplished, insofar as the department deems the selected options to be reasonable, protective of human health and the environment, and consistent with provisions of the rule.

137.9(6) *Free product and gross contamination.* The response action or strategy for an enrolled site shall take into account a stated policy of the Act to encourage environmental cleanup. To this end, the department requires that contaminants present as free product and gross contamination shall not be addressed through the implementation of institutional or technological controls. For purposes of this rule, gross contamination will be considered to be contamination present at concentrations in excess of a standard by an amount sufficient to reasonably expect that institutional or technological controls will not be adequately protective of human health or the environment.

The department recognizes that treatment or removal of free product or gross contamination may not, in some cases, be feasible. In such cases the department may grant a variance to this portion of the rule. It will be the responsibility of the participant to make a sufficient case that such a variance is warranted.

137.9(7) *Compliance verification strategy.* The risk evaluation/response action document shall outline a strategy for determining whether the relevant standards are met by the site and will continue to be met in the future. In some cases this may consist of sampling and statistical tests to verify that the standard has already been met, while in other cases the sampling and statistics may be used to demonstrate that a response action has achieved its stated goals and the site is now in compliance with standards. Some response strategies may also call for longer term monitoring. In this latter case, standard-based values shall be identified which, if exceeded, would indicate a failure of the response action and necessitate the development and implementation of a new response action. The terms under which monitoring may cease should also be proposed. The proposed strategy shall be consistent with rule 137.10(455H), dealing with demonstration of compliance, and shall indicate the standard to be applied and the point of compliance at which it is to be applied, consistent with rules 137.4(455H), 137.5(455H), and 137.6(455H) (the background, statewide, and site-specific standards, respectively).

137.9(8) *Risk evaluation/response action document submission.* A risk evaluation/response action document shall be submitted for review by the department. When considered in conjunction with the site assessment report, these documents shall present a complete picture of the site from its characterization, through the evaluation of risk, to the development of a strategy to address the situation. An effort shall be made to ensure that the reviewer, or other interested parties, can easily move back and forth through the documents to gain an understanding of the existing situation and proposed actions. The risk evaluation/response action document shall include a summary of findings regarding present risks and potential future risks; a pathway-specific identification of the standards to be applied, including the supporting rationale, if appropriate; a discussion of the proposed response actions, including remedial actions to be taken and institutional or technological controls to be implemented; and a discussion of the proposed verification strategy. Any modeling used for purposes of assessing future risk or establishing site-specific standards shall be presented in sufficient detail to permit evaluation of the results by the department. Any permits which will be necessary to implement the response action shall be identified to the department for inclusion in a consolidated standards permit.

137.9(9) *Department review and approval.* It is strongly recommended that the document be submitted for review and approval prior to proceeding with implementation of the response action. The final, department-approved document will be the basis for assessing subsequent activities at the site. Parties choosing to proceed with response actions without prior review and approval by the department proceed at their own risk and may not assume the response action implemented will result in a no further action certificate.

Parties choosing to implement a response action without prior review and approval by the department shall submit to the department a proposed risk evaluation/response action document accompanied by an

explanation of the reason(s) for proceeding without prior approval. Documentation shall also include a schedule for implementation, a description of construction or other activities to be undertaken, and date for submission of the final report demonstrating compliance, as described in rule 137.10(455H).

The department shall provide opportunity to comment on proposed response actions to any party that is potentially impacted by off-site migration of contaminants for which notification is required in accordance with subrule 137.8(6). The department shall consider reasonable comments from potentially impacted parties in determining whether to approve or disapprove a proposed response action or site closure.

567—137.10(455H) Demonstration of compliance.

137.10(1) *Purpose.* The purpose of the demonstration of compliance section is to provide a mechanism by which to verify that:

a. Appropriate and acceptable standards are complied with and that compliance can be reasonably expected to continue in the future;

b. Any and all remedial measures proposed under rule 137.9(455H) have achieved their purpose; and

c. Appropriate institutional and technological controls, or monitoring mechanisms, have been successfully put in place.

In some cases the demonstration of compliance may mark the final step, taken by the participant, prior to the issuance of a no further action certificate. In other cases it may mark the transition to the longer term closure activities associated with the site, such as monitoring, maintenance of technological controls, and continuing enforcement of institutional controls. In this latter case, demonstration of compliance activities may or may not result in the issuance of a no further action certificate, depending on the approach proposed in the response action. In some cases it may be necessary to successfully complete a monitoring program (or to fulfill other agreed-upon obligations) prior to the issuance of the no further action certificate.

In all cases, sampling of environmental media shall comply with QA/QC requirements addressed elsewhere in this rule.

137.10(2) *General requirements for demonstrating compliance with soil standards.*

a. For the standard being applied, the demonstration of compliance shall be at the point of compliance or point of exposure as set forth in rule 137.4(455H), 137.5(455H), or 137.6(455H) relating to background standards, statewide standards, and site-specific standards, and described in a site-specific context pursuant to subrule 137.9(7), relating to risk evaluation/response action.

b. Minimum sample numbers for the demonstration of compliance with the background standard for soils (paragraph 137.10(4)*"b"*) or with the statewide standard when applying subparagraph 137.10(5)*"a"*(1) shall be based on the volume of soil to which the selected standard is being applied as follows:

(1) For volumes less than or equal to 125 cubic yards, a minimum of 8 samples.

(2) For volumes greater than 125 cubic yards, but less than or equal to 3,000 cubic yards, a minimum of 12 samples.

(3) For each additional volume of less than or equal to 3,000 cubic yards, a minimum of 12 additional samples.

(4) Additional samples may be required based on site-specific conditions.

c. When applying the 95 percent upper confidence limit, according to EPA guidance, to demonstrate compliance with the statewide standard for soils (subparagraph 137.10(5)*"a"*(2)) or a site-specific standard for soils (subrule 137.10(6)), the minimum sample number shall be as specified in that guidance.

d. Sample locations for demonstration of compliance shall be selected in a systematic random fashion to be representative, both horizontally and vertically, of the volume of soil being evaluated for compliance.

e. Sampling for the purposes of demonstrating compliance shall be conducted after the completion of site assessment activities and after the implementation of applicable remedial measures.

137.10(3) *General requirements for demonstrating compliance with groundwater standards.*

a. For the standard being applied, the demonstration of compliance shall be at the point of compliance or point of exposure as set forth in rule 137.4(455H), 137.5(455H), or 137.6(455H), relating to background standards, statewide standards, and site-specific standards, and described in a site-specific context pursuant to subrule 137.9(7), relating to risk evaluation/response action.

b. Monitoring wells installed for the purpose of demonstrating compliance shall be of sufficient number and appropriate location to evaluate all hydrologic strata of concern, based on site-specific considerations, as identified pursuant to subrule 137.9(7), relating to risk evaluation/response action.

c. For statistical methods under subparagraph 137.10(5)*"b"*(1), compliance with the statewide groundwater standard shall be based on eight consecutive quarters of groundwater data.

As an alternative, the department may accept four consecutive quarterly sampling events or less with written approval from the department under the following conditions:

(1) There is adequate spatial monitoring of the plume upgradient which indicates a decreasing concentration trend toward the downgradient property boundary.

(2) Parameters affecting the fate and transport of regulated substances within the plume have been fully evaluated.

(3) Concentrations of regulated substances in the plume at the point of compliance monitoring wells along the downgradient property boundary are all less than or equal to the groundwater standard or the limit relating to the PQL, whichever is higher, in all samples collected during the quarters of monitoring.

(4) One of the following is met:

1. The age of the plume is sufficiently well known to permit a judgment to be made regarding its stability.

2. The remediation includes source removal or containment actions which would reduce chemical flux into the plume.

d. When applying the 95 percent upper confidence limit, according to EPA guidance, to demonstrate compliance with the statewide standard for groundwater (subparagraph 137.10(5)*"b"*(2)) or a site-specific standard for groundwater (subrule 137.10(6)), the minimum sample number shall be as specified in that guidance.

e. Sampling for the purposes of demonstrating compliance shall be conducted after the completion of site assessment activities and after the implementation of applicable remedial measures.

137.10(4) *Demonstration of compliance with a background standard.*

a. To apply a background standard the participant shall demonstrate to the department, in writing, that the apparent background contamination at the site is due to widespread or naturally occurring contamination and shall obtain the department's approval to use this subrule. Data collected for the purpose of determining the applicable background standard is subject to department approval, interpretation, and manipulation, if necessary for the purpose of establishing a meaningful background standard.

b. For soil, the minimum sample number to determine the background standard shall be 10 (unless a lesser number is approved by the department) and the number of samples from the affected area shall be based on volume as described in 137.10(2)*"b."* No sample collected from the affected area may exceed the sum of the background arithmetic mean and three times the sample standard deviation, as calculated based on the background sampling.

c. For groundwater, a minimum of 12 locations shall be sampled in the background reference area (unless a lesser number is approved by the department) and an equal number shall be collected from the affected area. In areas involving more than one hydrologic strata, more samples may be required. Sampling shall be conducted concurrently in the background reference area and the affected area. No sample collected from the affected area may exceed the sum of the background arithmetic mean and three times the sample standard deviation, as calculated based on the background sampling.

137.10(5) *Demonstration of compliance with the statewide standard.* The following requirements shall be met in order to demonstrate compliance with the statewide standard. Testing shall be performed individually for each contaminant being addressed and for which a no further action certificate is sought.

a. To demonstrate compliance with the statewide standard for soils in each affected area, in addition to (1) or (2) as follows, all other applicable requirements of this rule shall be met.

(1) Seventy-five percent of all soil samples, collected during a single event, shall be less than or equal to the statewide standard, with no individual sample exceeding 10 times the statewide standard.

(2) In accordance with EPA-approved methods, the 95 percent upper confidence limit of the arithmetic mean of soil sample values from the affected area shall be at or below the statewide standard.

b. To demonstrate compliance with the statewide standard for groundwater in each compliance monitoring well, in addition to (1) or (2) as follows, all other applicable requirements of this rule shall be met.

(1) Seventy-five percent of all samples collected in each compliance monitoring well over time shall be less than or equal to the statewide standard, with no individual sample exceeding 10 times the statewide standard.

(2) In accordance with EPA-approved methods, the 95 percent upper confidence limit of the arithmetic mean of samples collected from a compliance well over time shall be at or below the statewide standard.

137.10(6) *Demonstration of compliance with a site-specific standard.* To demonstrate compliance with a site-specific standard, the participant shall use the tests identified in 137.10(5)*"a"*(2) and 137.10(5)*"b"*(2), except that the 95 percent upper confidence limit of the arithmetic mean for samples from the medium of concern shall be at or below the site-specific standard.

137.10(7) *Compliance with cumulative risk.* In addition to or, for soil only, in lieu of compliance with standards for individual contaminants as prescribed above, cumulative risk criteria must be attained. Cumulative carcinogenic health risks shall not exceed 1 in 10,000. Noncarcinogenic health risks affecting the same target organ shall not exceed a cumulative hazard quotient of 1. Cumulative risk criteria are applicable to multiple contaminants in the same medium and multiple media in which exposure is likely to occur to the same individual. Cumulative risks shall be based on the same exposure assumptions that are used for determining the selected standard.

Risks associated with background levels of contaminants shall not be included in the cumulative risk determination. Background levels of contaminants shall be determined in accordance with subrule 137.10(4) or, if approved by the department, by the use of generally available information on background levels of contaminants.

In situations where the risk associated with exposure to a contaminant at a concentration equal to the selected standard is greater than the acceptable cumulative risk, the cumulative risk may be calculated assuming the risk associated with exposure to the contaminant at a concentration equal to the selected standard is equal to the acceptable cumulative risk criterion. The department will provide a guidance document for calculating cumulative risk and make it readily available to the public.

137.10(8) *Final report.* A final report shall be submitted which documents the accomplishment of all provisions set forth in the risk evaluation/response action document. This shall include, as applicable to the specific situation, discussions related to verification of compliance with selected standards; successful completion of approved remedial actions; implementation of necessary institutional or technological controls; and initiation of any required monitoring strategy. Sufficient details shall be included to permit the department to verify that the terms proposed in the response action have been met with regard to the statistical determination of compliance with standards.

137.10(9) *Department review and approval.* The final report is subject to review and approval by the department. Following review, the department will either approve the report or make a written response indicating the reason(s) why the report is unacceptable. Acceptance of the report may result in the issuance of a no further action certificate or it may mark a transition to the long-term closure activities associated with the site, as proposed in the response action. A decision that the report is unacceptable may be based upon an insufficiency of the report or it may be based on a judgment that the terms of the response action have not been met.

In cases where a participant has elected to proceed through this program without department interaction and without submitting site assessment (pursuant to rule 137.8(455H)) or risk evaluation/response action documents (pursuant to rule 137.9(455H)), the final report shall contain

the substantive information related to those rules in addition to information required under this rule. The intent is to create a document for departmental review and approval which clearly sets forth, in substance, the same process which would have been developed had the participant engaged in a stepwise approach including interaction with the department during the process.

567—137.11(455H) No further action classification.

137.11(1) *Eligibility.* An enrolled site shall be eligible to obtain a no further action classification, when the department determines the participant has met all compliance standards of this chapter applicable to the affected area and the hazardous substances actually identified and evaluated such that no further response action is required other than maintenance of institutional or technological controls or certain specified continuing site activities. Upon request of a participant or a protected party and compliance with applicable standards, the department will issue a no further action letter to each protected party requesting it.

A no further action classification may be conditioned upon the continued maintenance and effectiveness of any applicable institutional or technological control in accordance with rule 137.7(455H).

137.11(2) *No further action certificate.* A no further action letter shall be in a form recordable in the county real estate records as provided in Iowa Code chapter 558 and consistent with the model forms developed by the department. The no further action letter may be recorded as provided by law.

137.11(3) *No further action certificates conditioned on institutional and technological controls.* A no further action certificate conditioned upon the continuing effectiveness and maintenance of institutional and technological controls or other continuing requirements must be recorded with the consent of the fee titleholder for each parcel of affected property subject to the controls and for parcels of property for which prevention of exposure is dependent upon the continuing effectiveness and maintenance of the controls. If a participant is not able to record the no further action letter on a parcel within the affected area due to objections of the fee titleholder or other legal restraints, this alone shall not be a basis for denying or rescinding the no further action classification or the certificate or the legal protections attendant to the no further action classification. Any modification or termination of institutional and technological controls shall be noted in an amended no further action certificate and shall be recorded as to any property subject to an earlier recorded certificate or institutional control. If a no further action certificate is required to be recorded, the no further action classification is not effective until the document is recorded with the county recorder.

137.11(4) *Scope of liability protection.* Upon issuance of the no further action letter by the department, the liability protection provisions contained in Iowa Code Supplement chapter 455H, subchapter 3, apply. The scope of the no further action classification and the scope of liability protection extend only to that area of affected property as defined by actual and modeled contaminant data and the specific environmental condition for which a regulatory standard has been met and approved by a no further action classification. The scope of protection corresponds to the scope of the site assessment conducted by the participant, the exposure pathways actually evaluated by the assessment report and reviewed by the department, and the hazardous substances identified in that assessment for which compliance with a department-approved standard has been achieved. Liability protection does not apply to releases, sources of contamination, hazardous substances or other environmental conditions not expressly addressed in the participant's site assessment, response action or specifically referenced in the no further action certificate.

The no further action classification and certificate shall be void if the department demonstrates by clear, satisfactory, and convincing evidence that any approval under this chapter was obtained by fraud or material misrepresentation, knowing failure to disclose material information, or false certification to the department.

137.11(5) *Reopener and reclassification conditions.*

a. The department shall have grounds to reopen and rescind a no further action classification and consider reclassification of the affected area if specified conditions of the no further action classification and certificate are not maintained, or if institutional or technological controls fail to meet their intended

purpose or are determined to be ineffective and unenforceable. If the conditions upon which the no further action classification was issued cannot be corrected or reinstated, the department may rescind the classification. The effect of termination is to put all parties in the same position as if the no further action letter had not been issued.

b. If a no further action certificate is issued without conditions or technological and institutional controls and conditions should arise which might require further corrective action, the department may require further response action by a participant or protected party only as provided in Iowa Code Supplement section 455H.301. The department may require further response action against a statutorily responsible party who is not a participant or a protected party. If the participant was a person having control over a hazardous substance, as defined in Iowa Code section 455B.381, at the time of the release, a no further action certificate may provide or the department may require further response action to protect against an imminent and substantial threat to public health, safety, and welfare. A protected party who was a person having control over a hazardous substance, as defined above, may be required by the department to conduct a further response action, where appropriate, to protect against an imminent and substantial threat to public health, safety, and welfare.

These rules are intended to implement Iowa Code Supplement chapter 455H.

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APPENDIX D

IDNR LAND RECYCLING PROGRAM &
SUMMARY OF SIMPLIFIED RISK-BASED FORMULA
FOR DETERMINING GROUNDWATER AND SOIL
RESPONSE ACTION STANDARDS

Summary of Simplified Risk-Based Formulae for Determining Groundwater and Soil Response Action Standards

Standard	Cancer-Based	Non-Cancer Based
Statewide-Groundwater Protected Source	$\frac{1.75 \times 10^{-4}}{SF_o}$	$7 \times RfD_o$
Statewide-Groundwater Non-protected Source	$\frac{3.5 \times 10^{-3}}{SF_o}$	$35 \times RfD_o$
Statewide-Soil	$\frac{3.194}{(3.16 \times SF_d \times Abs) + SF_o}$	$\frac{78{,}214 \times RfD_o \times RfD_d}{(2.8 \times RfD_o \times Abs) + RfD_d}$
Site-Specific-Soil Deep Residential	$\frac{31.94}{(3.16 \times SF_d \times Abs) + SF_o}$	$\frac{782{,}140 \times RfD_o \times RfD_d}{(2.8 \times RfD_o \times Abs) + RfD_d}$
Site-Specific-Soil Nonresidential <2 ft. deep	$\frac{318}{(6.6 \times SF_d \times Abs) + SF_o}$	$\frac{387{,}121 \times RfD_o \times RfD_d}{(2 \times RfD_o \times Abs) + RfD_d}$
Site-Specific-Soil Nonresidential >2 ft. deep	$\frac{2710}{(3 \times SF_d \times Abs) + SF_o}$	$\frac{387{,}121 \times RfD_o \times RfD_d}{(3 \times RfD_o \times Abs) + RfD_d}$

NOTES: Cancer Slope Factor (SF); Reference Dose (RfD); Subscripts: "o" - oral, "d" - dermal

1. A standard for a protected groundwater source will be the drinking-water maximum contaminant level (MCL) if one exists, if not the lifetime health advisory level (HAL) will be the standard, and if neither a MCL nor HAL exist the standard will be risk-based as described herein.

2. Standards for Compounds classified as cancer group A or B will be cancer based.

3. Standards for Compounds classified as cancer group D or E will be non-cancer based.

4. Standards for Compounds classified as cancer group C will be determined as follows:

   a. Statewide Standard for Protected Groundwater Source: $0.7 \times RfD_o$

   b. Statewide Standard for Non-Protected Groundwater Source:
      If a SF_o exists, use the larger of: $(3.5 \times 10^{-3}) \div SF_o$ or $3.5 \times RfD_o$
      If no SF_o exists use: $35 \times RfD_o$

   c. All Soil Standards:
      If a SF exists, use the larger of: the cancer-based standard or
      0.1 times the non-cancer-based standard.
      In no SF exists use the non-cancer-based standard.

5. For the time being dermal Absorption factors (Abs) can be obtained from Chapter 3, Exhibit 3-4, page 16 at the following Internet address (other chemicals assumed Abs = 0): http://www.epa.gov/superfund/programs/risk/ragse/

6. Compliance with standards for individual chemicals does not guarantee compliance with cumulative risk standards, especially for site-specific soil standards.

APPENDIX E

LABORATORY QUALITY ASSURANCE / QUALITY CONTROL MANUALS

Quality Assurance Manual
Cover Page

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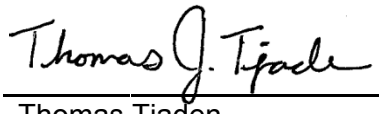
Quality Assurance Manual
Approval Signatures

Michael K. McGee, MS, CIH
Laboratory Director

6/17/2018
Date



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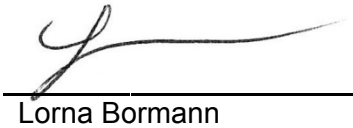

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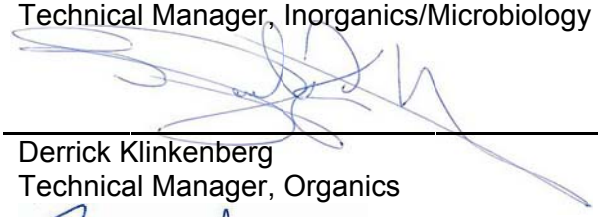

Derrick Klinkenberg
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7/9/2018
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6/1/2018
Date

SECTION 2. TABLE OF CONTENTS

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LIST OF APPENDICES

REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-C-S-001	Work Sharing Process
CA-I-P-002	Electronic Reporting and Signature Policy
CA-I-S-009	TALS Security and User Access
CA-L-P-002	Contract Compliance Policy
CA-Q-M-002	Corporate Quality Management Plan
CA-Q-P-003	Calibration Curves and Selection of Calibration Points
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-006	Detection Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001	Corporate Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization
CW-L-P-001	Records Retention Policy
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation
CW-L-S-004	Subcontracting
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-Q-S-005	Data Recall Process

REFERENCED LABORATORY SOPs

SOP Reference	Title
CF-FSS-01	Sampling Procedures
CF-FSS-02	Field Analysis Procedures
CF-GP-01	Analytical Balance Operation
CF-GP-04	Data Review for Metals
CF-GP-05	Data Review for Wet Chemistry Parameters
CF-GP-06	Data Review for Organic Parameters
CF-GP-13	Pipette Verification Procedures
CF-GP-20	Permit and Action Limit Exceedance Procedures
CF-GP-21	Project and Contract Review Procedures for Meeting Regulatory, Accreditation, and Client Requirements

SOP Reference	Title
CF-GP-22	Investigating a Proficiency Test Failure
CF-GP-23	Laboratory Personnel Training
CF-GP-24	Sub-Sampling and Sample Homogenization
CF-GP-26	Fisher Consignment Stockroom and Supply Handling
CF-IH-06	Calibration of Sampling Pumps
CF-QA-04	Quality Control Limits
CF-QA-06	Laboratory Document Control and Archiving
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CF-QA-09	Random Marginal Exceedances
CF-SRV-01	Customer Service and Login Procedures
CF-SS-02	Standards Preparation, Documentation, and Tracking
CF-SS-03	Reagents Preparation, Documentation, and Tracking
CF-WD-01	Waste Disposal

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica Cedar Falls' 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. The 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 NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP; Doc. No. CA-Q-M-002) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

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

- ANSI/ASQC, E4-1994, "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version).
- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002), US EPA, May 31, 2006).
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* US EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)*, Third Edition, US EPA, September 1986; and as amended by 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; Final Update IIIA, April 1998; Final Update IIIB, November 2004, Final Update IV, February 2007; Final Update V, July 2014; and Update VI (various dates).
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- EPA 815-R-05-004, *Manual for the Certification of Laboratories Analyzing Drinking Water*, EPA, January 2005).
- APHA, *Standard Methods for the Examination of Water and Wastewater*, 18th, 19th, 20th, 21st, 22nd, and on-line Editions.
- 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 the laboratory 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.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, influent/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 processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests 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 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 on the company's website (www.testamericainc.com). The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory 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, the Technical Manager(s) and the Quality Assurance (QA) Manager. 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 template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed every two years by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. 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. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to the Document Control & Updating procedures (refer to SOP No. CF-QA-06).

This manual has been assigned the unique document number CF-QA-01. The overall effective date and revision number of this manual is indicated on the Cover Page and left-hand header of subsequent pages. Each section of this manual also has an effective date and revision number, indicated in the right-hand header of each page. When the manual is published, each section shares the same overall effective date and revision number (i.e., if the manual has an overall revision number of 6, the initial revision number of each section is 6.0). If interim changes to

parts of the manual occur, the effective date of each applicable section will be updated, and the section revision number will increase incrementally (i.e., the section revision will become 6.1).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Cedar Falls is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President and Chief Executive Officer (CEO), Chief Operating Officer (COO), Executive Vice President (VP) Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Cedar Falls is presented in Figure 4-1.

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 briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for 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. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of the TestAmerica Cedar Falls laboratory.

4.2.2 President and Chief Executive Officer (CEO)

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

4.2.3 Chief Operation Officer (COO)

The COO reports directly to the President and CEO of TestAmerica. The COO oversees the operations of all TestAmerica laboratories and the EMLab P&K business unit. The VP's of Operations report directly to COO.

4.2.4 Vice President of Operations

Each Vice President (VP) of Operations reports directly to the Executive VP of Operations and is a part of the Executive Committee. Each VP of Operations is responsible for the overall administrative and operational management of their respective laboratories. The VP's responsibilities include allocation of personnel and resources, long-term planning, goal setting, and achieving the financial, business, and quality objectives of TestAmerica. The VP's ensure timely compliance with Corporate Management directives, policies, and management systems

reviews. The VP's are 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 Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to the President and CEO. With the aid of the Executive Committee, Laboratory Directors, Quality Directors, Safety Manager, EH&S Coordinators and QA Managers, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and EH&S Programs within TestAmerica. Additional responsibilities include:

- Review of QA/QC and EHS aspects of Corporate SOPs & Policies, national projects and expansions or changes in services.
- Work 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 laboratories and a summary of any quality related initiatives and issues.
- Preparation of a monthly report that includes EH&S metrics across the analytical laboratories and a summary of any EH&S 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.

4.2.6 Vice President of Client Service

The VP of Client Services leads the Client Service Organization (CSO) and is responsible for client satisfaction, driving operational excellence and improving client responsiveness. The VP provides direction to the Client Service Directors, Programs Managers and Project Managers.

4.2.7 Quality Assessment Director

The Quality Assessment Director reports to the VP-QA/EHS. The Quality Assessment Director has QA oversight of laboratories; responsible for the internal audit system, schedule and procedure; monitors laboratory internal audit findings; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Compliance Director, the Quality Systems Director, and the VP-QA/EHS, the Quality Assessment Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.8 Quality Compliance Director

The Quality Compliance Director reports to the VP-QA/EHS. The Quality Compliance Director has QA oversight of laboratories; monitors and communicates DoD / DoE requirements; develops corporate tools for ensuring and improving compliance; develops corporate assessment tools; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Systems Director and the VP-

QA/EHS, the Quality Compliance Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.9 Quality Systems Director

The Quality Systems Director reports to the VP-QA/EHS. The Quality Systems Director has QA oversight of laboratories; develops quality policies, procedures and management tools; monitors and communicates regulatory and certification requirements; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Compliance Director and the VP-QA/EHS, the Quality Systems Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.10 Quality Information Manager

The Quality Information Manager is responsible for managing all company official documents (e.g., Policies, Procedures, Work Instructions), the company's accreditation database, intranet websites, external laboratory subcontracting, regulatory limits for clients on the company's TotalAccess website; internal and external client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions. The Quality Information Manager reports to the VP-QA/EHS; and works alongside the Quality Assessment, Quality Compliance and Quality System Directors and EHS Managers to support both the Analytical Quality Assurance and EHS Programs within TestAmerica.

4.2.11 Technical Services Director

The Technical Services Director is responsible for establishing, implementing and communicating TestAmerica's Analytical Business'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.

4.2.12 Ethics and Compliance Officers (ECOs)

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – Corporate Counsel & VP of Human Resources and the VP-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 President and CEO, VPOs, Laboratory Director 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.

4.2.13 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.

4.2.14 Environmental Health and Safety Managers (Corporate)

The EHS Managers report directly to the VP-QA/EHS. The EHS Managers 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.

4.2.15 Laboratory Director

The Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective VPO. 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 managers for the appropriate fields of testing. If the Technical Manager 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 Manager 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.
- 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. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Technical Manager(s), and the Operations Manager(s) as direct reports.

4.2.16 Quality Assurance (QA) Manager or Designee

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 their Corporate Quality Director. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA staff to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- 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.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- 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 action database and the corrective and preventive action systems.
- 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 shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- 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.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluation of the thoroughness and effectiveness of training.

4.2.17 Technical Manager or Designee

The Technical Manager (also known as Operations Manager) reports directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision**.** The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i.e., SOPs, 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. He/she insures that the SOPs are

properly managed and adhered to at the bench. He/she develops 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 their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved 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.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- 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 Manager and QA Manager and in compliance with regulatory requirements.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.

4.2.18 Supervisors

Supervisors report to the Operations Manager. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform 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), 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 HR Departments. They evaluate 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 Manager and/or QA Manager.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Manager, and/or Laboratory Director.
- 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.
- 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.

4.2.19 Laboratory Analysts

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 worklists, benchsheets, lab notebooks and/or in LIMS.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, 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 supervisor, the Technical Manager, 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.

4.2.20 Safety Officer

The duties of the Safety Officer may be combined with another position in the laboratory. The Safety Officer 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 Safety Data Sheet (SDS) 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.
- Measure and record ventilation hood velocities according to the laboratory's schedule and procedures. Follow-up and/or schedule corrective action if fume hoods do not meet laboratory criteria.
- 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.

4.2.21 Hazardous Waste Coordinator

The duties of the Hazardous Waste Coordinator may be combined with another position in the laboratory. The Hazardous Waste Coordinator ensures that waste disposal systems and processes are maintained to ensure compliance with all local, state, and federal hazardous waste regulations. Specific responsibilities include, but are not limited to:

- Staying current in knowledge of hazardous waste regulations.
- Maintaining continued training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual (EHSM).
- Auditing 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.
- Managing waste generated by the facility and organizing waste streams for pickup by a licensed hazardous waste management contractor.

4.2.22 Project Manager (PM)

The PM reports to the Manager of Project Management (MPM) and serves as the interface between the laboratory's technical departments and the laboratory's clients. There is an entire staff of Project Managers and Project Management Assistants that make up the Project Management team. With the overall goal of total client satisfaction, the functions of this team 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 COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors 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.
- Monitor the status 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.
- Coordinate requests for sample containers and other services (data packages).

4.3 Deputies

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

Table 4-1. Deputies for Key Personnel

Key Personnel	Deputy
Mike McGee Laboratory Director	Keith Fitzpatrick Corporate Business Controller
Thomas Tjaden Quality Manager	Mike McGee Laboratory Director
Derrick Klinkenberg Organic Technical Manager	Mike McGee Laboratory Director
Lorna Bormann Inorganics Technical Manager	Mike McGee Laboratory Director
Chad Timmins EHS Coordinator	Mike McGee Laboratory Director
Drew Miller Hazardous Waste Coordinator	Mike McGee Laboratory Director
Brian Graettinger Manager of Project Management	Shirley Thompson Project Manager

Figure 4-1. Corporate and Laboratory Organization Charts

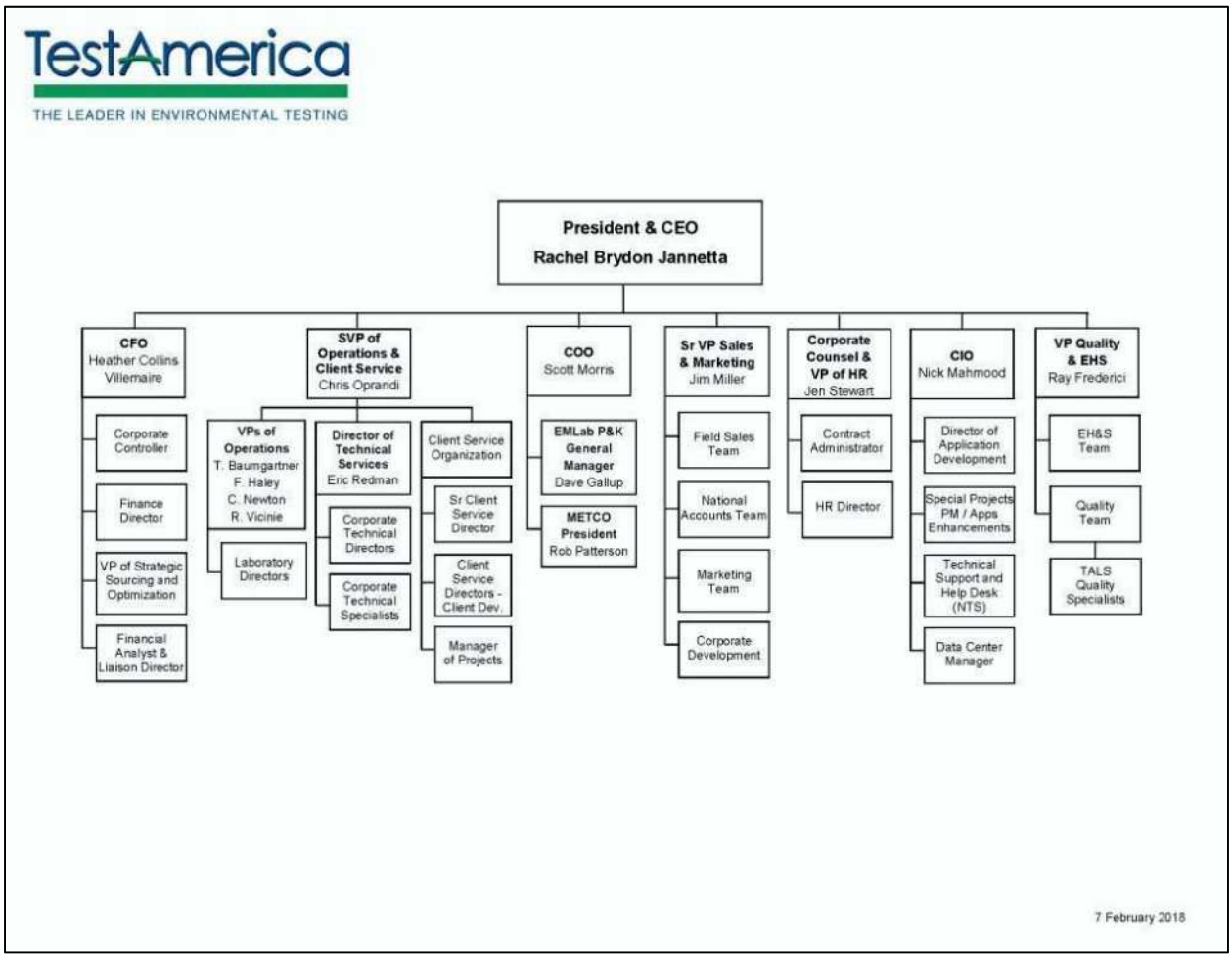
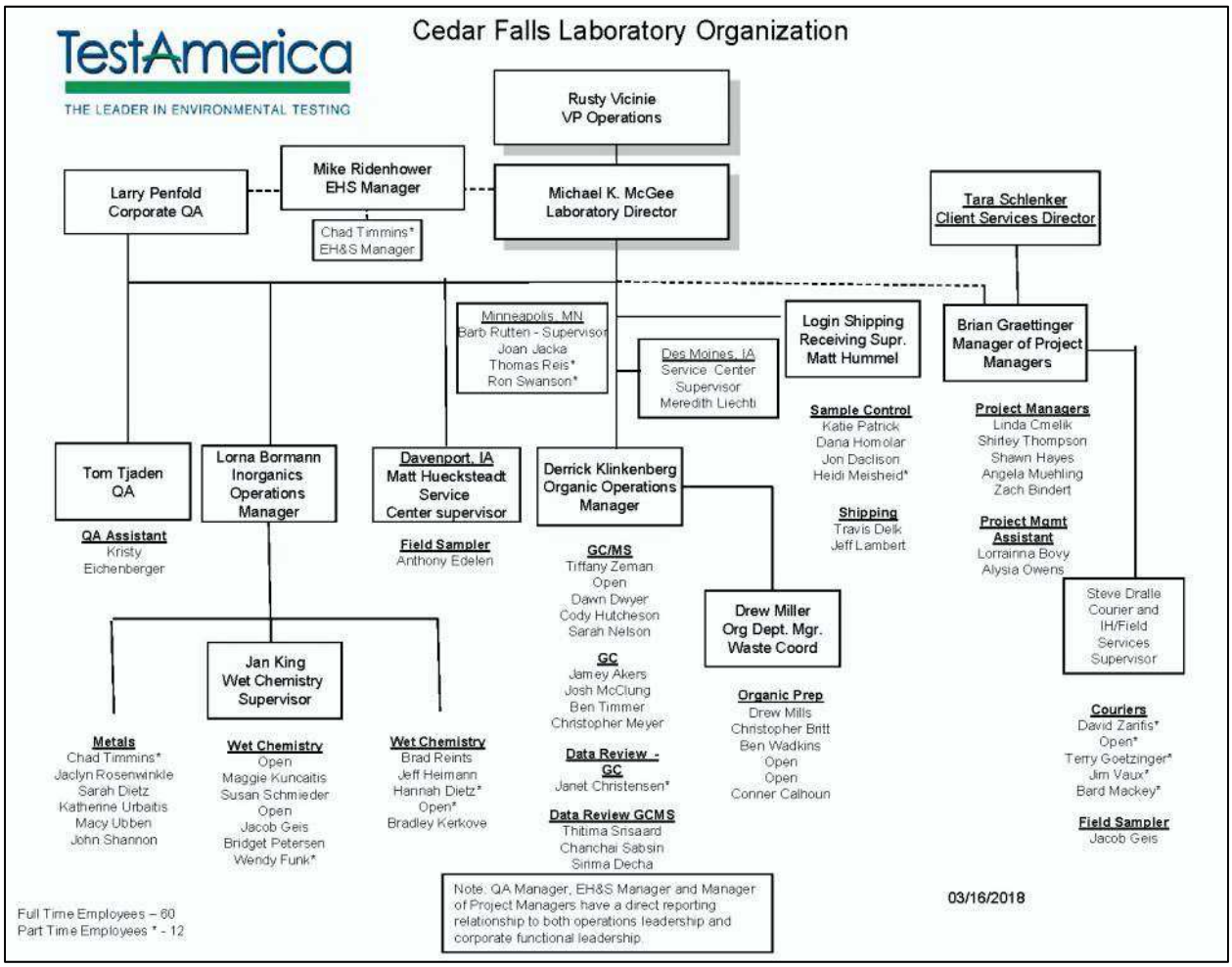


Figure 4-1. Corporate and Laboratory Organization Charts (continued)



SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

It is TestAmerica's Policy to:

- ❖ Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- ❖ Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- ❖ Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- ❖ Provide clients with the highest level of professionalism and the best service practices in the industry.
- ❖ To comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard and applicable accreditation programs, and to continually improve the effectiveness of the management system.

Every staff member at the laboratory 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 elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- 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. (Corporate SOP No. CW-L-S-002).
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-Q-S-005).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined 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 to 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 Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – 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.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

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

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

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 duplicate or matrix spike (MS) duplicate samples.

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.

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.

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 Selectivity

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..

5.4.7 Sensitivity

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 maintains quality control limits within method limit group (MLG) tables in LIMS that summarize the precision and accuracy acceptability limits for performed analyses. These MLG tables include an effective date, are updated each time new limits are generated and are managed by the laboratory's QA department. 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, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in the laboratory's control limits SOP (CF-QA-04).

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846 methods) and accreditation programs. The laboratory 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 Manager 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 addition, database audit trails in LIMS allow for retrieval of historical control limits. 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 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

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 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 QC Charts

As control limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these when control limits are updated to determine if adjustments need to be made or for corrective actions to methods. Periodically, lab management may also review QC charts to troublshoot non-conforming QC, such as failed PT results, client-requested investigations, etc. All findings 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 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6. DOCUMENT CONTROL

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:

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

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the 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 Corporate documents is found in Corporate SOP No. CW-Q-S-001, *Corporate Document Control and Archiving*. The laboratory's internal document control procedure is defined in SOP No. CF-QA-06.

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 actions. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a technical manager or supervisor 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 that document as the official document on file. That document is then provided to all applicable operational units (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 two years—excepting Safe Drinking Water Act documents which are reviewed annually—and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP No. CF-QA-06. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder for the applicable revision.

For changes to SOPs, also refer to SOP No. CF-QA-06. The SOP identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized by department in the QA office. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP No. CF-QA-06.

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 according to SOP No. CF-QA-06.

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

The laboratory 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 the laboratory'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 the laboratory'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 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 laboratory'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 the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the 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 same contract review process used for the initial review 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) or Project Management Assistant (PMA) is considered adequate. The PM or PMA 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. Laboratory SOP No. CF-GP-21, *Project and Contract Review Procedures for Meeting Regulatory, Accreditation, and Client Requirements*, provides additional details on this procedure. 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 Client Relationship Manager (CRM) or Proposal Team, 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 TestAmerica's Corporate 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):

- Contract Administrator
- VP of Operations
- Laboratory Project Manager
- Laboratory Directors and/or Corporate Technical Managers
- Laboratory Directors and/or Corporate Information Technology Managers
- Account Executives
- 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 Sales Director, Contract Administrator, Account Executive or Client Relations Manager 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.

The Contracts Department maintains copies of all signed contracts.

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. This information is archived on the TANet Oasis intranet. Comments and notes are also maintained in the LIMS for client and individual bids and contracts.

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

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. Each PM and PMA keeps a phone log of conversations with the client. A Quote Log is maintained and distributed to Sales, Project Managers, and the Laboratory Director on a weekly basis. Communications between Sales and Marketing should be captured in Salesforce and pertinent information should be copied to all appropriate Project Management and technical staff. Information regarding specific projects or clients is entered into the laboratory LIMS for reference by all laboratory staff. Documents and other client information are organized in a folder on the Bid server at the TestAmerica Cedar Falls laboratory. This folder and information is accessible to project managers and other staff, as warranted. National client contract information is available on the TANet Oasis intranet.

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, a PM and/or PMA is assigned to each client. It is the PM and PMA'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 primary 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 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 may introduce new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings, as needed. 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 acknowledged by both parties.

Changes may also be communicated to the laboratory during production meetings, as needed. 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 Technical 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).

The laboratory 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.

7.4 Special Services

The laboratory 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. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 Client Communication

PMs and PMAs are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

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

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

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.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica 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 the need arises to 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 TestAmerica's Corporate SOP's on Subcontracting Procedures (CW-L-S-004) and the Work Sharing Process (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 TNI/ISO 17025, state- or program-specific requirements, 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-TNI accredited work where required.

Project Managers (PM), Client Relationship Managers (CRM) or Account Executives (AE) for the Export Lab (TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing and when possible approval from the client shall be retained in the project folder. Standard TestAmerica Terms & Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., certain USACE projects) require notification prior to placing such work.

8.2 Qualifying and Monitoring Subcontractors

Whenever a PM, CRM, or AE 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:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.
- Subcontractors reviewed by TestAmerica – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI, or A2LA); technical specifications; legal and financial information.

A listing of vendors is available on the TestAmerica intranet site.

All TestAmerica laboratories are pre-qualified for work sharing 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 Corporate SOP No. CA-C-S-001, Work Sharing Process).

8.2.1 When the potential sub-contract laboratory has not been previously approved, AEs or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM) or Laboratory Director. The CRM or Laboratory Director requests that QA staff or the PM begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CW-L-S-004, Subcontracting Procedures.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. After the Corporate QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified for the finance/procurement system.

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. TestAmerica does not certify laboratories. The subcontractors on our approved list can only be recommended to the extent that we would use them.

8.3 Oversight and Reporting

8.3.1 The status and performance of qualified subcontractors will be monitored by the Corporate Quality department. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance, Legal and Corporate Quality personnel.

- 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 are posted using the Vendor Performance Report.
- Information shall 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. CSO personnel will notify all TestAmerica laboratories, Corporate Quality 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 CSO Personnel, Laboratory Directors, QA Managers and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it is current and scope-inclusive. For workshare laboratories (i.e., TestAmerica laboratories) certifications can be viewed on the company's Total Access Accreditation and Certification Management (TAACM) database.

8.3.2 For continued use of a subcontractor, verification of certification is placed upon the subcontractor for the defined project. Samples are subcontracted under Chain of Custody with the program defined as 'Accreditation Required' and the following statement for verification upon sample receipt:

Note: Since laboratory accreditations are subject to change, TestAmerica Laboratories, Inc. places the ownership of method, analyte & accreditation compliance upon our subcontract laboratories. This sample shipment is forwarded under Chain of Custody. If the laboratory does not currently maintain accreditation in the State of Origin listed above for analytes/tests/matrix being analyzed, the samples must be shipped back to the TestAmerica laboratory or other instructions will be provided. Any changes to accreditation status should be brought to TestAmerica Laboratories, Inc. attention immediately. If all requested accreditations are current to date, return the signed Chain of Custody attesting to said compliance to TestAmerica Laboratories, Inc.

8.3.3 All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for all samples workshared within TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI 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.

Note: The results submitted by a TestAmerica worksharing laboratory may be transferred electronically and the results reported by the TestAmerica worksharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and COC.

In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time.

The use of any emergency subcontractor will require the PM to complete a New Vendor Add Form in order to process payment to the vendor and add them to TALS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

SECTION 9. PURCHASING SERVICES AND SUPPLIES

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, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Company-Wide Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). 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.

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

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on its Solvent & Acid Lot Testing & Approval Program, SOP No. CA-Q-S-001. Approval information for the solvents and acids tested under this program is stored on the TestAmerica Sharepoint site, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis or reports for the materials, are stored in the same location. The laboratory also has a consignment system through Fisher Scientific, which contains many common laboratory reagents and supplies which have been approved for use. Laboratory SOP CF-GP-26 describes the consignment process.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be 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 may check the

item out of the on-site consignment system that contains items approved for laboratory use; or, if the item is not in consignment, the analyst completes the Material Request Sheet when requesting reagents, standards, or supplies.

The analyst must provide the master item number (from the master item list that has been approved by the Technical Manager), item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the Technical Manager prior to placing the order. The purchasing coordinator places the order.

9.3.2 Receiving

It is the responsibility of the receiving department to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials were 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. This is documented through the addition of the received date and initials to the information present on the daily order log.

The person receiving the shipment verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared “public” folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available 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

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, analytical reagent grade will 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 expiration 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 and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer’s or SOP’s expiration date unless ‘verified’ (refer to item 3 listed below):

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent 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/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained in the QA department.

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. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. 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 specific conductivity of less than 1 µmhos/cm (or specific resistivity of greater than 1 megaohm-cm) at 25°C. The specific conductivity is monitored daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers 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) water 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 bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section, and electronic copies are made and filed with the applicable standard or reagent in LIMS. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager or QA Manager.

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. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 Purchase of Equipment / Instruments / Software

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 Technical Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which 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, an identification name is assigned and added to the equipment list. The IT Department must also be notified so that they can synchronize the instrument for back-ups. The equipment item's 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 (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual becomes a controlled document and is retained by the laboratory.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as-needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the applicable Technical Manager and/or Laboratory Director.

Analytical balances are serviced and calibrated annually in accordance with SOP CF-GP-01. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed, and documentation of the review is filed with the certificates. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as reference thermometers, weight sets, etc, are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory.

9.6 Suppliers

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount 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 finance/procurement 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.

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

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

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a New Vendor Add Request Form.

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 Technical Services Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10. COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and 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 (e.g., 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 addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

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 12 (Corrective Actions) and is initiated by submitting complaints to the QA department in writing. The QA Manager summarizes complaints in the QA Monthly Report to management, along with a summary of any investigations performed and corrective actions taken.

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to procedures listed above.

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

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

10.3 Internal Complaints

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 non-conformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the laboratory and Quality 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 Systems Review (Section 16).

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, 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. 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. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, departures from documented policies and procedures are sometimes needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with other technical staff or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

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 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Manager and 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 TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.

11.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may 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. This information may also be documented in logbooks and/or data review checklists as appropriate. 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 Management within 24 hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data

Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., the VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

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, QA Manager, ECOs, VP of Operations and the Quality Directors 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.

11.3 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.

Corporate SOP entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

Corporate SOP entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-Q-S-005.

11.4 Prevention of Non-Conforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, the QA Department evaluates non-conformances to determine if any non-conforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

11.5 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 the fifth paragraph of Section 11.2.

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

The Laboratory Director 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 12 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 VP of Operations 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 to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., 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, Technical Manager, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must 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.

SECTION 12. CORRECTIVE ACTION

12.1 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 non-conforming 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 Memo (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 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(s) for investigating.
- 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.

12.2.1 Non-Conformance Memo (NCM) – an application within the LIMS, an NCM can be linked to specific methods, samples, and/or jobs and can be compiled in the report narrative. An NCM may be used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits
- Isolated reporting / calculation errors
- Client complaints.

12.2.2 Corrective Action Report (CAR) – within a comprehensive database known as ICAT, a CAR is used to document larger and more complex or systematic issues. A CAR is used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCMs
- Issues found while reviewing NCMs that warrant further investigation
- Internal and external audit findings
- Failed or unacceptable PT results (the lab also has an SOP (CF-GP-22) specific to investigating PT failures)
- Corrective actions that cross multiple departments in the laboratory

- Systematic reporting / calculation errors
- Client complaints regarding data quality
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports or hold time violations
- Health and Safety violations

This will provide background documentation to enable root cause analysis and preventive action.

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- 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 Technical Manager, Laboratory Director, or QA Manager (or designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- 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 NCM or CAR is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with

three or more incidents to triangulate a weakness. Corporate SOP Root Cause Analysis (No. CA-Q-S-009) describes the procedure.

Systematically analyze and document the root causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and CAR is entered into a database for tracking purposes and a monthly summary of all corrective actions can be printed out for review to aid in ensuring that the corrective actions have taken effect.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (ICAT) database developed by the company in 2015. (Previously, a local database served this purpose.) An incident is an event triggering the need for one or more corrective actions as distinct from a corrective action, a potential deficiency stemming from an incident that requires investigation and possibly fixing. The database is independent of TALS, available to all local and corporate managers, and capable of notifying and tracking multiple corrective actions per event, dates, and personnel. ICAT allows associated document upload, categorization (such as, external/internal audit, client service concerns, data quality issues, proficiency testing, etc.), and trend analysis. Refer to Figure 12-1.
- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). 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 out-of-control situation and problems encountered in solving the situation.

12.3.5 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.
- 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.

(Also refer to Section 15.1.4, Special Audits.)

12.4 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 11). The documentation of these procedures is through the use of an NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 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, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

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 by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

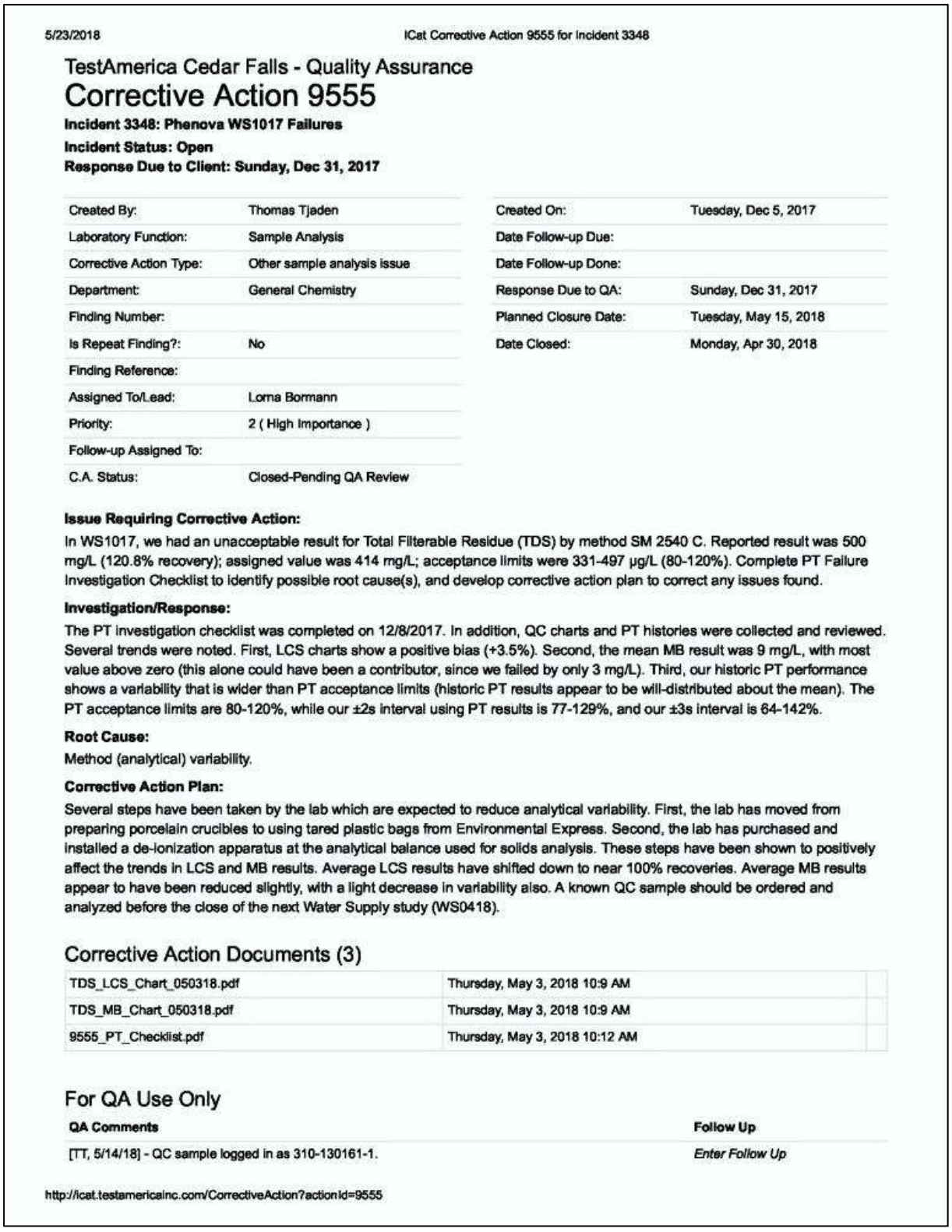
12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not 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.

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.

Figure 12-1. Example: Corrective Action Report



5/23/2018 ICat Corrective Action 9555 for Incident 3348

TestAmerica Cedar Falls - Quality Assurance

Corrective Action 9555

Incident 3348: Phenova WS1017 Failures

Incident Status: Open

Response Due to Client: Sunday, Dec 31, 2017

Created By:	Thomas Tjaden	Created On:	Tuesday, Dec 5, 2017
Laboratory Function:	Sample Analysis	Date Follow-up Due:	
Corrective Action Type:	Other sample analysis issue	Date Follow-up Done:	
Department:	General Chemistry	Response Due to QA:	Sunday, Dec 31, 2017
Finding Number:		Planned Closure Date:	Tuesday, May 15, 2018
Is Repeat Finding?:	No	Date Closed:	Monday, Apr 30, 2018
Finding Reference:			
Assigned To/Lead:	Lorna Bormann		
Priority:	2 (High Importance)		
Follow-up Assigned To:			
C.A. Status:	Closed-Pending QA Review		

Issue Requiring Corrective Action:

In WS1017, we had an unacceptable result for Total Filterable Residue (TDS) by method SM 2540 C. Reported result was 500 mg/L (120.8% recovery); assigned value was 414 mg/L; acceptance limits were 331-497 µg/L (80-120%). Complete PT Failure Investigation Checklist to identify possible root cause(s), and develop corrective action plan to correct any issues found.

Investigation/Response:

The PT investigation checklist was completed on 12/8/2017. In addition, QC charts and PT histories were collected and reviewed. Several trends were noted. First, LCS charts show a positive bias (+3.5%). Second, the mean MB result was 9 mg/L, with most value above zero (this alone could have been a contributor, since we failed by only 3 mg/L). Third, our historic PT performance shows a variability that is wider than PT acceptance limits (historic PT results appear to be will-distributed about the mean). The PT acceptance limits are 80-120%, while our ±2s interval using PT results is 77-129%, and our ±3s interval is 64-142%.

Root Cause:

Method (analytical) variability.

Corrective Action Plan:

Several steps have been taken by the lab which are expected to reduce analytical variability. First, the lab has moved from preparing porcelain crucibles to using tared plastic bags from Environmental Express. Second, the lab has purchased and installed a de-ionization apparatus at the analytical balance used for solids analysis. These steps have been shown to positively affect the trends in LCS and MB results. Average LCS results have shifted down to near 100% recoveries. Average MB results appear to have been reduced slightly, with a light decrease in variability also. A known QC sample should be ordered and analyzed before the close of the next Water Supply study (WS0418).

Corrective Action Documents (3)

TDS_LCS_Chart_050318.pdf	Thursday, May 3, 2018 10:9 AM
TDS_MB_Chart_050318.pdf	Thursday, May 3, 2018 10:9 AM
9555_PT_Checklist.pdf	Thursday, May 3, 2018 10:12 AM

For QA Use Only

QA Comments	Follow Up
[TT, 5/14/18] - QC sample logged in as 310-130161-1.	*Enter Follow Up*

http://icat.testamericainc.com/CorrectiveAction?actionId=9555

Table 12-1. Example: General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Analyst)*	- Instrument response < RL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Technical Manager(s))*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Initial Calibration Verification (Second Source) *(Analyst, Technical Manager(s))*	- % Recovery within LIMS 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 LIMS control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within LIMS control limits.	- 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. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.

Table 12-1. Example: General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within LIMS control limits.	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
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. - Surrogate results outside criteria shall be reported with qualifiers.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples *(QA Manager, Technical Manager(s))*	- 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, Technical Manager(s), Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.

Table 12-1. Example: General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, Corporate QA, Corporate Management)	- SOP CW-Q-S-005, Data Recall	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002 or your lab's CA SOP.
Client Complaints *(Project Managers, Lab Director/Manager, Sales and Marketing)*	- Determined on a case by case basis	- 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., project needs to be updated).
QA Monthly Report (Refer to Section 16 for an example) *(QA Manager, Lab Director/Manager, Technical Manager(s))*	- 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, Technical Manager(s))*	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Notes:
1. Except as noted below for certain compounds, the method blank should 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 reporting 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 reporting limit, the method blank must be below the reporting limit.

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 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 and continuous process of improvement activities 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 the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and 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. The metrics report is reviewed monthly be the laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used ~~to~~ in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lesson Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.

The laboratory's corrective action process 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 and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action/process improvement system:

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

13.1.2 Any Preventive Actions/Process Improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: facility changes, major accreditation changes, addition or deletion to division's capabilities or instrumentation, key personnel changes, or laboratory information management system (LIMS) changes.

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management 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 (5) years after it has been issued. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2.

14.1 Overview

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 14-1. More detailed information on retention of specific records is provided in CW-L-P-001, *Records Retention Policy* and CW-L-WI-001, *TestAmerica Records Retention/Storage Schedule*. Quality records are maintained by the QA department on the Corporate QA server, which is backed up as part of the regular company systems. 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).

Table 14-1. Record Index

	Record Types[1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals - Published Methods	Indefinitely
QA Records	- Certifications - Method and Software Validation / Verification Data	Indefinitely
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - Data Investigation	5 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)

Table 14-1. Record Index

	Record Types[1]:	Retention Time:
Project Records	- Sample Receipt & COC Documents - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Financial and Business Operations	Refer to CW-L-WI-001
	EH&S Manual, Permits	Indefinitely
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies	Indefinitely
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to CW-L-WI-001
	IT Records	Refer to CW-L-WI-001
	Corporate Governance Records	Refer to CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Verification, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

14.1.1 All records are 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. 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 and shall be documented with an access log**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility for the purpose of retrieving data. Records are maintained for a minimum of five years unless otherwise 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 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 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.

Table 14-2. Example: Special Record Retention Requirements

Program	Retention Requirement[1]
Drinking Water – All States	10 years (lab reports and raw data) 10 years - Radiochemistry (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
Minnesota Department of Commerce records associated with Petrofund program	7 years
Ohio VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement
OSHA	30 years

[1] Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 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, refer to Section 19.14.1 for more information.

14.1.4 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 two (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 COC is stored electronically with the job in LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are

provided with a work order, they are also stored with the job in LIMS.

- 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***).*** Instrument data is stored sequentially by instrument. 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 log 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 (alternatively, this data may be captured entirely by the electronic record in LIMS). Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. 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.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned.
- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'**.**

14.2 Technical and Analytical Records

14.2.1 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 (5) years unless otherwise specified by a client or regulatory requirement. 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 reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. 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:

- Laboratory sample ID code;

- Date of analysis; time of analysis is also 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 LIMS, a specific logbook, or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- 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.

14.2.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a documented supervisory or peer review on a monthly basis.

14.3 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.

14.3.1 Sample Handling Records

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.

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage and Disposal

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 upon request.

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.

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.

The laboratory has a record management system (a.k.a., document control) 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. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the LIMS—no logbooks are used to record that data. Records are considered archived when noted as such in the records management system (a.k.a., document control).

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory 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 five (5) years of such action.

14.5.2 Records Disposal

Records are removed from the archive and destroyed after five (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. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits (QA Technical Audits)	Joint responsibility: a) QA Manager or designee b) Technical Manager or designee (Refer to CW-Q-S-003)	QA Technical Audits Frequency: • 50% of methods annually
SOP Method Compliance	Joint responsibility: a) QA Manager or designee b) Technical Manager or designee (Refer to CW-Q-S-003)	SOP Compliance Review Frequency: • SDWA SOPs annually • All other SOPs every 2 years
Special	QA Department or designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful PTs per year for each TNI field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, other program quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be

performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period. All analysts should be reviewed over the course of a two year period through at least one QA Technical Audit.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years (annually for methods and administrative SOPs related to drinking water programs). It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities (e.g., new IDOC), it is recommended that the work products of the analyst are reviewed within 3 months of completing the documented training.

15.1.4 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.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of proficiency testing (PT) samples provided by a third party. The laboratory generally participates in the following types of PT studies: drinking water, non-potable water, hazardous waste (solid/soil), sewage/sludge, and state-specific studies for underground storage tank (UST) programs. The laboratory also participates in quarterly performance testing for industrial hygiene programs.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, 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.

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.

15.2 External Audits

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. Laboratory supervisors 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 the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory 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.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, 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 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the corrective action process and database (Section 12). The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the manager or supervisor where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

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.

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.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Managers, their Quality Director as well as the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, VP of Operations or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare 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 Senior Management Team and VPs of Operations.

16.2 Annual Management Review

The senior laboratory management team (Laboratory Director, Technical Managers, 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. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. 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 cannot be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 and Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review 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 Laboratory Director and 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 lab management 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).
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate VP of Operations 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)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 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. TestAmerica's Corporate Internal Investigations SOP shall be followed (SOP No. CW-L-S-002). 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.

TestAmerica's President and CEO, Executive VP of Operations, VP of Client & Technical Services, VPs of Operations and Quality Directors receive a monthly report from the VP-QA/EHS summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific labs.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory'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 Figure 4-1 (Section 4).

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.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staff members that possess 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. Some accreditation programs (e.g., AIHA-LAP) have specific education requirements that must be satisfied for management staff and analysts. 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 located on the TestAmerica intranet site's Human Resources web-page (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.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	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
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	College degree in an applied science; Or, 2 years of college chemistry	Or, 5 years of prior analytical experience
Spectra Interpretation	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 Managers - **General**	Bachelor's 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 Managers - **Wet Chem** only (no advanced instrumentation)	Associate's degree in an applied science or engineering; Or, 2 years of college with 16 semester hours in chemistry	And, 2 years relevant experience.
Technical Managers - **Microbiology**	Bachelor's degree in applied science with at least 16 semester hours in general microbiology and biology. An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of relevant 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 Technical Manager, 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.

17.3 Training

The laboratory 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	Prior to lab work	All
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 Refresher	Annually	All
Initial Demonstration of Capability (IDOC)	Prior to unsupervised method performance	Technical
Continuing Demonstration of Capability (CDOC)	Annually	Technical

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 19.

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

- 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 19).
- 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 violations). This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in the Laboratory Training SOP (CF-GP-23).

17.4 Data Integrity and Ethics Training Program

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 followed by technical data integrity training within 30 days, 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 a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement 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.
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- 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.

SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 12,000 ft^2 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 personnel protective equipment (PPE) including safety glasses, protective clothing, gloves, face shields, 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, shipping, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, microbiological sample analysis, and administrative functions.

18.2 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 humidity and temperature levels in the laboratory.

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.

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

18.3 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.
- Closed areas for GC/MS analytical work.
- Designated areas for waste disposal and chemical storage.

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 the following documents and procedures for specific requirements for microbiological laboratory facility requirements.

- Standard Methods, 9020 B-2005, Sec. 3
- TNI Standard, Volume 1, Module 5, Section 1.7.3.7

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

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

Employees wear assigned lab coats that have their names sewn on the front while on the premises.

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 the laboratory. In addition to signing into a visitor's logbook, 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: "Notice - Authorized Personnel Only."

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory 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.

19.2 Standard Operating Procedures (SOPS)

The laboratory 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.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled *Writing a Standard Operating Procedure*, Doc. No. CW-Q-S-002.
- SOPs are reviewed at a minimum of every 2 years (annually for drinking water SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

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 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.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

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), 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.

19.4.1 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.

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.

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:

- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Analysis and Sampling Procedures.* 40 CFR Part 136 as amended by the most recent Method Update Rule.
- Code of Federal Regulations (CFR), Title 40, Parts 136, 141, 143, 172, 173, 178, 179, 257, 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water*. EPA/815/R-05/004, January 2005.
- *Methods for Chemical Analysis of Water and Wastes.* EPA/600/4-79/020, revised March 1983 where applicable.
- *Methods for the Determination of Inorganic Substances in Environmental Samples*. EPA/600/R-93/100, August 1993.
- *Methods for the Determination of Metals in Environmental Samples*. EPA/600/4-91/010, June 1991. Supplement I: EPA/600/R-94/111, May 1994.
- *Technical Notes on Drinking Water Methods*. EPA/600/R-94/173, October 1994.
- *NIOSH Manual of Analytical Methods (NMAM)*, 4th and 5th Editions.
- *Standard Methods for the Examination of Water and Wastewater*. 20th/21st/22nd & on-line editions (www.standardmethods.org); American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)*, Third Edition, US EPA, September 1986; and as amended by 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;

Final Update IIIA, April 1998; Final Update IIIB, November 2004, Final Update IV, February 2007; Final Update V, July 2014; Update VI (various dates); and New Test Methods On-line (www.epa/gov/SW-846).

- *Annual Book of ASTM Standards*. ASTM International, West Conshohocken, PA.
- IDEXX Laboratories, Inc. (www.idexx.com).
- "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments" (Book 5, Chapter A1), *Techniques of Water-Resources Investigations of the United States Geological Survey*, U.S. Department of the Interior, Denver, CO, Revised 1989 unless otherwise stated.

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.

19.4.2 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.

A demonstration of capability (DOC, Lab SOP No. CF-GP-23) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst hasn't performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL 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 or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- 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.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

The procedure for completing an IDOC is summarized below. For a full description, refer also to the laboratory SOP CF-GP-23, *Personnel Training*.

19.4.3.1 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

19.4.3.2 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.3 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.4 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.5 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.6 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.2 above.
- Beginning with 19.4.3.2 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If

this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 19-1 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.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP 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.

19.6 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. 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 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.

19.6.1 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.

19.6.1.1 Determination of Method Selectivity – 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.

19.6.1.2 Determination of Method Sensitivity – 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.

19.6.1.3 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 concentration of analyte that can be quantitatively determined with acceptable precision and bias. 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.

19.6.1.4 Determination of Interferences – A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range – Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 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.

19.6.1.7 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.

19.6.1.8 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.

19.7 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 can be differentiated from method blanks. 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. Generally, the analyst prepares at least seven method blanks and 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. The replicates used in the MDL study must be prepared in at least three different batches on three separate calendar dates and analyzed on three separate calendar dates.

Refer to the Corporate SOP No. CA-Q-S-006 for details on the laboratory's MDL process.

19.8 Instrument Detection Limits (IDL)

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.

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 times the absolute value of the standard deviation.

If IDL is > than the MDL, it may be used as the reported MDL.

19.9 Verification of Detection and Reporting Limits

Once an MDL is established, it must be verified quarterly, on each instrument, by analyzing at least two quality control samples (prepared as a sample) at the same spiking concentration used for the initial MDL study. The analytes must be qualitatively identified. 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.

At least once every 13 months, the MDL is recalculated using the data collected by the quarterly verifications and on-going method blank data from routine analytical batches. Refer to Corporate SOP CA-Q-S-006 for details on verification and re-evaluation of MDL values.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit and annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, 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. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

19.11 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.12 Estimation of Uncertainty of Measurement

19.12.1 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.

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

19.12.3 The minimum 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 and the variability due to matrix effects). 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.

19.12.4 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 uncertainties at approximately the 99% confidence level with a coverage factor of k=3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L.

19.12.5 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.), the method specifies the form of presentation of calculated results, and the laboratory follows the method exactly as written, no further discussion of uncertainty is required.

19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from the 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 Contract Terms & Conditions for reanalysis protocols may supersede the following items):

- 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 ≤ 5 times 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 & 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.
- Due to the potential for increased variability, reanalysis may not be applicable to non-homogenous, Encore, and sodium bisulfate preserved samples. Consult the Technical Manager, QA Manager, or Laboratory Director if unsure.

19.14 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.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in Corporate IT SOP CA-I-S-009, *TALS Security and User Access*, and Corporate IT white paper CA-I-W-001, *TALS LIMS Audit Trails and Data Security*. The laboratory is currently running TALS which is a custom in-house developed 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 MS SQL Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 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. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.14.1.2 Ensure Information Availability – Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 Maintain Confidentiality – Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.14.2 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, 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 reviewed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s), and uploaded to the documents section of the LIMS batch.

Manual integration of chromatographic peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices*.

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.

19.14.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. 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/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.14.2.2 In general, concentration results are reported in milligrams per liter (mg/L) or micrograms per liter (µg/L) for aqueous samples, and milligrams per kilogram (mg/Kg) or micrograms per kilogram (µg/Kg) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1%. Units are defined in each lab SOP.

19.14.2.3 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 two or three significant figures on the final report, depending client specifications and the formatter used.

19.14.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.14.2.5 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. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a backup server.

19.14.3 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 12.
- 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 Technical Manager/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are outlined in several laboratory SOPs (e.g., CF-SRV-01 for Sample Control, CF-GP-04 for Metals Data Review, CF-GP-05 for Wet Chemistry Data Review, CF-GP-06 for Organics Data Review, and CF-GP-21 for Project Management) to ensure that reported data are free from calculation and transcription errors, and that QC parameters have been reviewed and evaluated before data is reported. The laboratory also abides by Corporate QA SOP CA-Q-S-002 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.

19.14.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.14.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS and data qualifiers are added as needed. All first level reviews are documented.

19.14.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- 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.

19.14.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.14.4.5 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.14.4.6 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 the COC is followed, cover letters / narratives are present, flags are appropriate, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

19.14.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.14.4.8 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

19.14.5 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 TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline for our internal practices.

19.14.5.1 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.

19.14.5.2 Analysts shall not increase or decrease peak areas 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 principles and policy and is grounds for immediate termination.

19.14.5.3 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.

19.14.5.4 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.

Figure 19-1. Example: Demonstration of Capability Documentation (Page 1)

Analyst Demonstration of Capability

TestAmerica Cedar Falls

[redacted]

10/23/2014

Preparation Method(s):	Distill/Ammonia
Analytical Method(s):	350.1
Matrix:	Water
Method Description:	Nitrogen, Ammonia
Preparation SOP No:	CF-WCA-13
Analytical SOP No:	CF-WCA-13

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability (DOC).
2. The test method(s) was performed by the analyst identified on this certificate.
3. A copy of test method(s) and laboratory SOPs are available for all personnel on-site. **These documents have been reviewed by the analyst as part of this DOC.**
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

[redacted]

Analyst	**Signature**	**Date**
Technical Manager	**Signature**	**Date**
Quality Assurance Manager	**Signature**	**Date**

Page 1 of 2

Figure 19-1. Example: Demonstration of Capability Documentation (Page 2)

Analyst Demonstration of Capability

ANALYST DEMONSTRATION OF CAPABILITY

Method 350.1
Method Desc: Nitrogen, Ammonia
Analyst:

Laboratory: TestAmerica Cedar Falls
Limit Group: WC 350.1 - All - iQC_QC

Current Limits		
Recovery	Precision	RPD

Demonstration of Capability		
Recovery	Precision	RPD

Ammonia

Analysis Dates: 7/23/2014 to 10/23/2014

All values within Control limits

LCL	UCL	Std Dev	RPD	Units
90	110	15	20	%

Mean	Std Dev	Units		Amount	Amount/RL
102.2	2.563293	%	Pass	1.002312	8

Laboratory ID	Anal Date	Batch	Smp	Analyst	Prep Analyst	Result	Units	Spike Amount	RL	% Rec	% D	In Rec Limits?
LCS 310-57494/2-A	08/18/2014	57934	24			4.082	mg/L	4.002312	0.500	102		Pass
LCS 310-57517/2-A	08/18/2014	57934	45			4.197	mg/L	4.002312	0.500	105		Pass
LCS 310-57627/2-A	08/18/2014	57934	55			3.955	mg/L	4.002312	0.500	99		Pass
LCS 310-57641/2-A	08/18/2014	57934	89			4.133	mg/L	4.002312	0.500	103		Pass

Precision = standard deviation of percent recoveries of spiked control samples.

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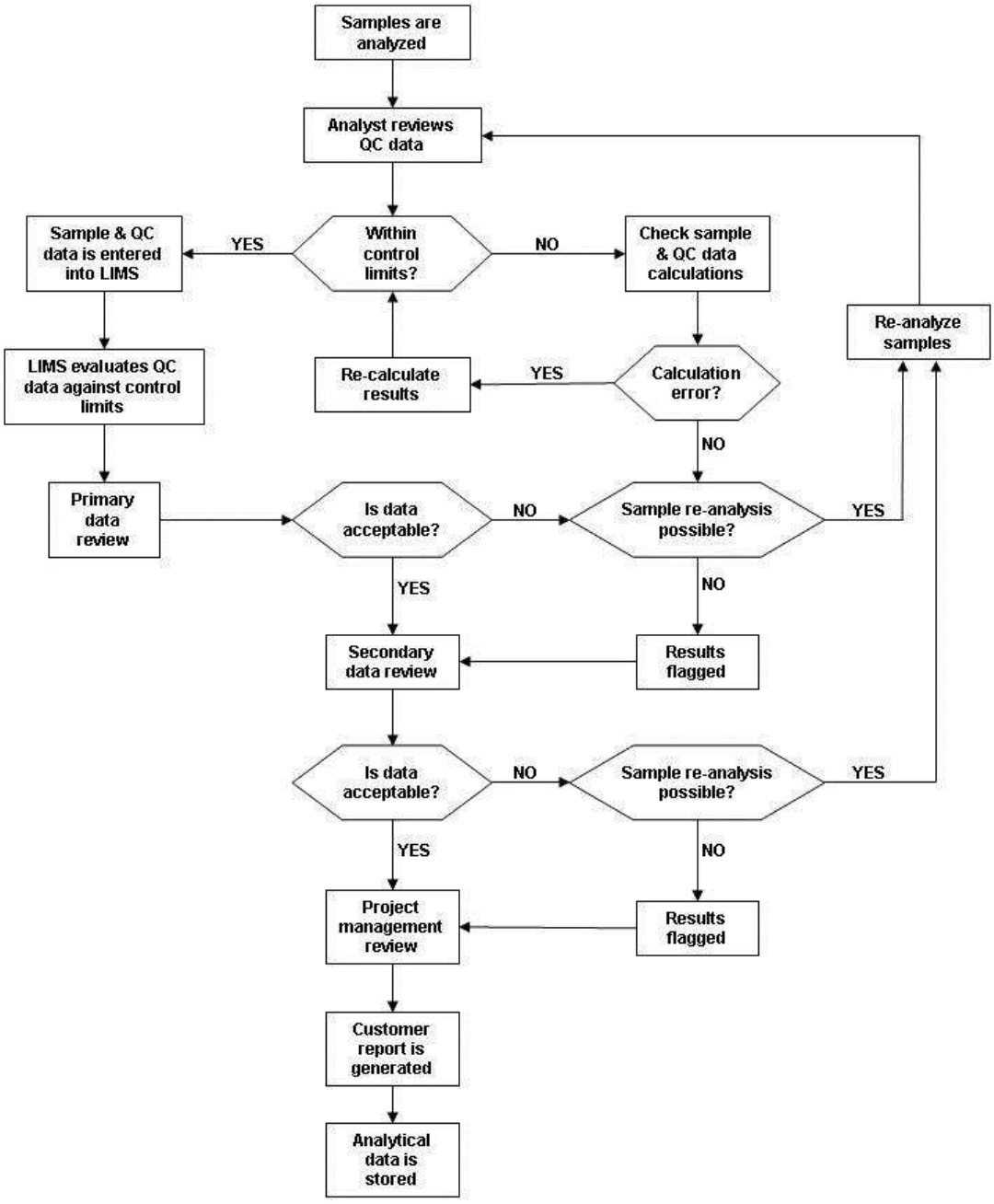
Figure 19-2. Example: Work Flow



SECTION 20. EQUIPMENT AND CALIBRATIONS

20.1 Overview

The laboratory 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 SOPs. A summary of laboratory instrumentation is presented in Table 20-1. The QA Department maintains the master list of all instrumentation used in the laboratory.

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

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance 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.

Routine preventive maintenance procedures and frequency, such as 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.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

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.

- 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.
- 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.) must also be documented in the instrument records.

- 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 signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

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.

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.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations.

20.3 Support Equipment

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, thermal/pressure sample preparation 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.

20.3.1 Weights and Balances

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 initial serviceable use with at least four 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 in the QA Department. Additional information on the verification and operation of balances is in laboratory SOP CF-GP-01, *Analytical Balance Operation*.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 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 µmhos/cm.

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

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

20.3.3 Thermometers

All liquid-in-glass thermometers (such as those containing mercury or alcohol) are calibrated on an annual basis with a NIST-traceable thermometer. Dial-type thermometers, digital thermometers, thermocouples and other similar electronic temperature measuring devices are calibrated quarterly.

Infrared (IR) thermometers are calibrated on a semi-annual basis. IR thermometers are calibrated over the full range of use, including ambient (20-30°C), iced (4°C), and frozen (0°C to -5°C), per the Drinking Water Manual. Each day of use a single verification of the IR thermometer is made by checking the temperature of a bottle of water at the temperature of interest that contains a calibrated thermometer.

Mercury NIST thermometers are recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. Digital (electronic) NIST thermometers are recalibrated annually. The NIST thermometers have increments of at least 0.5 degrees Celsius, and have ranges applicable to method and certification requirements. The NIST traceable thermometers are used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks (hard-copy or electronic). Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in applicable preparation/analytical batches in LIMS or method-specific logbooks.

More information on the procedures used to calibrate and/or verify thermometers is found in laboratory SOP CF-QA-08, *In-House Calibration and/or Verification of Laboratory Support Equipment*.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

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. Freezer temperatures are kept ≤ -10°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.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis. Details on the procedures used to verify pipette performance is located in laboratory SOP CF-GP-13, *Pipette Verification Procedures*.

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 cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company or an equivalent manufacturer. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” (or however named) from the manufacturer attesting established precision and accuracy.

20.3.6 Autoclaves

Each batch processed in the autoclave is marked with autoclave tape. The autoclave tape must turn dark to verify sterilization temperatures were achieved. An autoclave thermometer is also used to verify autoclave temperatures with every batch. The duration and temperatures reached during each autoclave batch are recorded in the autoclave logbook.

Sterilization is also verified monthly with “temptubes.” A temptube is a small culture tube that contains a solid pellet. The temptube is processed through a normal sterilization cycle. The pellet in the tube must be completely melted as verification that temperatures sufficient for sterilization were maintained for the proper duration.

The autoclave’s internal timing device is checked quarterly against a stopwatch to verify the actual time elapsed. The results of this test are documented in the Bacteria QA Logbook.

A spore check is performed on the autoclave on a monthly basis according to the following procedure:

- A sealed ampoule containing *Geobacillus stearothermophilus* and Bromocresol Purple (pH indicator) is put through a normal autoclave sterilization cycle. After autoclaving, the autoclaved test ampoule is incubated for 48 hours at 55-60°C along with a control ampoule which has NOT been autoclaved. The control ampoule should show a positive test by exhibiting a color change to or toward yellow and/or turbidity. If the control ampoule does not show a positive result the test should be considered invalid.
- A successful sterilization cycle would result in the test ampule having no color change or turbidity (remains purple) after incubation. A failed sterilization is indicated by a color change to or toward yellow and/or turbidity in the test ampule.
- The test results are documented in the Bacteria QA Logbook.

20.3.7 Field Sampling Devices (ISCO Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number for traceability purposes. This number is also recorded on the sampling documentation.

20.3.8 Soil Gas Sampling Pumps

The laboratory provides sampling pumps for soil gas collection by clients. Each sampling pump is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The sampling pump calibration process is described in laboratory SOP CF-IH-06, *Calibration of Sampling Pumps*.

20.4 Instrument Calibrations

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 detection/quantitation limits, the working range of the analytical instrumentation, and any fluctuations that may occur from day to day. In general, calibration generation and review will be in compliance with laboratory analytical SOPs and Corporate QA Policy No. CA-Q-P-003, *Calibration Curves & Selection of Calibration Points*.

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, and type of calibration (e.g., average calibration/response factors, linear or non-linear regression, 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 12).

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

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

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). 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 at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exceptions to these rules are ICP methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

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). Such verification is sometimes called Initial Calibration Verification (ICV) (or Independent Calibration Verification). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. 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. Initial calibration verification is with a standard source secondary to the calibration standards (second-source standard), but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here 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 used by the laboratory.

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, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

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 then bracketing calibration verification 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).

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, QC, or standard that can be injected within 12-hours of the beginning of the shift.

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 method SOPs. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with unacceptable calibration verification may be useable under the following special conditions:

a) When the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b) When the acceptance criteria for the CCV 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 CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit

standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

Samples reported by the conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for 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 (these calculations are available in the laboratory method SOPs). 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.

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.

20.5 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 should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should 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.

20.6 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 spectrometer, 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.

Table 20-1. Example: Instrumentation List

GC	GC/MS	ICP	ICPMS	GFAA	Hg	FIA	IC	TOC	UV/Vis	BOD
9	9	2	1	1	1	2	2	1	2	1

Table 20-2. Example: Schedule of Routine Maintenance

Preventative Maintenance Procedures For Laboratory Equipment

Instrument/ Equipment Type	Maintenance	Frequency
Gas Chromatograph	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	Daily*
	Replace Injection port liners/re-silonize liners	GC, As needed; GC/MS, Daily*
	Replace injection port liner o-ring	GC, As needed; GC/MS, Daily*
	Replace inlet seal and ring	GC, As needed, GC/MS, Daily*
	Replace column ferrules	GC, As needed, GC/MS, Daily*
	Clip column ends, injector (daily) and detector (as needed)	GC, As needed; GC/MS, Daily*
	Replace syringes on autosamplers	As needed*
	Replace heated-zones heaters and sensors	As needed*
	Replace inlet assembly	As needed*
	Fill solvent rinse and empty solvent rinse-waste vials (on autosampler tower)	Daily or as needed
	Replace column	As needed*
Flame Ionization Detector (FID)	Clean/replace jet	As needed*
	Clean collector	As needed*
	Check and/or adjust gas flows	As needed*
	Replace graphite ferrule	After each cleaning (OI detectors only)
Electron Capture Detector (ECD)	Perform wipe test	Every six months*
	Remove and send to authorized agency for cleaning	As needed*
	Check and/or adjust gas flows	As needed*
	Replace gas supply cylinders	As needed*
Photoionization Detector (PID)	Clean window	As needed*
	Replace o-ring seat	As needed*
	Replace Lamp	As needed*
	Check and/or adjust gas flows	As needed*
	Adjust Lamp power supply intensity	As needed*
Mass Spectrometer (MS)	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*
	Change rough pump oil	After each source cleaning
	Refill calibration compound (PFTBA) vial	As needed

* Date and Maintenance performed are recorded in the Maintenance Log of the instrument/equipment

Preventative Maintenance Procedures For Laboratory Equipment

Instrument/ Equipment Type	Maintenance	Frequency
Purge and Trap Equipment	Refill rinse water supply/Empty rinse water waste	Weekly or as needed
	Refill spiking solutions vials	As needed
	Rinse sparge tubes	After each sample
	Clean or replace 6-port valve	As needed*
	Replace Transfer lines (from Autosampler to LSC and from LSC to GC)	As needed*
	Adjust gas flows and pressures	As needed
	Perform leak check	As needed
Graphite Furnace, Atomic Absorption (GFAA)	Change Graphite contact rings	As needed*
	Clean quartz windows	As needed
	Change graphite tubes and platforms	As needed*
	Refill rinse water	Daily
	Check water cooler water level and filter	Monthly
	Change argon and other gases	As needed*
	Clean or replace sampling probe	As needed
Total Organic Carbon (TOC)	Clean UV Reactor Chamber	Weekly*
	Clean Sparger Tube	Weekly*
	Clean Mist Trap	Weekly*
	Refill rinse/reagent water	As needed
	Clean Gas/Liquid Separator	Weekly*
	Change Liquid in Gas/Liquid Separator	As needed
	Replace Components of Chlorine Scrubber	Weekly*
	Change compressed gas tanks	As needed*
	Replace NDIR Detector	As needed*
	Replace UV Reactor	As needed*
	Replace Tubing	As needed
Inductively Coupled Plasma, Atomic Emission Spectrometer (ICP-AES)	Replace Peristaltic pump tubing	Daily
	Clean autosampler, change tubing	As needed*
	Clean nebulizer and torch assembly	As needed*
	Replace nitrogen and argon tanks	As needed*
	Refill rinse water receptacle	Daily
	Empty waste receptacle	Daily
	Check for internal standard and sample flow through peristaltic pump tubing	As often as possible
	Replace internal standard solution receptacle	As needed
	Operate and check vents	Daily
	Perform Hg alignment	Daily*
	Check water level and water filter on recirculating-cooling unit, refill and replace filter	Check daily, refill and replace as needed
	Check purge windows	Daily, replace as needed
	Replace nebulizer and o-rings	As needed*
	Replace torch	As needed*
	Replace mixing chambers	As needed*
	Clean or replace air filters	Monthly
	Check pneumatic filters	Weekly, replace as needed
	Perform wave calibration (UV and Vis)	Quarterly*
	Calibrate Detector	Quarterly*

Preventative Maintenance Procedures For Laboratory Equipment

Instrument/ Equipment Type	Maintenance	Frequency
Inductively Coupled Plasma, Mass Spectrometer (ICP-MS)	Replace Peristaltic pump tubing	Weekly
	Clean autosampler, change tubing	As needed*
	Clean nebulizer and torch assembly	Weekly*
	Replace torch	As needed*
	Clean cones	Weekly*
	Refill rinse water receptacle	Daily
	Empty waste receptacle	Daily
	Check for internal standard and sample flow through peristaltic pump tubing	As often as possible
	Replace internal standard solution receptacle	As needed
	Check water level and water filter on recirculating-cooling unit, refill and replace filter	Check daily, refill and replace as needed
pH Meters	Clean or replace electrode	As needed
	Refill electrode electrolyte	As needed
Balances	Clean pan and platform	Before each use
	Check Level bubble	Daily
	Check calibration	Daily
	Check sensitivity	Weekly
	Cleaning and calibration by authorized service	Annually
Conductivity Meter	Clean probe	As needed
Dissolved Oxygen Meter	Replace membrane	As needed
	Clean probe	As needed
ZHE Vessels	Replace o-rings and screens	As needed
TCLP and ZHE Tumblers	Check Rotation Rate	Monthly
Spectrophotometers	Clean and check tubing	As needed
	Verify wavelength calibration	Six months
Mechanical Pipettes	Clean and check calibration	Quarterly*
Thermometers	Check calibration	Annually, Quarterly for Digitals, Semiannual for IR Thermometer*
Ovens	Check and/or adjust temperature, record temperature on log sheet	Daily
Refrigerators and Freezers	Check and/or adjust temperature, record temperature on log sheet	Daily
	Defrost freezers	As needed
Flow Injection Analyzer (Lachat)	Replace* or Rinse 100mL syringes on autosampler	Daily
	Replace tubes on autodilutor	As needed*
	Clean autosampler surfaces	As needed
	Spray silicone on cloth and rub on pump rollers	As needed
	Clean or replace o-rings and ports on valves	As needed*
	Clean union and T's on manifold and replace o-rings on manifold	As needed
	Dry and clean detector surfaces	As needed
	Replace flow cell o-rings and flares	As needed*
	Replace manifold tubing	As needed*

Preventative Maintenance Procedures For Laboratory Equipment

Instrument/ Equipment Type	Maintenance	Frequency
Hg Analyzer (FIMS)	Adjust Pump timing	As needed
	Change Argon supply tank	As needed*
	Change drying tube	Daily or as needed
	De-clog drying tube and/or reductant tubing	Daily or as needed
	Change system tubing	2-3 weeks*
	Rinse tubing prior to operation and following operation	Daily
	Clean optical cell	As needed (when aperture is out of line)

SECTION 21. MEASUREMENT TRACEABILITY

21.1 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. (Refer to Section 20.3). With the exception of Class A Glassware and Glass microliter syringes, 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. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

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), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia–Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- 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. The receipt of all reference standards must be documented. Reference standards are labeled with a unique standard ID number and expiration date. All documentation received with the reference standard is retained as a QC record and references the standard ID 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. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered 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 (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 the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

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 TestAmerica's Corporate SOP (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 appropriate area (e.g., Metals, Organics, etc.) and are also scanned into the reagent record in LIMS. 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 also the the laboratory's method specific SOPs, as well as general laboratory SOPs CF-SS-02 (*Standard Preparation, Documentation, and Tracking*) and CF-SS-03 (*Reagent Preparation, Documentation, and Tracking*).

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.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique ID number. 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; unique container number(s)
- Solvent type and lot number
- Preparation Date
- Expiration Date
- 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.

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

- Expiration Date (include prep date for reagents)
- Standard ID from LIMS
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. For items supplied through consignment, the date of receipt is equivalent to the date the item was taken out of consignment. Special Health/Safety warnings must also be available to the analyst. This information is always available in the material's Safety Data Sheet (SDS), accessible on the company intranet website.

21.4.3 In addition, the following information may be helpful:

- 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)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an 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 preparation/analytical batch records.

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 as specified in the laboratory SOP.

SECTION 22. SAMPLING

22.1 Overview

The laboratory's main responsibility in the sample collection process lies in supplying clients with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory.

The laboratory provides limited field services to local clients only. The laboratory typically provides the following field services:

- Wastewater Sampling
- Field Parameter Analysis (Field pH, Field Temperature, Field Residual Chlorine)

Field sampling procedures are described in laboratory SOP CF-FSS-01, *Sampling Procedures*. Field parameter analysis procedures are described in laboratory SOP CF-FSS-02, *Field Analysis Procedures*.

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

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

22.3 Definition of Holding Time

The date and time of sampling documented on the 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, etc.), the holding time is based on calendar day measured. Maximum allowable holding times expressed in "hours" (e.g., 8 hours, 24 hours, etc.) are measured from

date and time zero. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling, regardless of how long the holding time is.

The holding time of composite samples begins at the end of sample compositing, not at the collection of the first sample aliquot in the composite. The EPA interprets the hold time to be the period of time that has elapsed between the end of sampling and the beginning of preparation (or analysis).

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times 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.

22.5 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.

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.

Guidelines on taking sample aliquots & subsampling are located in laboratory SOP CF-GP-24, *Subsampling and Sample Homogenization*.

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is 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 23-1.

23.1.1 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 23-1). 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 and time 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.

When the sampling personnel deliver the samples directly to TestAmerica personnel, 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 personnel. 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 service center. When sampling personnel deliver the samples through an independent carrier (e.g., Fed-Ex, UPS, etc.), the COC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent carriers and lab couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler.

23.2 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. Refer also to SOP CF-SRV-01, *Customer Service and Login Procedures*, for further information.

23.2.1 Laboratory Receipt

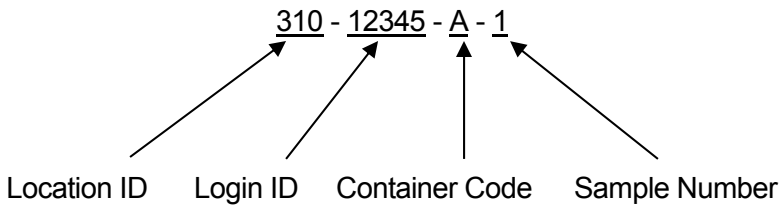
When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. A cooler receipt form is filled out (Figure 23-3). Any non-conformance, irregularity, or compromised sample receipt must be documented in LIMS and brought to the immediate attention of the PM or PMA, who will in turn contact 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.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This PRIMARY container ID is made up of the following information (consisting of 4 components):

Example:



The above example states the sample was logged in at the TestAmerica Cedar Falls laboratory (location 310). The login (job) ID is 12345 (unique to each client's particular job occurrence or sample delivery group). The container code indicates it is the first container ("A") of the first sample ("1"). Subsequent containers of the same sample would be assigned container codes of "B", "C", "D", etc.

If the sample or portion of sample from the primary container goes through a preparation step that creates a "new" container associated to that sample, then the new container is considered SECONDARY and gets another unique ID. For example, a client sample received in a 1-Liter amber bottle may be prepared by a liquid/liquid extraction, and an vial of solvent extract is created from this step. The vial would be a secondary container. The secondary container ID has 5 components:

Example: 310 - 12345 - A - 1 - **A** ← Secondary Container Occurrence

This example would indicate the sample from the primary container listed above went through a step that created the first occurrence of a secondary container ("A").

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) 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 (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- 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.

23.3.1 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 appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks 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 policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. CF-SRV-01, Customer Service and Login Procedures.

23.4 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, freezers or protected locations suitable for the sample matrix. 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 the sample container 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, including empty sample containers, are returned to the secure sample control area. All 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 the sample archive area where they are disposed of. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be 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.

23.5 Hazardous Samples and Foreign Soils

Refer to laboratory SOP No. CF-SRV-01 and Section 13 of the safety manual for the handling of hazardous samples.

The laboratory does not knowingly accept soil samples from foreign countries, U.S. territories, or areas within the United States that are under Federal Domestic Soil Quarantine.

23.6 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). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). 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.

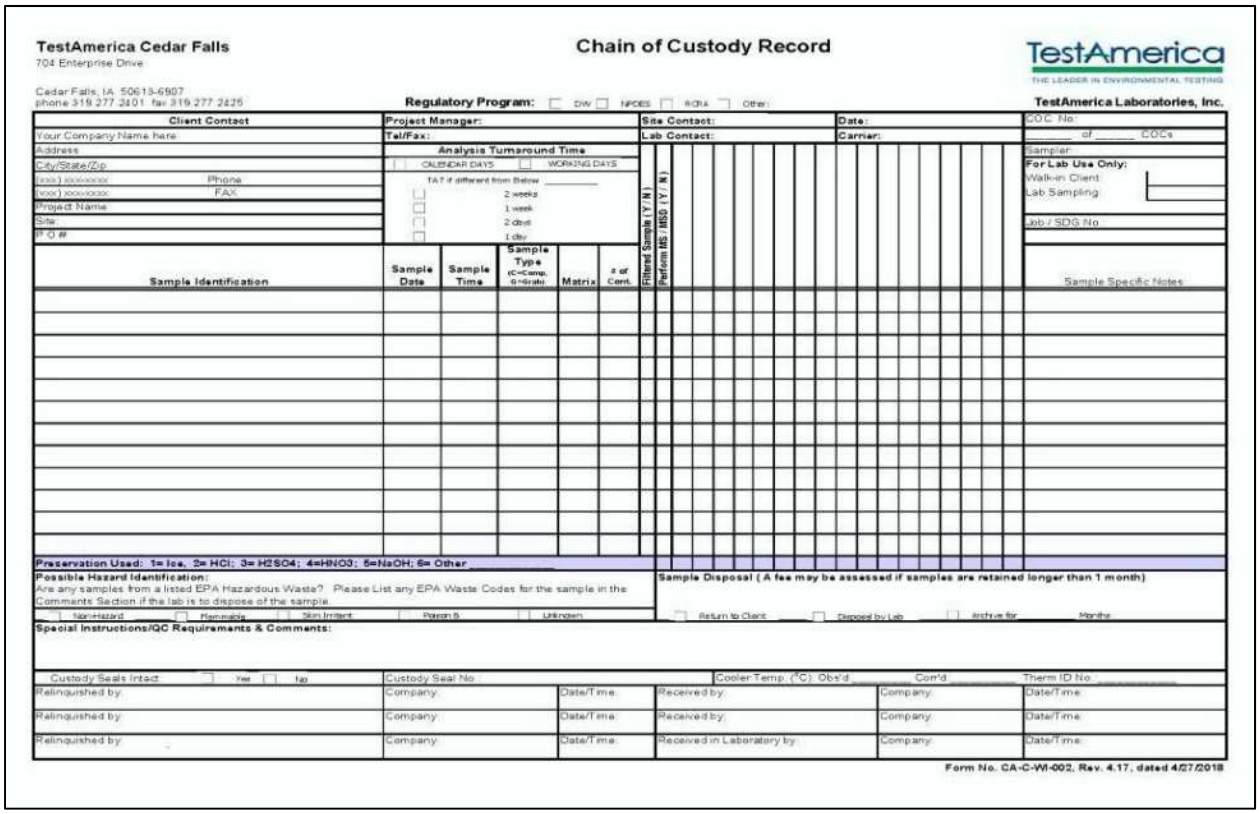
Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 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, 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 (SOP No. CF-WD-01). 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, return to 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 should be completed.

Figure 23-1. Example: Chain of Custody (COC)



TestAmerica Cedar Falls
704 Enterprise Drive
Cedar Falls, IA 50613-6907
phone 319.277.2401 fax 319.277.2425

Chain of Custody Record

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TestAmerica Laboratories, Inc.

Regulatory Program: ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

Client Contact	Project Manager:	Site Contact:	Date:	COC No:
Your Company Name here	Tel/Fax:	Lab Contact:	Carrier:	____ of ____ COCs
Address	Analysis Turnaround Time			Sampler:
City/State/Zip	☐ CALENDAR DAYS ☐ WORKING DAYS			For Lab Use Only:
(xxx) xxx-xxxx Phone	TAT if different from Below ____			Walk-in Client:
(xxx) xxx-xxxx FAX	☐ 2 weeks			Lab Sampling:
Project Name:	☐ 1 week			
Site:	☐ 2 days			Job / SDG No.:
P O #	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type (C=Comp, G=Grab)	Matrix	# of Cont.	Filtered Sample (Y / N)	Perform MS / MSD (Y / N)	Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification:
Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample.
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ Return to Client ☐ Disposal by Lab ☐ Archive for ____ Months

Special Instructions/QC Requirements & Comments:

Custody Seals Intact: ☐ Yes ☐ No | Custody Seal No.: | Cooler Temp. (°C): Obs'd ____ Corr'd ____ | Therm ID No.: ____

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received in Laboratory by:	Company:	Date/Time:

Form No. CA-C-WI-002, Rev. 4.17, dated 4/27/2018

Figure 23-2. Example: Sample Acceptance Policy

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704 Enterprise Drive • Cedar Falls, IA 50613
800-750-2401 • 319-277-2425 Fax

Sample Acceptance Policy

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 noted on the laboratory report defining the nature and substance of the variation. In these instances, the client will be notified either by e-mail, telephone, or fax as soon as possible after the receipt of the samples.

Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

1) Samples must arrive with labels intact with a Chain of Custody (COC) filled out completely. The following information must be recorded:
   - Client name, address, phone number, and fax number (if available)
   - Project name and/or number
   - Sample Identification
   - Date, time, and location of sampling
   - Collector's name
   - Sample matrix description
   - Sample container description
   - Number of each type of container
   - Preservatives used
   - Analysis requested
   - Requested turnaround time (TAT)
   - Special instructions, as necessary
   - 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
   - The date and time of receipt must be must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab. These date and times must be exactly the same.
   - Information must be legible
2) Sample(s) must be properly labeled:
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date, time, and sampler's ID
   - Include preservative used
   - Information must be legible in indelible ink
3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested. For information or additional guidance on sample containers, refer to your Project Manager and/or TestAmerica Corporate QA document no. CA-Q-WI-025, *Sample Reference Guide*.

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Figure 23-2. Example: Sample Acceptance Policy (continued)

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Sample Acceptance Policy

4) Samples must be preserved according to the requirements of the requested analytical method. For information or additional guidance on sample preservation, refer to your Project Manager and/or TestAmerica Corporate QA document no. CA-Q-WI-025, *Sample Reference Guide*.

5) Most analytical methods require chilling samples to 4°C. For these methods, the criteria are met if the samples are chilled to at or below 6°C and above 0°C (exceptions to the chilling requirement would include samples submitted for metals analysis or bulk asbestos samples). For methods with other temperature criteria, the samples must arrive within ± 2°C of the required temperature or within the method-specified range (e.g., some bacteriological methods require <10°C).

   - Samples that are hand-delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case, the samples shall be considered acceptable as long as there is evidence that the cooling process has begun (i.e., received on ice).
   - If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.
   - Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample with fifteen (15) minutes of collection.

6) Chemical preservation will be verified prior to analysis and documented, either in sample control or at the analysts' level. The Project Manager will be notified immediately if there is a discrepancy. If analysis will still be performed, all affected results will be flagged to indicate improper preservation.

7) Sample Holding Times:

   - TestAmerica will make every effort to analyze samples within the regulatory holding time (HT). Samples must be received at the laboratory with enough time to perform the sample analysis. Except for short hold time analyses (≤48 hr HT), samples must be received with at least 48 hours (two working days) remaining on the holding time to ensure analysis.
   - Analyses that are designated as "field" analyses (e.g., pH, Dissolved Oxygen, Residual Chlorine, Carbon Dioxide, Sulfite, Redox Potential) should be analyzed ASAP by field sampling personnel (within 15 minutes of sampling) prior to delivering to the lab. However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt at the testing laboratory (excluding weekends). Samples analyzed in the laboratory for field parameters will be qualified on the final report with an 'HF' to indicate holding time exceedance.
   - For information or further guidance on sample holding times, refer to your Project Manager and/or TestAmerica Corporate QA document no. CA-Q-WI-025, *Sample Reference Guide*.

8) All samples submitted for volatile organic analyses should have a Trip Blank submitted with the samples. If requested, TestAmerica will supply a Trip Blank with all bottle orders containing volatile organic analyses.

9) The Project Manager will be notified if any sample is received in damaged condition. TestAmerica will then contact the client with the details of the sample condition upon receipt and request further instruction from the client.

10) Recommendations for packing samples for shipment:

   - Pack samples on ice rather than "Blue" ice packs
   - Soil samples should be placed in plastic, sealable bags. The containers often have dirt around the top of the jar and do not seal very well, thus are prone to intrusion from the water from melted ice.
   - Water samples are best wrapped with bubble wrap or bubble sleeves, and then placed in plastic, sealable bags.
   - Fill extra cooler space with bubble wrap.

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Figure 23-3. Example: Cooler Receipt Form

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Place COC scanning label here

Cooler/Sample Receipt and Temperature Log Form

Client Information				
Client:				
City/State:			Project:	
Receipt Information				
Date/Time Received:			Received By:	
Delivery Type: ☐ UPS ☐ FedEx ☐ FedEx Ground ☐ US Mail ☐ Spee-Dee ☐ TA Courier ☐ TA Field Services ☐ Client Drop-off ☐ Other:_____				
Condition of Cooler/Containers				
Sample(s) received in Cooler?	☐ Yes	☐ No	*If yes:* Cooler ID:	
Multiple Coolers?	☐ Yes	☐ No	*If yes:* Cooler # _____ of _____	
Cooler Custody Seals Present?	☐ Yes	☐ No	*If yes:* Cooler custody seals intact? ☐ Yes ☐ No	
Sample Custody Seals Present?	☐ Yes	☐ No	*If yes:* Sample custody seals intact? ☐ Yes ☐ No	
Trip Blank Present?	☐ Yes	☐ No	*If yes:* Which VOA samples are in cooler? ↓	
Temperature Record				
Coolant: ☐ Wet ice ☐ Blue ice ☐ Dry ice ☐ Other:_____ ☐ NONE				
Thermometer ID:			Correction Factor (°C):	
• **Temp Blank Temperature** – If no temp blank, or temp blank temperature above criteria, proceed to Sample Container Temperature				
Uncorrected Temp (°C):			Corrected Temp (°C):	
• **Sample Container Temperature**				
Container type(s) used:				
Uncorrected Temp (°C):			Corrected Temp (°C):	
Exceptions Noted				
1) If temperature exceeds criteria, was sample(s) received same day of sampling? ☐ Yes ☐ No a) *If yes:* Is there evidence that the chilling process began? ☐ Yes ☐ No				
2) If temperature is <0°C, are there obvious signs that the integrity of sample containers is compromised? (e.g., bulging septa, broken/cracked bottles, frozen solid?) ☐ Yes ☐ No				
Note: If yes, contact PM before proceeding. If no, proceed with login				
Additional Comments				

Document: CF-LG-WI-002
Revision: 23
Date: 12/31/2017

TestAmerica-Cedar Falls

General temperature criteria is 0 to 6°C
Bacteria temperature criteria is 0 to 10°C

SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continually evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Method 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. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization/subsampling, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying, concentration, and/or cleanup. 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.

24.3 Negative Controls

Table 24-1. Example: Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	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.
	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. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	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.
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.

Table 24-1. Example: Negative Controls

Control Type	Details
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or 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.
Field Blanks [1]	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)
Equipment Blanks [1]	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. (TNI)
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

[1] When known, these field QC samples 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."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.3.1 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:

Table 24-2. Negative Controls for Microbiology

Control Type	Details
Sterility Checks (Media)	are analyzed for each lot of pre-prepared media, ready-to-use media and for each batch of medium prepared by the laboratory.
Filtration Blanks	blanks are run at the beginning and end for each sterilized filtration unit used in a filtration series. For pre-sterilized single use funnels a sterility check is performed on at least one funnel per lot.
Sterility checks (Sample Containers)	are performed on at least one container per lot of purchased, pre-sterilized containers. If containers are prepared and sterilized by the laboratory, one container per sterilization batch is checked. Container sterility checks are performed using non-selective growth media.
Sterility Checks (Dilution Water)	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.
Sterility Checks (Filters)	are also performed on at least one filter from each new lot of membrane filters using non-selective growth media.

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.

24.4 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)), which entails both the preparation and measurement steps; and (2) matrix effects (Matrix Spike (MS)) [matrix spikes are not applicable to air] or sample duplicate (DU, DUR), 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.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

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.

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.

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.).

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.

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.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- 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.

24.4.2 Positive Controls for Microbiological Methods

- 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.
- In addition, every analytical batch also contains a pure culture of known positive reaction.
- A pure culture of known negative reaction is also tested with each analytical batch to ensure specificity of the procedure.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Controls

Control Type	Details	
Matrix Spikes (MS)	Use	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;
	Typical Frequency [1]	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. 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. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	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. 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.
	Description	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.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (DU or DUR) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.

Table 24-3. Sample Matrix Controls

Control Type	Details	
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP/ICPMS methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] 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.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific 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.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized. Refer also to laboratory SOP CF-QA-04, *Quality Control Limits*.

Laboratory generated percent recovery acceptance (control) limits are generally established by taking ± 3 standard deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- 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).
- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for poor-performing analytes such as Benzidine will be 5% and the analyte must be detectable and identifiable.
- The maximum acceptable recovery limit will be 150%.

- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.
- If either the high or low end of the control limit changes by ≤ 5% from previous, the control chart is visually inspected and, using professional judgment, the control limit may optionally be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.1 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.

24.6.2 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 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Or, for TNI 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

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).
- 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.

Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

More detail on the marginal exceedance procedures used by the laboratory is found in laboratory SOP CF-QA-09, *Random Marginal Exceedances*.

24.6.3 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 lab's method SOPs and in Section 12.

24.6.4 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).

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratory's sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 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 conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

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 19.

25.2 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 or his/her designee. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g., Analytical Report) with a "sample results" column header.

25.2.2 Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. job 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 XX of YY, where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC). Any COCs involved with subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact.

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 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.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc.).

25.2.11 Practical quantitation limits or reporting limits.

25.2.12 Method detection limits (if requested).

25.2.13 Definition of data qualifiers and reporting acronyms.

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17 A statement to the effect that the results relate only to the items tested and the sample(s) as received by the laboratory.

25.2.18 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.19 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.20 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.

25.2.21 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.22 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.23 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, or preliminary report). A complete report must be sent once all of the work has been completed.

25.2.24 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.25 A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level 1 is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data).
- Level II is a Level I report plus summary information, including results for the method blank, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. Faxed reports are followed by hardcopy, if requested by client. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services in addition to the test report as described in Section 25.2. When TNI accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. TestAmerica Cedar Falls offers a variety of EDD formats including EQUIS (client-specific formats), Illinois EPA, MPCA, Missouri TerraBase, Excel (multiple formats), and Text files.

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.

25.4 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.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

25.4.1 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.

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.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CW-L-S-004).

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 TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 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.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are to meet all requirements of this document, including cover letter.

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

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 12).

The revised report is retained on the document server in the LIMS, as is the original report and all other project documents. The revised report can be accessed through PM Desktop under the Job Number in the Deliverable folder. The revised deliverable is identified as Rev(1) next to the report which as been revised. Any further revisions would be identified as Rev(2), Rev(3), etc.

When the report is re-issued, a notation of "Revision: X" is placed on the cover/signature page of the report, where "X" is the number of revision. A brief explanation of reason for the re-issue is placed at the top of the Job Narrative page. (For example: *Report was revised on 11/3/17 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/08 at 10:47am.*)

25.9 Policies on Client Requests for Amendments

25.9.1 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 undetected. This policy has few exceptions. Exceptions are:

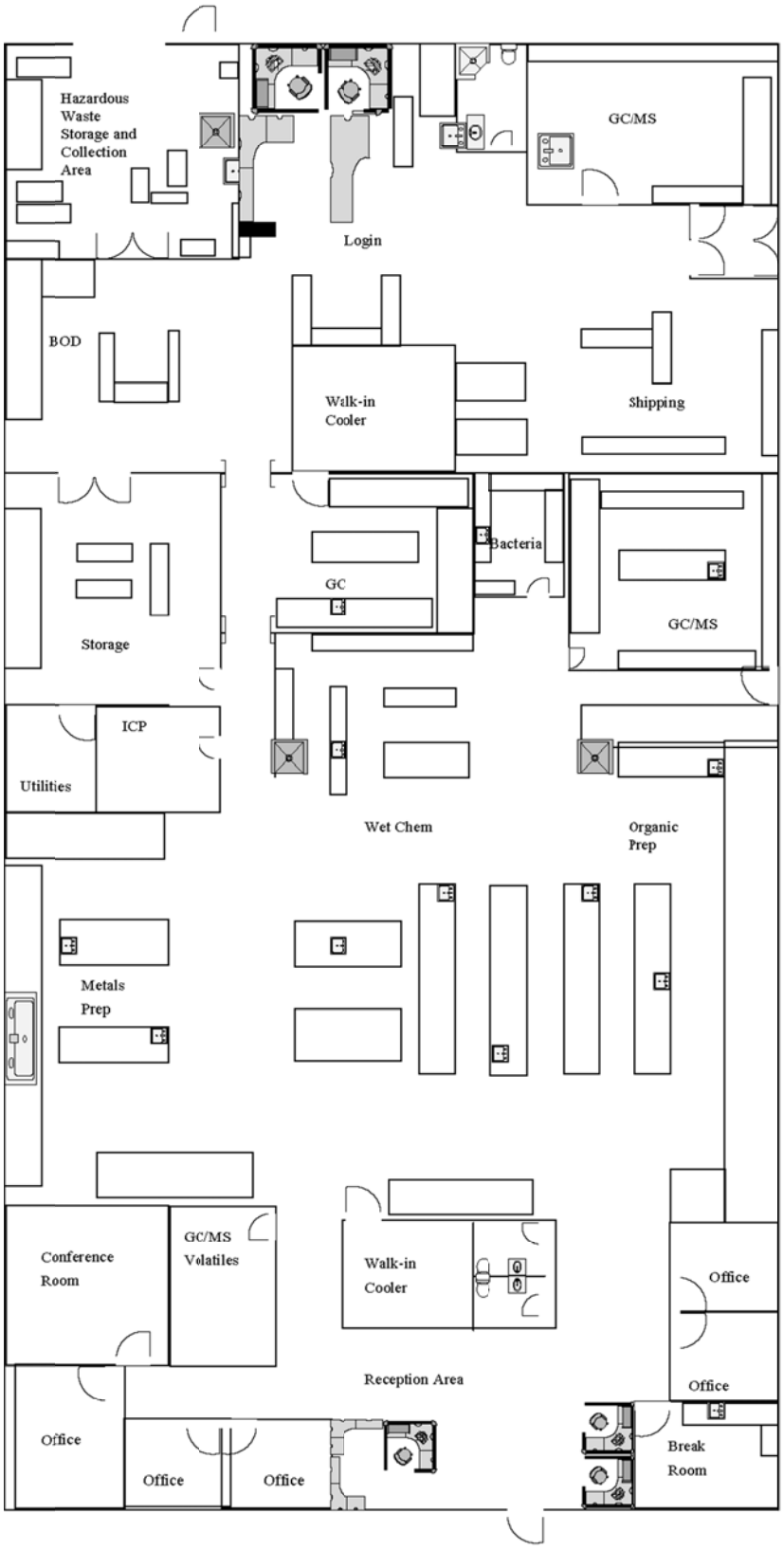
- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).

- 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 *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order 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.

Appendix 1. Laboratory Floor Plan



Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

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.

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.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

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 laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that 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 (1) to twenty (20) environmental samples of the same quality systems 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 twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

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)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

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.

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. TNI and its representatives agree to safeguard 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. (TNI)

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)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. 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 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).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

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)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

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 Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

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 Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): 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 of the procedure unless otherwise noted in a reference method. 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.

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.

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 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

LOD Verification [a.k.a., MDL Verification]: A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

(QS) 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 or Saline/Estuarine. 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.

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 & Emissions: 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 sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test

result 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 Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

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.

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)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

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.

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.

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. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

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. (TNI)

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. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item or service is of the type of quality needed and expected by the client. (TNI)

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 that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

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. (TNI)

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 activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved 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 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

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.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedure (SOP): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

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.

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 Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

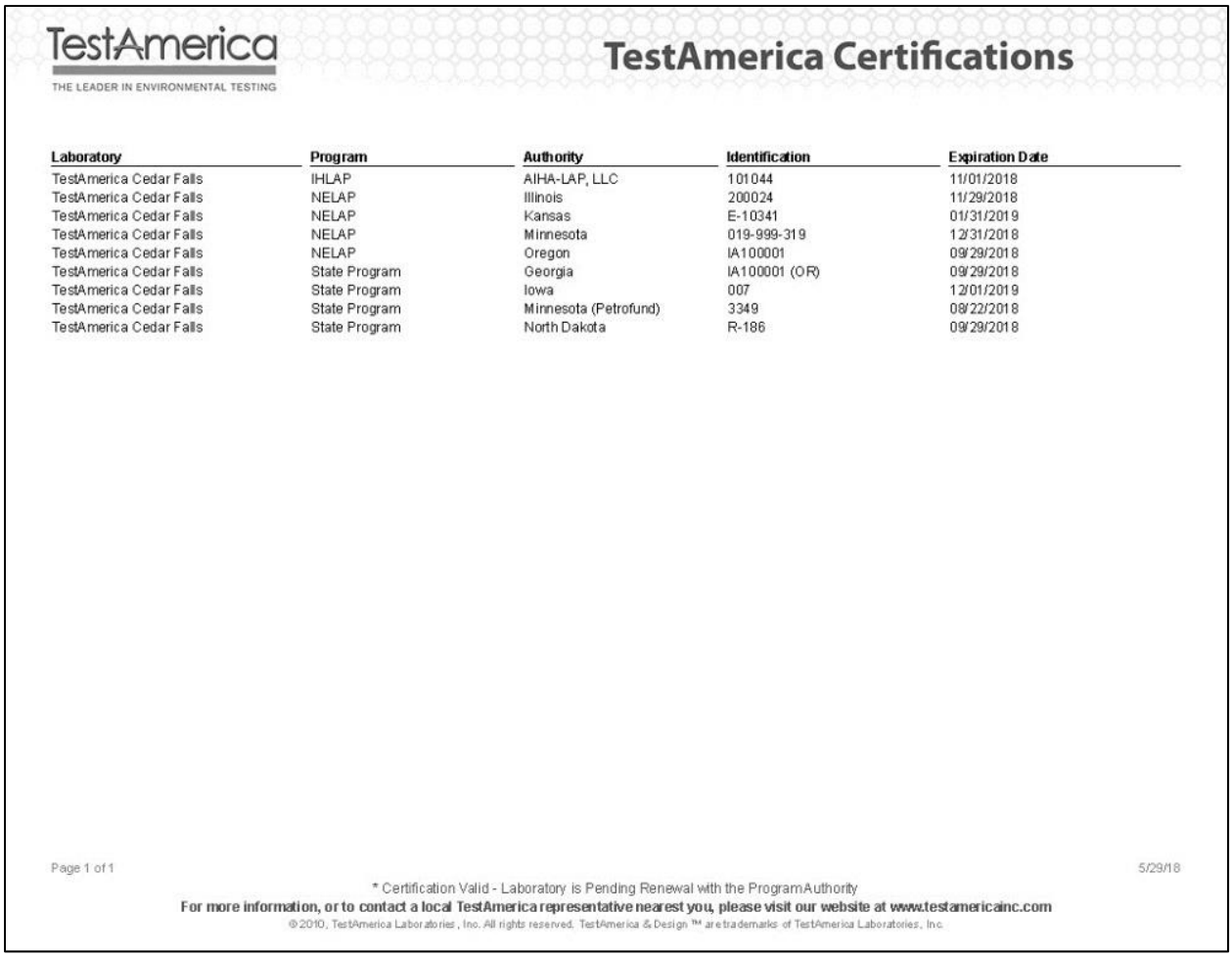
Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
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
ICP/MS – ICP/Mass Spectrometry
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
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
SDS - Safety Data Sheet
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
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 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Cedar Falls maintains accreditations, certifications, and approvals 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:



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Certifications

Laboratory	Program	Authority	Identification	Expiration Date
TestAmerica Cedar Falls	IHLAP	AIHA-LAP, LLC	101044	11/01/2018
TestAmerica Cedar Falls	NELAP	Illinois	200024	11/29/2018
TestAmerica Cedar Falls	NELAP	Kansas	E-10341	01/31/2019
TestAmerica Cedar Falls	NELAP	Minnesota	019-999-319	12/31/2018
TestAmerica Cedar Falls	NELAP	Oregon	IA100001	09/29/2018
TestAmerica Cedar Falls	State Program	Georgia	IA100001 (OR)	09/29/2018
TestAmerica Cedar Falls	State Program	Iowa	007	12/01/2019
TestAmerica Cedar Falls	State Program	Minnesota (Petrofund)	3349	08/22/2018
TestAmerica Cedar Falls	State Program	North Dakota	R-186	09/29/2018

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* Certification Valid - Laboratory is Pending Renewal with the Program Authority

For more information, or to contact a local TestAmerica representative nearest you, please visit our website at www.testamericainc.com

The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications.

APPENDIX F

STANDARD OPERATING PROCEDURES

1.0 Operation

This section discusses the methodologies for maintenance and use of photoionization detectors (PID) in the field in order to screen for volatile organic compounds (VOCs).

2.0 Objectives and Rationale

The following section summarizes the field procedures and handling for PIDs to ensure accurate results and good maintenance of the equipment. This SOP should be used in conjunction with the user's guide for each PID; these are stored with the appropriate PID for reference and should be taken into the field with when the PID is beingused.

3.0 Equipment and Maintenance

Blackstone owns a MiniRAE 3000. This PIDs are stored in a secure location with the user's guide, battery chargers, spare lamps (10.6 and 11.7 eV), spare filters, calibration gas and pressure gauges. For PID-specific information, refer to the user's guide for a step-by-step process of using each PID. Each PID will require laboratory calibration. This is completed to verify that PID is in good working condition. The PID may be sent in for laboratory calibration more frequently if it is malfunctioning and replacing the lamp and filters does not remedy the problem.

Prior to using to using the PID it is good practice to charge the PID for at least 10 hours. Confirm that the PID is fully charged by checking the battery icon on the PID display. These units can also be powered using disposable batteries and these should be stored and carried with the PID in case of an emergency.

Prior to leaving the field, confirm that all the PID components are properly stowed in the carrying case.

4.0 Calibration

Calibration of the PID should be conducted onsite each time it is used at the beginning and ending of the field activities. If field activities take longer than a day then the PID should be recalibrated at the beginning and ending of each day. Calibration of the PID should also be completed after replacing the lamp and allowing for a five-minute run period prior to calibration. Calibration completion and results of the calibration should be recorded.

PID calibration is a two-point calibration including fresh air and span gas. Before collecting any PID samples, calibrate the PID starting first with fresh air and then completing the span gas calibration. If the fresh air calibration does not result in a 0.0 ppm reading (±10%) then attach the carbon filter and complete the fresh air calibration again. After successfully completing the fresh air calibration, calibrate the PID using the span gas.

Iso-Butylene is the span gas that is used to complete the second step of the calibration. This calibration should result in a reading of 100 ppm (±10%). Once this is completed, sample

collection can begin. If the calibration is not completed within this range, repeat the span gas calibration. If this is not successful, check that all the filters are placed correctly in the PID, restart the device, and complete the span gas calibration again. If the calibration is still leading to a result that is out of the acceptable range then replace the lamp with a lamp of the same rating, allow for the PID to run for at least five minutes, and then complete the two-point calibration starting with the fresh air calibration.

5.0 Screening Procedure

All samples collected in association with a sampling event should be treated in the same manner so that the results of screening are comparable. Prior to collecting a PID reading, the soil sample should be characterized and then placed in a re-sealable baggie. Fill the baggie to ½ full. Wait ten minutes before screening the sample. Then, disturb a small portion of the soil sample in the sealed baggie. Next the baggie should be opened to less than one inch at the top and the PID sensor should be inserted into the baggie. Close the opening around the PID sensor and record the value on the PID once it has stabilized. Next, remove the PID sensor from the baggie and reseal it completely. Additional field screening may be needed per the sampling plan or as specified by the project manager; however, the PID should be the first field screening instrument used when multiple instruments are to be used. After all the field screening is complete, collect the soil sample for analysis from the remaining undisturbed portion of the soil interval within the baggie into the appropriate sample containers.

The PID should read 0.0 after it is removed from the baggie. Allow for the PID to decrease to a value of 0.0 before using the PID to collect another reading. If the PID does not appear to be responding to the samples or appears to be acting correctly, confirm that the PID is operating correctly by completing a bump test with the calibration gas. Complete any calibration and repairs if the device is not functioning correctly.

The PID should be allowed to run during field activities to monitor the ambient air conditions. If a sustained level of 5 ppm or any unusual chemical odor is observed for at least five minutes, personnel will don a half-face air purifying respirator.

1.0 Sample Custody Procedures

Sample custody procedures are based on EPA-recommended procedures which emphasize careful documentation of sample collection and sample transfer. To ensure that all the important information pertaining to each sample is recorded, the documentation procedures listed herein will be executed.

In order to maintain and document sample custody, the following custody procedures will be strictly followed. A sample is considered to be under custody if:

- It is in actual possession of the responsible person.
- It is in view, following physical possession.
- It is in the possession of a responsible person and is locked or sealed to prevent tampering.
- It is in a secure area awaiting transfer of custody.

2.0 Chain-of-Custody Record

Sample custody is documented by a "Chain-of-Custody Record". The custody record is completed by the individual designated by the Project Manager as being responsible for sample handling and shipment to a designated laboratory. The information recorded will include:

Project Manager	Print the name of the project representative to whom lab reports and correspondence are to be addressed.
Sampler Name	Provide name and signature of sampler/samplers.
Project Number	Print the project number. Same information to be used on lab reports.
Sample I.D.	Write the identification number of the sample.
Date/Time Sampled	Date and military time the sample was collected.
Sample Material	Print the type of material sampled.
Special Instructions	In the special instructions section of the chain-of-custody form; sample method, turnaround time, or other conditions of note may be listed as required in the sampling plan or deemed appropriate in the field.

Analysis Required	Print or mark the type of analysis and laboratory quantification levels required. Make sure these levels are at least as low as required. Detection limits may be specified in special instructions box.
Relinquished By	Print the name of the person giving up the sample and provide signature. First person to relinquish must be same as sampler.
Company	Print the name of the organization giving up the sample.
Date/Time	Print the date and time at which the sample was given.
Received By	Obtain the signature of the receiving person.
Organization	Print the name of the receiving organization.
Date/Time	Print the date and time at which the sample was received.

The chain-of-custody form will consist of an original and two carbon copies. The sampler will retain the second carbon copy and ship the original and first carbon copy to the lab.

3.0 Transfer of Custody

The field personnel initially taking the sample(s) are responsible for the care and custody of the sample(s) until it is properly transferred or delivered to laboratory personnel. All samples must be accompanied by a chain-of-custody record, consisting of an original and carbonless copy. A second copy is retained in the Blackstone Environmental project file.

When transferring in possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. The condition of the shipping container and the samples if the shipping container is opened shall be noted on the chain of custody form or a sample receipt form, which will remain with the chain-of-custody. The company from which the sample is relinquished and to which it is delivered and the reason for transfer will be noted. This record documents the transfer of samples from the custody of the sampler to that of another person, or the permanent laboratory.

It is the Project Manager's responsibility to ensure that all shipping data are consistent and that they are made part of the permanent job file. The first noted person to relinquish must be the same as the sampler name.

1.0 EQUIPMENT DECONTAMINATION SOP

All non-disposable equipment will be thoroughly decontaminated in accordance to the following procedures prior to leaving a site or containerized for decontamination off site to prevent contamination from contaminating other sites. All non-disposable sampling equipment will be thoroughly decontaminated in accordance with the following procedures prior to collecting each sample, between sample points, and at the end of field activities to avoid cross-contamination. Under no circumstances is potentially contaminated equipment to be allowed to leave the site.

Decontamination Procedure (the sampling plan may require a different procedure)

1. Remove as much media from the equipment as possible prior to washing.
2. Wash the equipment using detergent water (Alconox is preferred if compatible with the equipment and analysis to be performed).
3. Rinse the material with tap water.
4. Repeat steps 2 and 3 twice more.
5. Rinse with deionized water.
6. *If required*,* rinse with another agent (methanol, acetone, etc.)
7. *If six is required,* rinse again with deionized water.
8. Place on a rack in a contaminant free area to air dry or wipe dry with a contaminant free towel.

**A solvent rinse is required in the event that oily sheen and or residue is observed. Capture this rinsate in a container provided by the analytical laboratory and return it to the analytical laboratory for proper disposal or containerize with site IDW for proper disposal; whichever is appropriate.*

Drilling Equipment

The drilling subcontractor is responsible for the decontamination of reusable drilling equipment (augers, etc.). The environmental professional should verify that decontamination procedures used by the driller are adequate and fully executed in between sampling points.

1.0 Investigative Derived Waste SOP

This SOP addresses the management and handling of investigation derived waste (IDW) which includes all soil, groundwater, decontamination rinse water and disposable sampling equipment (such as gloves, sampling filters, etc.) generated while completing environmental site assessment field activities.

Soil and drill cuttings will be thin spread on the subject property. Decontamination rinse water, including water mixed with Alconox (or similar) from each sampling event will be discharged to a permeable surface on the subject property following assessment activities.

Disposable sampling materials from each sampling event will be bagged and disposed of on-site if possible. If no dumpster is available to the field personnel on the subject property, then this material will be disposed of at the offices of Blackstone.

1.0 OBJECTIVES AND RATIONALE

The following sections summarize the procedures for lead-based paint sampling to ensure reliable samples and accurate reporting of all lead bearing substances using a certified x-ray fluorescence (XRF) analyzer. This SOP should be used in conjunction with the XRF SOP (See Appendix F of QAPP) and the XRF user manual. The user manual will be stored with the device.

The certified XRF analyzer will have proper leak tests performed on the analyzer every six months with documentation. The analyzer will be properly stored and braced during transportation and placed as far away from the driver and passengers as possible.

The inspector performing the inspection has been certified through the Iowa Department of Public Health (IDPH) and appropriately trained to safely operate the XRF analyzer.

2.0 FIELD SAMPLING PROCEDURE

During the inspection pre-calibration and post-calibration tests will be performed on the analyzer. These tests will be executed by taking the average of 3 readings performed on a 1.02 mg/cm2 SRM paint film from NIST and then compared to the PCS for the device. Note: a calibration test will also be taken every 4 hours or at the end of the inspection, whichever is sooner. In addition to calibrating the analyzer, a detailed identification scheme illustration will be documented and labeled according to how samples were obtained.

The standard procedure for sampling is to test one component type in each room equivalent. All sampling combinations will be recorded and documented in the report. A room equivalent is defined as an identifiable part of a building such as a room, exterior side, or exterior area of a building (e.g. kitchen, hallway, bathroom, side of exterior).

A component type is made up of a component and substrate. A component is defined as separate parts of a room equivalent (e.g. floor, wall, window sash, door). A substrate is defined as the material of which the component is made (e.g. wood, brick, drywall, metal, plaster, concrete).

1.0 Operation

This section discusses the methodologies to be used for maintenance and use of the portable X-ray fluorescence (XRF) device in the field.

2.0 Objectives and Rationale

The following section summarizes the field procedures and handling of the XRF to ensure accurate results and good maintenance of the device. This SOP should be used in conjunction with the user's manual. The user's manual is stored with the XRF and it travels with the device into the field.

3.0 Equipment and Maintenance

Blackstone operates an Innov-X Systems XRF Analyzer. The XRF is stored in a secure location with the user's manual, iPAQ and iPAQ accessories, battery and battery charger assembly, power cord, standardization clip, and check standards. For XRF step-by-step instructions and information, refer to the user's manual.

Prior to using the XRF, make sure both batteries are fully charged. If the XRF is going to be used over several days, charge both batteries overnight so that the XRF can used the following day. The XRF batteries have a charge indicator; check both before taking the XRF into the field to see that they are fully charged.

Prior to leaving the field at the end of the day, confirm that all of the XRF components and accessories are properly stored away.

4.0 Standardization and Calibration

Standardization of the XRF should be conducted onsite each time it is used, at the beginning of field activities each day. Standardization of the XRF should also be completed after replacing the battery or iPAQ unit, restarting the XRF or iPAQ, or after four hours of use.

Following each standardization, the SRM check standards should be analyzed for at least 1 minute (60 seconds). Results of the calibration check should be recorded on the calibration check log which is stored in the XRF field case. If the measured calibration results have a range that exceed 20% of the reported value for arsenic and lead than increase the exposure time to 120 seconds and reshoot the standard.

After turning on the XRF and iPAQ unit and initiating the Innov-X Systems Analyzer software, a standardization request will appear on the iPAQ. Place the standardization clip, Alloy 316, on the XRF, in front of the analyzing window. Once this is done, start the calibration. The XRF will

complete the standardization and will report if it was successful or if any errors were encountered. If the standardization results in errors, check that the clip is situated correctly on the front of the XRF unit and complete the standardization again. If it fails again, restart the XRF and replace the battery and try again. If this is unsuccessful, the XRF offers a soft reset option. If the soft reset standardization is not successful, then contact Innov-X Systems service center at 781-938-5005 for further assistance.

5.0 Sampling Procedure

Prior to screening the soil, the soil sample should be placed in a re-sealable baggie and agitated to homogenize the sample. Place the sealed bag on a flat surface and then place the XRF directly on the face of the bag. The soil sample should completely fill the XRF's analyzer window. The soil sample should have a sample thickness of 0.5 inches to provide an accurate result using the XRF.

Engage the instrument and shoot through the bag into the soil for 60 seconds. Record the XRF sample number and any parameters, as desired by the sampler. It is important to review the XRF results in the field, as the results of this filed screening tool are utilized to select the interval for lab analysis. In general, intervals with elevated arsenic and lead screening levels should be selected for laboratory analysis. All results will also be downloaded and saved in the project folder upon returning to the office.

If the XRF malfunctions in the field, restart the device and replace the battery. If it continues to malfunction, check the user manual for what may be causing the issue and call the Innov-X Systems service center at 781-938-5005, if needed.

When the XRF is turned on but not in use, the trigger lock should be on. This prevents accidental radiation exposure to the user and to other personnel on the site.

1.0 Objectives and Rationale

The following sections summarize the field procedures for soil sampling to ensure quality control and to secure fully representative samples that meet the requirements of the sampling plan. Soil samples can be collected from a variety of conditions and locations as required by a permit, a sampling plan, or the project manager. The sampling method may vary; however, basic protocol must be followed to ensure sample integrity, data utility, and employee, public, and environmental safety. Suitable collection equipment will be constructed of a compatible material that does not affect the sample media or the analytical constituents. All samples will be placed into an appropriate laboratory–provided sample container. The sample will then be labeled, logged, and placed in a cooler containing ice in order to preserve sample integrity.

Field personnel should always wear appropriate personal protective equipment while handling sampling equipment and containers, sampling soils, and decontaminating equipment. Disposable nitrile gloves should be changed as each soil horizon/interval is handled.

The procedures for soil collection presented in this SOP are designed to ensure the following:

- all samples and field measurements are consistent with the project objectives;
- samples are collected in a manner that provides the highest level of safety for field personnel, the public, and the environment;
- samples are collected efficiently and provide the highest achievable data quality; and
- ensure no cross-contamination between soil samples via good housekeeping procedures and proper equipment decontamination.

2.0 Sampling Equipment Methodologies

2.1 Surface Soils (soils to a depth of one foot)

Surface soils will be sampled utilizing a spade, scoop, hand auger, or trowel constructed of a material compatible with the analyte of interest and the material sampled. Unless otherwise specified by the sampling plan, a minimum of one inch of soil will be removed to expose fresh soil. The sample will then be collected from fresh soil using the appropriate sampling equipment.

2.2 Subsurface Soils (soils deeper than one feet)

Subsurface soils will be sampled by excavating (less than 5 feet or deeper in certain cases) or by drilling to the appropriate depth. Samples will not be collected from any drilling method requiring the use of drilling fluids or the flights of any drilling system unless specifically required in the sampling plan.

When collecting the sample with excavation type equipment the following procedure will be followed:

- Excavate to a depth within ½ of the excavation equipment's excavating capacity (½ a backhoe's bucket or ½ a spade full);
- Decontaminate the excavation equipment; and then

- Collect the sample remembering the sample is the deeper half of the material excavated.

When collecting the sample with drilling equipment follow the following procedure:

Hollow Stem Auger Drilling

- If a split spoon sampler is used with a liner constructed of compatible material:
- Drill to a depth within ½ the split spoon's length above the desired sample depth;
- Insert an uncontaminated liner; and
- Continue drilling to collect the sample per established drilling methods.
- If a split spoon sampler is used without a liner:
- Drill to a depth within ½ the split spoon's length above the desired sample depth;
- Decontaminate the split spoon per procedures listed in that section; and
- Continue drilling to collect the sample.

Bucket Augering

- Drill to a depth within ½ the bucket's length above the desired sample depth;
- Decontaminate the bucket
- Continue drilling to collect the sample.

Direct Push Technologies

- Push to within one foot of the desired depth;
- Remove the push rod and add the sampler section; and
- Re-push through the sample point.

In some cases, soil conditions will not allow for the removal of the push rod without the collapse of the push hole. In this case a continuous sample method may be employed. If continuous sampling is to be undertaken; the sampler should be decontaminated prior to collecting the sample of interest.

2.0 Soil Sampling Methodologies

This section discusses the methodologies to be used in collecting soil samples. The collection technique to be used will be based on the test parameters that will be analyzed. All methods utilized must be acceptable according to industry practices.

Sample horizons are chosen for laboratory analysis based on the site-specific sampling and analysis plan (SAP) in conjunction with field observations. Soil borings are logged for lithology and observations such as staining or odors. Field screening tools can be used in conjunction with the observations made by an environmental professional to provide the most useful information possible. Field observation relies on the skill and experience of the field personnel to visually identify signs of the analyte, materials related to the analyte, and process or site conditions that may indicate a relatively higher likelihood of the analytes presence. Experience used in conjunction with field screening, provides an effective tool in maximizing representative data

collection. Specific instruments utilized by Blackstone are listed and discussed in the equipment-specific SOPs.

Soil intervals are containerized in a re-sealable baggie after the material has been logged. Care should be taken to disturb the sample **as little** as possible during any transfer of soil to the baggie and the baggie should be sealed in between any activity prior the completion of the VOC/SVOC sample collection.

The sample is then screened as applicable according to the requirements of the SAP. When sampling for VOCs/SVOCs, the first field screening conducted should be for organic vapors. This is completed by inserting a PID into the baggie and collecting the field screening measurement. This measurement in conjunction with physical observations allows the field personnel the opportunity to select the appropriate soil interval for the VOC/SVOC samples to yield the most valuable data. PID screening should be conducted as quickly as possible from each soil interval's baggie and samples for VOC analyses should be taken promptly from the least disturbed portion of the soil interval within the selected soil-interval baggie. Note that the 2 Sodium Bisulfate vials receive 5 grams of soil (1 Terra Core® plug) and that the methanol vial receives 5 grams of soil (2 Terra Core® plugs). SVOC samples should be collected immediately following VOC sample collection from the remaining least disturbed portion of the soil interval within the selected soil-interval baggie.

XRF screening (when applicable) may be completed following PID screening and VOC/SVOC sample collection. Sample intervals within baggies should be broken up and homogenized prior to XRF screening and sampling for metals analysis.

Following the screening and sampling for VOC/SVOC analyses and metals analysis, samples may be taken from the homogenized baggies for any remaining analytical parameters specified by the SAP.

Method 5035A: Field Preservation, Collection and Handling instructions of Vials (Sodium Bisulfate / Methanol)

Materials

- Two sodium bisulfate preserved, pre-weighted vials per sample for low level analysis.
- One methanol preserved, pre-weighted vial per sample for medium-high level analysis
- One 4-ounce jar per sample for percent total solids determination
- 1 Terra Core

Instructions for Sample Collection

1. With plunger seated in the handle, push the Terra Core into exposed soil until the sample chamber is filled. A 5-gram sample will be collected with the plate is in place.
2. Wipe all soil or debris from outside of Terra Core sampler.

3. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.
4. Rotate the plunger that was seated om the handle top 90 degrees until it is aligned with the slots in the body. Place the mouth of the sampler into the 40ml VOA vial and extrude the sample by pushing the plunger down. Quickly place lid back on 40 ml VOA vial.
5. Repeat process for each additional vial.
6. A single Terra Core can be used to collect sample aliquots for each of the three vials.
7. Mark each sample container with your specific identification. Do not any additional labels or tape to the pre-tared vials. Store samples at 4°C. The holding time for VOC analysis is two weeks from the time of sample collection.
8. A fourth container needs to be submitted to the laboratory for percent total solid determination. Fill the provided container to capacity. If extractable organic analyses, i.e. semi-volatiles, PNAs, or pesticides/PCBs will be performed, the fourth container should be a 4-ounce jar.

Note: Methanol is a flammable substance. If samples will be shipped to the laboratory via couriers such as UPS or Federal Express, DOT requirements must be met.

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Recommended Use Of The Terra Core®

NOTE: The Terra Core® Sampler is a single use device. It cannot be cleaned and/or reused.

Step 1

Have ready a 40ml glass VOA vial containing the appropriate preservative. With the plunger seated in the handle, push the Terra Core® into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 or 10 grams of soil.

Step 2

Wipe all soil or debris from the outside of the Terra Core® sampler. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.

Step 3

Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the 40ml VOA vial containing the appropriate preservative and extrude the sample by pushing the plunger down. Quickly place the lid back on the 40ml VOA vial. **Note:** When capping the 40ml VOA vial, be sure to remove any soil or debris from the top and/or threads of the vial.

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1.0 Objectives and Rationale

The following sections summarize the field procedures for groundwater sampling to ensure quality control and to secure fully representative samples. Preservation of samples is required to retain integrity. The most common preservation techniques include pH adjustment and temperature control. Field personnel will use U.S. EPA-recommended container types and adhere to U.S. EPA recommended preservation techniques and holding times for the parameters of concern.

The procedures presented are designed to ensure the following:

- All sample and field measurements are consistent with project objectives.
- Samples are identified, preserved, and transported in such a manner as to ensure the integrity and validity of the samples.
- Field measurements are collected in a manner to allow for comparison between existing and newly collected data so as to provide an adequate data base for achieving the objectives and ensuring quality.

1.2 Quality Assurance Procedures

The sampler will always wear gloves while measuring water levels, purging wells, sampling wells, and decontaminating equipment. Gloves will be changed after each sample to prevent cross contamination. Pre-washed and pre-preserved sample containers will be provided by the laboratory chosen by Blackstone and will be prepared in accordance with U.S. EPA quality control procedures.

When collecting a sample for a particular analysis (e.g., volatile organic compounds) requiring multiple containers, bottles will be filled from the same bailer or sample hose, if possible. If an additional sample volume is required to fill the bottles, it will be retrieved in the same manner for consistency. Agitation will be avoided, particularly when volatile organic analyses will be conducted. Bottles will be filled in the order of most volatile to least volatile analysis type (VOCs to SVOCs to inorganics). Samples for volatile organic analysis will be collected in glass vials treated with hydrochloric acid as a preservative, allowing no headspace between the liquid and the lid of the vial. This will be accomplished by filling the bottle such that a meniscus forms over the lip, and then fitting the cap securely. Headspace will be checked by inverting the bottle and tapping the lid to see if air bubbles are visible in the bottle. If an air bubble appears, the procedure is repeated. If air bubbles are still present following resampling, an un-preserved vial will be collected, allowing no headspace using the same methodology as is used for filling a preserved vial. The un-preserved vial will be submitted to the laboratory, and the lack of preservative will be indicated on the chain of custody. A note indicating the shortened hold time will also be included on the chain of custody. Groundwater samples will be filled directly from the sampling device into the various bottles, except samples to be analyzed for dissolved metals, which will pass through an appropriate dedicated filter.

2.0 Groundwater Sampling Procedures

2.1 Sampling Procedure for Temporary (Drop Screen) Monitoring Wells

During Environmental Site Assessments, groundwater is sampled via temporary wells.

- A stainless-steel drop screen is placed at the interval of interest.
- A peristaltic pump using dedicated tubing is deployed for purging each temporary well.
- Once turbidity has decreased to ~100 NTU, samples are collected via a peristaltic pump.

2.2 Sampling Procedure for Permanent Monitoring Wells

2.2.1 Measurement of Potentiometric Levels

Static water level measurements will be recorded as feet below the measure point elevation (usually from the north side top of casing) to the nearest 0.01 foot. This is measured with a battery-operated water level indicator with tone or light that indicates when the probe has reached water in the well.

- Rinse probe and tape with deionized water before use at each well.
- Turn meter on and test battery.
- Lower probe slowly into well casing.
- When tone sounds and/or light flashes, raise and lower probe slightly to verify level.
- Measure water level depth to nearest 0.01-foot from the north side of the top of casing or marked point on the casing.
- Turn meter off and measure well depth, if required, making sure that the tape isn't sagging.
- Record the date, time, water level, well depth, and point of measurement on casing (if other than top of casing).

2.2.2 Well Purging /Stabilization

The water standing in the well may not be representative of the formation groundwater quality. Therefore, if the well will be sampled using a technology other than low-flow technology, it is necessary to purge the well prior to sampling to achieve a representative sample. Before purging the well, the static water level must be measured using a water level indicator. After the static water level has been measured and recorded, a pump or bailer will be lowered down the well and the water will be removed.

For a well to be properly purged, the well must have a minimum of three (3) saturated well casing volumes evacuated or the well must be evacuated until dry. If the well is bailed or pumped until dry, it must be allowed to recover and then the sample will be collected from the well. When utilizing low-flow sampling techniques, traditional well purging is not required. Low-flow methods specifically target the screened interval and pull water directly from the formation. This technique reduces disturbance to the water column which decreases turbidity and also places less stress on the surrounding formation.

A peristaltic pump or a bladder pump is lowered to the middle of the screened interval. Water is purged at a rate of 500 mL/minute or less and stabilization parameters are recorded every 3 to 5 minutes. Stabilization parameters include:

- water level (±10%),
- pH (± 0.2 NTU),
- specific conductivity (±10%),
- temperature (±0.5º C),
- dissolved oxygen (± 0.2 mg/L),
- turbidity (±10% or <5 NTU), and
- ORP (±10%).

Stabilization is achieved once three consecutive readings are within the stabilization range for all parameters. Sampling using low-flow methods is the preferred technique; however, bailers may also be used. Situations that would require the use of a bailer include pump malfunction or the inability to use a low-flow pump. For example, if a well is too deep to employ a peristaltic pump, and there is not enough water in the well to reach the inlet of a deployable bladder pump, a bailer or a passive sampling method such as a diffusion sampler would be used.

2.2.3 Sampling procedure

- Calibrate all field instruments at the start of each field day per equipment specific SOP. Record information on the appropriate calibration form.
- Open the monitoring well and record condition.
- Allow for the well to come to equilibrium after it has been opened and then measure static water level.
- If a bailer is being utilized to collect the groundwater sample, calculate the volume of three well casings and remove this volume while trying to minimize agitation within the well.

The next three steps apply to sample collection via low-flow pump only and should be ignored when a bailer is used.

- Prepare the sampling device for use.
- Connect the YSI 556 or similar flow cell device in line with the pump, when applicable.
- Begin pumping or purging the well while measuring stabilization parameters as indicated above.
- When stabilization is achieved, fill bottles from most volatile to least volatile (If taking splits / dupes / MS/MSDs, fill the bottles for each analyte at the same time, rather than filling by bottle set).
- Decontaminate the water level meter and any non-dedicated sampling equipment following the decontamination SOP.
- Secure the monitoring well.

3.0 REPORT PROCEDURE

Following field sampling, employee will prepare an inspection report that will include the following components:

1. All recorded sampling documentation
2. The address of the building
3. The date of the inspection
4. A summary of all lead-bearing substances in the building and control or abatement options for each hazard
5. Testing protocol used (e.g. IAC Chap. 70)
6. The instrument manufacturer, model, serial number, and all other applicable information pertaining to the device being used
7. The inspector and all documentation of licensing/certification
8. The illustration of the identification scheme

APPENDIX G

EPA METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed below for soil and sediment samples. Some common elements are not listed in this method because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). These light elements are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed below are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF. The following RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-0
Barium (Ba)	7440-39-3
Cadmium (Cd)	7440-43-9
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Lead (Pb)	7439-92-1
Mercury (Hg)	7439-97-6
Nickel (Ni)	7440-02-0
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4
Thallium (Tl)	7440-28-0
Tin (Sn)	7440-31-5

Analytes	CAS Registry No.
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

In addition, the following non-RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Calcium (Ca)	7440-70-2
Iron (Fe)	7439-89-6
Manganese (Mn)	7439-96-5
Molybdenum (Mo)	7439-93-7
Potassium (K)	7440-09-7
Rubidium (Rb)	7440-17-7
Strontium (Sr)	7440-24-6
Thorium (Th)	7440-29-1
Titanium (Ti)	7440-32-6
Zirconium (Zr)	7440-67-7

1.2 This method is a screening method to be used with confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)). This method's main strength is that it is a rapid field screening procedure. The method's lower limits of detection are typically above the toxicity characteristic regulatory level for most RCRA analytes. However, when the obtainable values for precision, accuracy, and laboratory-established sensitivity of this method meet project-specific data quality objectives (DQOs), FPXRF is a fast, powerful, cost effective technology for site characterization.

1.3 The method sensitivity or lower limit of detection depends on several factors, including the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. Example lower limits of detection for analytes of interest in environmental applications are shown in Table 1. These limits apply to a clean spiked matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (100 -600 second) count times. These sensitivity values are given for guidance only and may not always be achievable, since they will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of performance-based sensitivity is presented in Sec. 9.6.

1.4 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced and trained in the use and operation of an XRF instrument. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use either sealed radioisotope sources or x-ray tubes to irradiate samples with x-rays. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This latter process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples. The three electron shells include the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α), beta (β), or gamma (γ) etc., which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than

the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.77 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments, specifically, in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF -- Field portable x-ray fluorescence.

3.2 MCA -- Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS -- Site-specific calibration standards.

3.4 FP -- Fundamental parameter.

3.5 ROI -- Region of interest.

3.6 SRM -- Standard reference material; a standard containing certified amounts of metals in soil or sediment.

3.7 eV -- Electron volt; a unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One, Chapter Three, and the manufacturer's instructions for other definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte’s concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup (i.e., against the cup window), the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_β line of element Z-1 with the K_α line of element Z. This is called the K_α/K_β interference. Because the K_α:K_β intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_α and K_β energies are 4.95 and 5.43 keV, respectively, and the Cr K_α energy is 5.41 keV. The Fe K_α and K_β energies are 6.40 and 7.06 keV, respectively, and the Co K_α energy is 6.92 keV. The difference between the V K_β and Cr K_α energies is 20 eV, and the difference between the Fe K_β and the Co K_α energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_α/lead (Pb) L_α and sulfur (S) K_α/Pb M_α. In the As/Pb case, Pb can be measured from the Pb L_β line, and As can be measured from either the As K_α or the As $K_ß$ line; in this way the interference can be corrected. If the As K_β line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_α line. If the As K_α line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in reporting of a "nondetect" or a "less than" value (e.g., <300 ppm) for As, regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator’s decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-

atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)).

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as Method 3050, or a total digestion procedure, such as Method 3052, is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project-specific data quality objectives (DQOs).

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method (see Table 8), the confirmatory method used was Method 3050, and the FPXRF data compared very well with regression correlation coefficients (r often exceeding 0.95, except for barium and chromium). The critical factor is that the digestion procedure and analytical reference method used should meet the DQOs of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Sec. 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10° F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The user is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

NOTE: No MSDS applies directly to the radiation-producing instrument because that is covered under the Nuclear Regulatory Commission (NRC) or applicable state regulations.

5.2 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals.

Licenses for radioactive materials are of two types, specifically: (1) a general license which is usually initiated by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) a specific license which is issued to named persons for the operation of radioactive instruments as required by local, state, or federal agencies. A copy of the radioactive material license (for specific licenses only) and leak tests should be present with the instrument at all times and available to local and national authorities upon request.

X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply, however, if the tube is properly positioned within the instrument, this is only a negligible risk. Any instrument (x-ray tube or radioisotope based) is capable of delivering an electric shock from the basic circuitry when the system is inappropriately opened.

5.3 Radiation monitoring equipment should be used with the handling and operation of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs or badges should be worn in the area of maximum exposure. The maximum permissible whole-body dose from occupational exposure is 5 Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for

use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

6.1 FPXRF spectrometer -- An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation sources -- FPXRF instruments use either a sealed radioisotope source or an x-ray tube to provide the excitation source. Many FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron Fe-55 (^{55}Fe), cadmium Cd-109 (^{109}Cd), americium Am-241 (^{241}Am), and curium Cm-244 (^{244}Cm). These sources may be contained in a probe along with a window and the detector; the probe may be connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. This is due to the ever increasing time required for the analysis rather than a decrease in instrument performance. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum necessary for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of

accelerating voltage is governed both by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material and by the instrument's ability to cool the x-ray tube. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample presentation device -- FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For FPXRF instruments operated in the intrusive mode, the probe may be rotated so that the window faces either upward or downward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors -- The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon Si(Li). The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The Si(Li) detector must be cooled to at least -90 °C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a Si(Li) detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 L. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2-270 eV; silicon pin diode-250 eV; Si(Li)–170 eV; and gas-filled, proportional counter-750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data processing units -- The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amplitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in ppm on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 3,000 to 5,000 sets of numerical analytical results. Most FPXRF instruments are menu-driven from software built into the

units or from PCs. Once the data–storage memory of an FPXRF unit is full or at any other time, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery and battery charger.

6.3 Polyethylene sample cups -- 31 to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film -- Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 µm thick.

6.5 Mortar and pestle -- Glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers -- Glass or plastic to store samples.

6.7 Sieves -- 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels -- For smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags -- Used for collection and homogenization of soil samples.

6.10 Drying oven -- Standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Pure element standards -- Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if designated for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.3 Site-specific calibration standards -- Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.3.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of 10 samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.3.2 Each sample should be oven-dried for 2 to 4 hr at a temperature of less than 150 °C. If mercury is to be analyzed, a separate sample portion should be dried at ambient temperature as heating may volatilize the mercury. When the sample is dry, all large, organic debris and nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be homogenized (see Sec. 7.3.3) and then a representative portion ground with a mortar and pestle or other mechanical means, prior to passing through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.3.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 g of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 g of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.4 Blank samples -- The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the established lower limit of detection. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.5 Standard reference materials -- Standard reference materials (SRMs) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. When these SRMs are unavailable, alternate standards may be used (e.g., NIST 2702).

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, "Inorganic Analytes."

9.0 QUALITY CONTROL

9.1 Follow the manufacturer’s instructions for the quality control procedures specific to use of the testing product. Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results.

9.2 Energy calibration check -- To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting,

which would indicate drift within the instrument. As discussed in Sec. 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (more than 10 °F).

9.2.1 The energy calibration check should be run at a frequency consistent with manufacturer's recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.2 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak. The intensity and channel number of the pure element as measured using the source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank samples -- Two types of blank samples should be analyzed for FPXRF analysis, specifically, instrument blanks and method blanks.

9.3.1 An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window. The instrument blank can be silicon dioxide, a polytetraflurorethylene (PTFE) block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the established lower limit of detection should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. If the method blank does not contain the target analyte at a level that interferes with the project-specific data quality objectives then the method blank would be considered acceptable. In the absence of project-specific data quality objectives, if the blank is less than the lowest level of detection or less than 10% of the lowest sample concentration for the analyte, whichever is greater, then the method blank would be considered acceptable. If the method blank cannot be considered acceptable, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration verification checks -- A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ±20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision measurements -- The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent. If both in situ and intrusive analytical techniques are used during the course of one day, it is recommended that separate precision calculations be performed for each analysis type.

The equation for calculating RSD is as follows:

$$RSD = (SD/Mean\ Concentration) \times 100$$

where:

RSD = Relative standard deviation for the precision measurement for the analyte
SD = Standard deviation of the concentration for the analyte
Mean concentration = Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the sensitivity, but decreases sample throughput.

9.6 The lower limits of detection should be established from actual measured performance based on spike recoveries in the matrix of concern or from acceptable method performance on a certified reference material of the appropriate matrix and within the appropriate calibration range for the application. This is considered the best estimate of the true method sensitivity as opposed to a statistical determination based on the standard deviation of

replicate analyses of a low-concentration sample. While the statistical approach demonstrates the potential data variability for a given sample matrix at one point in time, it does not represent what can be detected or most importantly the lowest concentration that can be calibrated. For this reason the sensitivity should be established as the lowest point of detection based on acceptable target analyte recovery in the desired sample matrix.

9.7 Confirmatory samples -- The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on project-specific data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument calibration -- Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments, namely: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental parameters calibration -- FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are necessary, although site-specific samples with confirmed and validated analytical results for all elements present could be used.

- Cost is reduced because fewer confirmatory laboratory results or calibration standards are necessary.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Sec. 7.3. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective energy FP calibration -- The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_s - C_k) / C_k) \times 100$$

where:

%D = Percent difference
C_k = Certified concentration of standard sample
C_s = Measured concentration of standard sample

10.2.2 BFP calibration -- BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended

count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical calibration -- An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Sec. 7.3; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is necessary. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are necessary to perform an adequate empirical calibration. The exact number of standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton normalization method -- The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline reading. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_{α} peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later during analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For in situ analysis, remove any large or nonrepresentative debris from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Also, the soil surface must be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide example performance data for this method, this modest amount of sample preparation was found to take less than 5 min per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for in situ analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on the desired method sensitivity. Due to the heterogeneous nature of the soil sample, in situ analysis can provide only “screening” type data.

11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 g or 250 cm^3, which is enough soil to fill an 8-ounce jar. However, the exact dimensions and sample depth should take into consideration the heterogeneous deposition of contaminants and will ultimately depend on the desired project-specific data quality objectives. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Sec. 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the time necessary for homogenization procedure using the fluorescein dye ranged from 3 to 5 min per sample. As demonstrated in Secs. 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, the direct analysis through the plastic bag is possible without the more labor intensive steps of drying, grinding, and sieving given in Secs. 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps should be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 g) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hr in the convection or toaster oven at a temperature not greater than 150 °C. Samples may also be air dried under ambient temperature conditions using a 10- to 20-g portion. Regardless of what drying mechanism is used, the drying process is considered complete when a constant sample weight can be obtained. Care should be taken to avoid sample cross-contamination and these measures can be evaluated by including an appropriate method blank sample along with any sample preparation process.

CAUTION: Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 min per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 µm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle, and sieves must be thoroughly cleaned so that any cross-contamination is below the established lower limit of detection of the procedure or DQOs of the analysis. If all recommended sample preparation steps are followed, there is a high probability the desired laboratory data quality may be obtained.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in ppm and can be downloaded to a personal computer, which can be used to provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation. See the manufacturer's instructions regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The sections to follow discuss three performance evaluation factors; namely, precision, accuracy, and comparability. The example data presented in Tables 4 through 8 were generated from results obtained from six FPXRF instruments (see Sec. 13.3). The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States. The soil samples contained several of the target analytes at concentrations ranging from "nondetect" to tens of thousands of mg/kg. These data are provided for guidance purposes only.

13.3 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI_2 detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode

detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.4 All example data presented in Tables 4 through 8 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.5 Precision measurements -- The example precision data are presented in Table 4. These data are provided for guidance purposes only. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from "nondetects" to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 4 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the lower limit of detection for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 4. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the lower limit of detection so that an RSD value calculated at 5 to 10 times this limit was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 5 shows these results. These data are provided for guidance purposes only. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the lower limit of detection for the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the in situ homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 5 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square,

measurements of different soil samples were actually taking place within the square. Table 5 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five instead of ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy measurements -- Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 6 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 6 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 6. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 6.

Table 7 provides a more detailed summary of accuracy data for one particular FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. These data are provided for guidance purposes only. Table 7 shows the certified value, measured value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability -- Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 8. Similar trends in the data were seen for all instruments. These data are provided for guidance purposes only.

Table 8 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. These data are provided for guidance purposes only. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--intrusive, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not

ground; and preparation 4–intrusive, with sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 8 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 8 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Sec. 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time necessary to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 min. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 min per sample. Lastly, when grinding and sieving is conducted, time has to be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 A. D. Hewitt, "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_α Peak Normalization Analysis," American Environmental Laboratory, pp 24-32, 1994.

13.8.2 S. Piorek and J. R. Pasmore, "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer," Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals, Las Vegas, Nevada, February 24-26, 1993, Vol 2, pp 1135-1151, 1993.

13.8.3 S. Shefsky, "Sample Handling Strategies for Accurate Lead-in-soil Measurements in the Field and Laboratory," *International Symposium of Field Screening Methods for Hazardous Waste and Toxic Chemicals*, Las Vegas, NV, January 29-31, 1997.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex, X-MET 920 User's Manual.

2. Spectrace Instruments, "Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction," 1994.

3. TN Spectrace, Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.

4. Unpublished SITE data, received from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method. A flow diagram of the procedure follows the tables.

TABLE 1

EXAMPLE INTERFERENCE FREE LOWER LIMITS OF DETECTION

Analyte	Chemical Abstract Series Number	Lower Limit of Detection in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: Refs. 1, 2, and 3
These data are provided for guidance purposes only.

TABLE 2

SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	432	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Refs. 1, 2, and 3

TABLE 3

SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Ref. 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4

EXAMPLE PRECISION VALUES

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the Lower Limit of Detection					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84[a]	NR	24.80[a]	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85[a]	NR	24.92[a]	20.92[a]	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69[a]	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32[a]	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

These data are provided for guidance purposes only.
Source: Ref. 4

[a] These values are biased high because the concentration of these analytes in the soil samples was near the lower limit of detection for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the established lower limit detection.

TABLE 5

EXAMPLES OF PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive-Undried and Unground	Intrusive-Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium[a]	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel[a]	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver[a]	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

These data are provided for guidance purposes only.
Source: Ref. 4
[a] These values may be biased high because the concentration of these analytes in the soil samples was near the lower limit of detection.
ND Not detected.
NR Not reported.

TABLE 6

EXAMPLE ACCURACY VALUES

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	--	--	--	--	--	--	--	--	--	--	--	--
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	--	--	--	--	9	18-848	168.2	262	--	--	--	--
Cd	2	99-129	114.3	NA	--	--	--	--	6	81-202	110.5	45.7	--	--	--	--
Cr	2	99-178	138.4	NA	--	--	--	--	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	6	26-187	108.6	52.9
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	--	--	--	--	--	--	--	--
Ni	3	99-122	109.8	12.0	--	--	--	--	--	--	--	--	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	--	--	--	--	--	--	--	--	7	86-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5

Source: Ref. 4. These data are provided for guidance purposes only.
n: Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.
SD: Standard deviation; NA: Not applicable; only two data points, therefore, a SD was not calculated.
%Rec.: Percent recovery.
-- No data.

TABLE 7

EXAMPLE ACCURACY FOR TN 9000[a]

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	--	--	--	--	--	--	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	--	--	--	--	--	--	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	--	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	--	--	--	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	--	--	--	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Ref. 4. These data are provided for guidance purposes only.
[a] All concentrations in milligrams per kilogram.
%Rec.: Percent recovery; ND: Not detected; NA: Not applicable.
-- No data.

TABLE 8

EXAMPLE REGRESSION PARAMETERS FOR COMPARABILITY[1]

	Arsenic				Barium				Copper			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	286	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

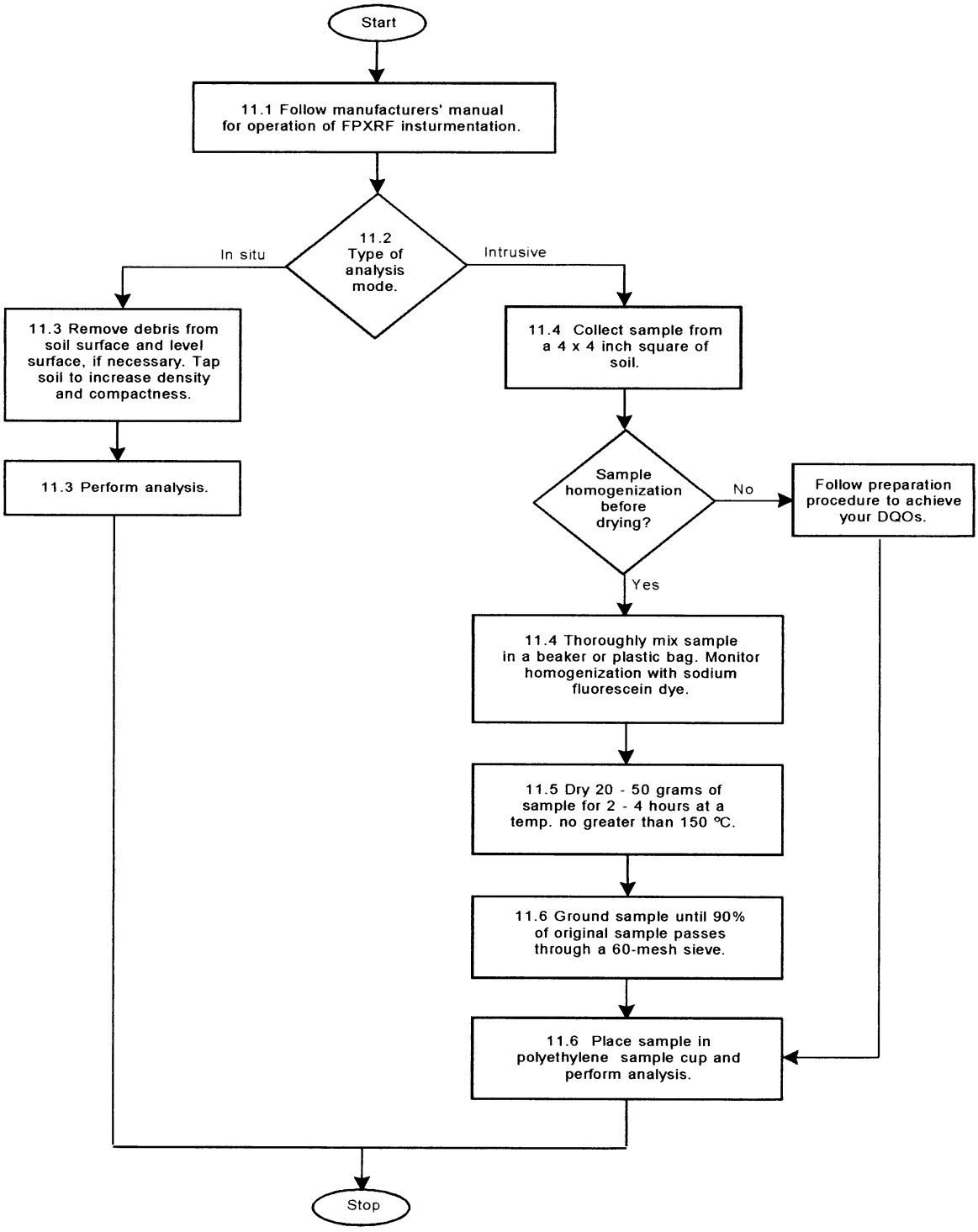
Source: Ref. 4. These data are provided for guidance purposes only.

[1] Log-transformed data

n: Number of data points; r^2: Coefficient of determination; Int.: Y-intercept

— No applicable data

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT



APPENDIX H

EPA §61.145 STANDARD FOR DEMOLITION AND RENOVATION – ASBESTOS

§ 61.145 Standard for demolition and renovation.

(a) *Applicability.* To determine which requirements of paragraphs (a), (b), and (c) of this section apply to the owner or operator of a demolition or renovation activity and prior to the commencement of the demolition or renovation, thoroughly inspect the affected facility or part of the facility where the demolition or renovation operation will occur for the presence of asbestos, including Category I and Category II nonfriable ACM. The requirements of paragraphs (b) and (c) of this section apply to each owner or operator of a demolition or renovation activity, including the removal of RACM as follows:

(1) In a facility being demolished, all the requirements of paragraphs (b) and (c) of this section apply, except as provided in paragraph (a)(3) of this section, if the combined amount of RACM is

(i) At least 80 linear meters (260 linear feet) on pipes or at least 15 square meters (160 square feet) on other facility components, or

(ii) At least 1 cubic meter (35 cubic feet) off facility components where the length or area could not be measured previously.

(2) In a facility being demolished, only the notification requirements of paragraphs (b)(1), (2), (3)(i) and (iv), and (4)(i) through (vii) and (4)(ix) and (xvi) of this section apply, if the combined amount of RACM is

(i) Less than 80 linear meters (260 linear feet) on pipes and less than 15 square meters (160 square feet) on other facility components, and

(ii) Less than one cubic meter (35 cubic feet) off facility components where the length or area could not be measured previously or there is no asbestos.

(3) If the facility is being demolished under an order of a State or local government agency, issued because the facility is structurally unsound and in danger of imminent collapse, only the requirements of paragraphs (b)(1), (b)(2), (b)(3)(iii), (b)(4) (except (b)(4)(viii)), (b)(5), and (c)(4) through (c)(9) of this section apply.

(4) In a facility being renovated, including any individual nonscheduled renovation operation, all the requirements of paragraphs (b) and (c) of this section apply if the combined amount of RACM to be stripped, removed, dislodged, cut, drilled, or similarly disturbed is

(i) At least 80 linear meters (260 linear feet) on pipes or at least 15 square meters (160 square feet) on other facility components, or

(ii) At least 1 cubic meter (35 cubic feet) off facility components where the length or area could not be measured previously.

(iii) To determine whether paragraph (a)(4) of this section applies to planned renovation operations involving individual nonscheduled operations, predict the combined additive amount of RACM to be removed or stripped during a calendar year of January 1 through December 31.

(iv) To determine whether paragraph (a)(4) of this section applies to emergency renovation operations, estimate the combined amount of RACM to be removed or stripped as a result of the sudden, unexpected event that necessitated the renovation.

(5) Owners or operators of demolition and renovation operations are exempt from the requirements of §§ 61.05(a), 61.07, and 61.09.

(b) *Notification requirements.* Each owner or operator of a demolition or renovation activity to which this section applies shall:

(1) Provide the Administrator with written notice of intention to demolish or renovate. Delivery of the notice by U.S. Postal Service, commercial delivery service, or hand delivery is acceptable.

(2) Update notice, as necessary, including when the amount of asbestos affected changes by at least 20 percent.

(3) Postmark or deliver the notice as follows:

(i) At least 10 working days before asbestos stripping or removal work or any other activity begins (such as site preparation that would break up, dislodge or similarly disturb asbestos material), if the operation is described in paragraphs (a) (1) and (4) (except (a)(4)(iii) and (a)(4)(iv)) of this section. If the operation is as described in paragraph (a)(2) of this section, notification

is required 10 working days before demolition begins.

(ii) At least 10 working days before the end of the calendar year preceding the year for which notice is being given for renovations described in paragraph (a)(4)(iii) of this section.

(iii) As early as possible before, but not later than, the following working day if the operation is a demolition ordered according to paragraph (a)(3) of this section or, if the operation is a renovation described in paragraph (a)(4)(iv) of this section.

(iv) For asbestos stripping or removal work in a demolition or renovation operation, described in paragraphs (a) (1) and (4) (except (a)(4)(iii) and (a)(4)(iv)) of this section, and for a demolition described in paragraph (a)(2) of this section, that will begin on a date other than the one contained in the original notice, notice of the new start date must be provided to the Administrator as follows:

(A) When the asbestos stripping or removal operation or demolition operation covered by this paragraph will begin after the date contained in the notice,

(*1*) Notify the Administrator of the new start date by telephone as soon as possible before the original start date, and

(*2*) Provide the Administrator with a written notice of the new start date as soon as possible before, and no later than, the original start date. Delivery of the updated notice by the U.S. Postal Service, commercial delivery service, or hand delivery is acceptable.

(B) When the asbestos stripping or removal operation or demolition operation covered by this paragraph will begin on a date earlier than the original start date,

(*1*) Provide the Administrator with a written notice of the new start date at least 10 working days before asbestos stripping or removal work begins.

(*2*) For demolitions covered by paragraph (a)(2) of this section, provide the Administrator written notice of a new start date at least 10 working days before commencement of demolition. Delivery of updated notice by U.S. Postal Service, commercial delivery service, or hand delivery is acceptable.

(C) In no event shall an operation covered by this paragraph begin on a date other than the date contained in the written notice of the new start date.

(4) Include the following in the notice:

(i) An indication of whether the notice is the original or a revised notification.

(ii) Name, address, and telephone number of both the facility owner and operator and the asbestos removal contractor owner or operator.

(iii) Type of operation: demolition or renovation.

(iv) Description of the facility or affected part of the facility including the size (square meters [square feet] and number of floors), age, and present and prior use of the facility.

(v) Procedure, including analytical methods, employed to detect the presence of RACM and Category I and Category II nonfriable ACM.

(vi) Estimate of the approximate amount of RACM to be removed from the facility in terms of length of pipe in linear meters (linear feet), surface area in square meters (square feet) on other facility components, or volume in cubic meters (cubic feet) if off the facility components. Also, estimate the approximate amount of Category I and Category II nonfriable ACM in the affected part of the facility that will not be removed before demolition.

(vii) Location and street address (including building number or name and floor or room number, if appropriate), city, county, and state, of the facility being demolished or renovated.

(viii) Scheduled starting and completion dates of asbestos removal work (or any other activity, such as site preparation that would break up, dislodge, or similarly disturb asbestos material) in a demolition or renovation; planned renovation operations involving individual nonscheduled operations shall only include the beginning and ending dates of the report period as described in paragraph (a)(4)(iii) of this section.

(ix) Scheduled starting and completion dates of demolition or renovation.

(x) Description of planned demolition or renovation work to be performed

and method(s) to be employed, including demolition or renovation techniques to be used and description of affected facility components.

(xi) Description of work practices and engineering controls to be used to comply with the requirements of this subpart, including asbestos removal and waste-handling emission control procedures.

(xii) Name and location of the waste disposal site where the asbestos-containing waste material will be deposited.

(xiii) A certification that at least one person trained as required by paragraph (c)(8) of this section will supervise the stripping and removal described by this notification. This requirement shall become effective 1 year after promulgation of this regulation.

(xiv) For facilities described in paragraph (a)(3) of this section, the name, title, and authority of the State or local government representative who has ordered the demolition, the date that the order was issued, and the date on which the demolition was ordered to begin. A copy of the order shall be attached to the notification.

(xv) For emergency renovations described in paragraph (a)(4)(iv) of this section, the date and hour that the emergency occurred, a description of the sudden, unexpected event, and an explanation of how the event caused an unsafe condition, or would cause equipment damage or an unreasonable financial burden.

(xvi) Description of procedures to be followed in the event that unexpected RACM is found or Category II nonfriable ACM becomes crumbled, pulverized, or reduced to powder.

(xvii) Name, address, and telephone number of the waste transporter.

(5) The information required in paragraph (b)(4) of this section must be reported using a form similiar to that shown in Figure 3.

(c) *Procedures for asbestos emission control.* Each owner or operator of a demolition or renovation activity to whom this paragraph applies, according to paragraph (a) of this section, shall comply with the following procedures:

(1) Remove all RACM from a facility being demolished or renovated before any activity begins that would break up, dislodge, or similarly disturb the material or preclude access to the material for subsequent removal. RACM need not be removed before demolition if:

(i) It is Category I nonfriable ACM that is not in poor condition and is not friable.

(ii) It is on a facility component that is encased in concrete or other similarly hard material and is adequately wet whenever exposed during demolition; or

(iii) It was not accessible for testing and was, therefore, not discovered until after demolition began and, as a result of the demolition, the material cannot be safely removed. If not removed for safety reasons, the exposed RACM and any asbestos-contaminated debris must be treated as asbestos-containing waste material and adequately wet at all times until disposed of.

(iv) They are Category II nonfriable ACM and the probability is low that the materials will become crumbled, pulverized, or reduced to powder during demolition.

(2) When a facility component that contains, is covered with, or is coated with RACM is being taken out of the facility as a unit or in sections:

(i) Adequately wet all RACM exposed during cutting or disjoining operations; and

(ii) Carefully lower each unit or section to the floor and to ground level, not dropping, throwing, sliding, or otherwise damaging or disturbing the RACM.

(3) When RACM is stripped from a facility component while it remains in place in the facility, adequately wet the RACM during the stripping operation.

(i) In renovation operations, wetting is not required if:

(A) The owner or operator has obtained prior written approval from the Administrator based on a written application that wetting to comply with this paragraph would unavoidably damage equipment or present a safety hazard; and

(B) The owner or operator uses of the following emission control methods:

(*1*) A local exhaust ventilation and collection system designed and operated to capture the particulate asbestos material produced by the stripping and removal of the asbestos materials. The system must exhibit no visible emissions to the outside air or be designed and operated in accordance with the requirements in § 61.152.

(*2*) A glove-bag system designed and operated to contain the particulate asbestos material produced by the stripping of the asbestos materials.

(*3*) Leak-tight wrapping to contain all RACM prior to dismantlement.

(ii) In renovation operations where wetting would result in equipment damage or a safety hazard, and the methods allowed in paragraph (c)(3)(i) of this section cannot be used, another method may be used after obtaining written approval from the Administrator based upon a determination that it is equivalent to wetting in controlling emissions or to the methods allowed in paragraph (c)(3)(i) of this section.

(iii) A copy of the Administrator's written approval shall be kept at the worksite and made available for inspection.

(4) After a facility component covered with, coated with, or containing RACM has been taken out of the facility as a unit or in sections pursuant to paragraph (c)(2) of this section, it shall be stripped or contained in leak-tight wrapping, except as described in paragraph (c)(5) of this section. If stripped, either:

(i) Adequately wet the RACM during stripping; or

(ii) Use a local exhaust ventilation and collection system designed and operated to capture the particulate asbestos material produced by the stripping. The system must exhibit no visible emissions to the outside air or be designed and operated in accordance with the requirements in § 61.152.

(5) For large facility components such as reactor vessels, large tanks, and steam generators, but not beams (which must be handled in accordance with paragraphs (c)(2), (3), and (4) of this section), the RACM is not required to be stripped if the following requirements are met:

(i) The component is removed, transported, stored, disposed of, or reused without disturbing or damaging the RACM.

(ii) The component is encased in a leak-tight wrapping.

(iii) The leak-tight wrapping is labeled according to § 61.149(d)(1)(i), (ii), and (iii) during all loading and unloading operations and during storage.

(6) For all RACM, including material that has been removed or stripped:

(i) Adequately wet the material and ensure that it remains wet until collected and contained or treated in preparation for disposal in accordance with § 61.150; and

(ii) Carefully lower the material to the ground and floor, not dropping, throwing, sliding, or otherwise damaging or disturbing the material.

(iii) Transport the material to the ground via leak-tight chutes or containers if it has been removed or stripped more than 50 feet above ground level and was not removed as units or in sections.

(iv) RACM contained in leak-tight wrapping that has been removed in accordance with paragraphs (c)(4) and (c)(3)(i)(B)(*3*) of this section need not be wetted.

(7) When the temperature at the point of wetting is below 0 °C (32 °F):

(i) The owner or operator need not comply with paragraph (c)(2)(i) and the wetting provisions of paragraph (c)(3) of this section.

(ii) The owner or operator shall remove facility components containing, coated with, or covered with RACM as units or in sections to the maximum extent possible.

(iii) During periods when wetting operations are suspended due to freezing temperatures, the owner or operator must record the temperature in the area containing the facility components at the beginning, middle, and end of each workday and keep daily temperature records available for inspection by the Administrator during normal business hours at the demolition or renovation site. The owner or operator shall retain the temperature records for at least 2 years.

(8) Effective 1 year after promulgation of this regulation, no RACM shall

be stripped, removed, or otherwise handled or disturbed at a facility regulated by this section unless at least one onsite representative, such as a foreman or management-level person or other authorized representative, trained in the provisions of this regulation and the means of complying with them, is present. Every 2 years, the trained onsite individual shall receive refresher training in the provisions of this regulation. The required training shall include as a minimum: applicability; notifications; material identification; control procedures for removals including, at least, wetting, local exhaust ventilation, negative pressure enclosures, glove-bag procedures, and High Efficiency Particulate Air (HEPA) filters; waste disposal work practices; reporting and recordkeeping; and asbestos hazards and worker protection. Evidence that the required training has been completed shall be posted and made available for inspection by the Administrator at the demolition or renovation site.

(9) For facilities described in paragraph (a)(3) of this section, adequately wet the portion of the facility that contains RACM during the wrecking operation.

(10) If a facility is demolished by intentional burning, all RACM including Category I and Category II nonfriable ACM must be removed in accordance with the NESHAP before burning.

NOTIFICATION OF DEMOLITION AND RENOVATION

Operator Project #	Postmark	Date Received	Notification #

I. TYPE OF NOTIFICATION (O=Original R=Revised C=Cancelled):

II. FACILITY INFORMATION (Identify owner, removal contractor, and other operator)

OWNER NAME:		
Address:		
City:	State:	Zip:
Contact:		Tel:
REMOVAL CONTRACTOR:		
Address:		
City:	State:	Zip:
Contact:		Tel:
OTHER OPERATOR:		
Address:		
City:	State:	Zip:
Contact:		Tel:

III. TYPE OF OPERATION (D=Demo O=Ordered Demo R=Renovation E=Emer.Renovation):

IV. IS ASBESTOS PRESENT? (Yes/No)

V. FACILITY DESCRIPTION (Include building name, number and floor or room number)

Bldg Name:		
Address:		
City:	State:	County:
Site Location:		
Building Size:	# of Floors:	Age in Years:
Present Use:	Prior Use:	

VI. PROCEDURE, INCLUDING ANALYTICAL METHOD, IF APPROPRIATE, USED TO DETECT THE PRESENCE OF ASBESTOS MATERIAL:

VII. APPROXIMATE AMOUNT OF ASBESTOS, INCLUDING: 1. Regulated ACM to be removed 2. Category I ACM Not Removed 3. Category II ACM Not Removed	RACM To Be Removed	Nonfriable Asbestos Material Not To Be Removed		Indicate Unit of Measurement Below	
		Cat I	Cat II	UNIT	
Pipes				LnFt:	Ln m:
Surface Area				SqFt:	Sq m:
Vol RACM Off Facility Component				CuFt:	Cu m:

VIII. SCHEDULED DATES ASBESTOS REMOVAL (MM/DD/YY) Start: Complete:

IX. SCHEDULED DATES DEMO/RENOVATION (MM/DD/YY) Start: Complete:

Continued on page two

Figure 3. Notification of Demolition and Renovation

NOTIFICATION OF DEMOLITION AND RENOVATION (continued)

X. DESCRIPTION OF PLANNED DEMOLITION OR RENOVATION WORK, AND METHOD(S) TO BE USED:		
XI. DESCRIPTION OF WORK PRACTICES AND ENGINEERING CONTROLS TO BE USED TO PREVENT EMISSIONS OF ASBESTOS AT THE DEMOLITION AND RENOVATION SITE:		
XII. WASTE TRANSPORTER #1		
Name:		
Address		
City:	State:	Zip:
Contact Person:		Telephone:
WASTE TRANSPORTER #2		
Name:		
Address:		
City:	State:	Zip:
Contact Person:		Telephone:
XIII. WASTE DISPOSAL SITE		
Name:		
Location:		
City:	State:	Zip:
Telephone:		
XIV. IF DEMOLITION ORDERED BY A GOVERNMENT AGENCY, PLEASE IDENTIFY THE AGENCY BELOW:		
Name:	Title:	
Authority:		
Date of Order (MM/DD/YY):	Date Ordered to Begin (MM/DD/YY):	
XV. FOR EMERGENCY RENOVATIONS		
Date and Hour of Emergency (MM/DD/YY):		
Description of the Sudden, Unexpected Event:		
Explanation of how the event caused unsafe conditions or would cause equipment damage or an unreasonable financial burden:		
XVI. DESCRIPTION OF PROCEDURES TO BE FOLLOWED IN THE EVENT THAT UNEXPECTED ASBESTOS IS FOUND OR PREVIOUSLY NONFRIABLE ASBESTOS MATERIAL BECOMES CRUMBLED, PULVERIZED, OR REDUCED TO POWDER.		
XVI. I CERTIFY THAT AN INDIVIDUAL TRAINED IN THE PROVISIONS OF THIS REGULATION (40 CFR PART 61, SUBPART M) WILL BE ON-SITE DURING THE DEMOLITION OR RENOVATION AND EVIDENC THAT THE REQUIRED TRAINING HAS BEEN ACCOMPLISHED BY THIS PERSON WILL BE AVAILABLE FOR INSPECTION DURING NORMAL BUSINESS HOURS. (Required 1 year after promulgation)		
(Signature of Owner/Operator)		(Date)
XVII. I CERTIFY THAT THE ABOVE INFORMATION IS CORRECT.		
(Signature of Owner/Operator)		(Date)

Figure 3. Notification of Demolition and Renovation

[55 FR 48419, Nov. 20, 1990; 56 FR 1669, Jan. 16, 1991]

§61.146 Standard for spraying.

The owner or operator of an operation in which asbestos-containing materials are spray applied shall comply with the following requirements:

(a) For spray-on application on buildings, structures, pipes, and conduits, do not use material containing more than 1 percent asbestos as determined using the method specified in appendix E, subpart E, 40 CFR part 763, section 1,