

**Guidance
for Field
Characterization
Technology
Verifications**

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1.0 INTRODUCTION

It is through characterization that the type, quantities, and locations of contaminants present at a site are adequately determined. Characterization can be done at various stages of environmental operations from initial screening (phase I), site characterization (phase II), monitoring effectiveness of remediation, and ultimately for final survey. Along these stages characterization data is needed for the optimal selection of remedial actions, health and safety, risk management and ultimately for cost-effective remediation and restoration. Currently, billions of dollars are being spent on characterizing radiological and hazardous contaminants in man-made structures, and terrestrial and aquatic environments. Most of these funds are spent on the collection and laboratory analysis of samples. Current advances in technology have produced a number of field instruments capable of generating accurate data comparable to laboratory instruments, resulting in cost savings and faster characterization of sites. Unfortunately, for the most part these field instruments have not been accepted by regulators as an acceptable replacement for standard fixed laboratory instruments.

It would be of great benefit to acquire regulatory acceptance for improved real-time characterization technologies, including those for characterizing facility surfaces, bulk material, soil, sediment, surface water, groundwater, ambient air, soil gas, and generated waste. Benefits of regulatory acceptance for these field technologies include:

- a lower cost and quicker alternative to sample collection and lab analysis
- a wider coverage compared to sample collection and analysis, resulting in a higher reliability in determining areas of contamination
- improved reliability in waste categorization and likely cost savings (e.g., mixed LLW versus TRU)
- near real-time data to the field for improved efficiency

This document is intended to provide guidance for conducting field characterization technology verifications that will generate high quality data that can be used to provide the documentation necessary to receive regulatory approval for that field technology.

The assistance and guidance of the Interstate Technology Regulatory Cooperation (ITRC) in developing this document has been appreciated and will help in improving the acceptance of this guidance across multiple states/regions. The ITRC is a state-led, national coalition of personnel from regulatory and technology programs and has as its main goal to reduce barriers and speed interstate deployment of better, more cost-effective, innovative environmental technologies. Recognition of this guidance document by state agencies can help create greater consistency in the technical requirements among states and reduce the fragmentation of markets for technologies caused by differing state requirements. Use of this guidance document, by providing the framework for a consistent verification approach, will enable state agencies to review verification plans and reports quickly and thoroughly.

2.0 DESCRIPTION OF VERIFICATION PROCESS

After a technology need is identified, a search is performed to find applicable and available technologies. The technology search can include a review of published technology demonstrations as well as information from the technology vendors.

A characterization technology is a good candidate for the verification process if it meets the following criteria:

- Able to meet user performance requirements
- Applicable to a number of different sites
- Cost-competitive with current methods
- Performs better than current methods in areas such as data quality, sample preparation, or time required to receive analytical results
- Uses techniques that are easier or safer than current methods
- Is commercially available and ready to deploy to the field or mobile laboratory

Once the selection of the technology is complete, the verification process consists of three main steps: (1) planning for verification (2) implementing the verification test, and (3) preparing a report with the test data and documentation and requesting regulatory approval of the field technology.

2.1 Verification Planning

The following issues need to be addressed as part of the verification planning process:

- Identification of sites that will provide the appropriate physical or chemical environment, including contaminated media; it is important that the site provides enough challenges to make the test results widely applicable but not so difficult a challenge that the risks of failure become significant
- Determination of logistical and support requirements (for example, field equipment, power and water sources, mobile laboratory, communications network, specific personnel skills and training)
- Planning for sampling and analytical support
- Preparation of a technology-specific verification test plan that addresses the experimental design, sampling design, QA/QC, health and safety considerations,

scheduling of field and laboratory operations, resource planning including data analysis procedures, and reporting requirements

- Implementation of the test plan

Each of these issues is addressed in further detail in this document.

2.2 Verification Implementation

The verification is implemented per the test plan developed during the planning process. It is key to actively solicit regulatory input during the planning and implementation of the technology verification process since the ultimate goal is to receive regulatory acceptance of the field technology. Adherence to this draft guidance should facilitate rapid acceptance.

2.3 Report Preparation and Request for Regulatory Acceptance

The data generated during the verification test are used to generate a report documenting the capabilities, limitations, and field applications of the technology in order to support acceptance of the field technology by the regulators and other stakeholders. This acceptance is much more likely if the regulators have been involved in the entire verification process, rather than simply presenting the regulators with a completed report. In this way, their questions and concerns can be addressed as a part of the process. One example of a sampling and analysis process to gain regulatory acceptance of a field characterization technology by showing comparability to an accepted reference method is shown in Attachment 1.

The goal for this guidance document is to smooth the process towards regulatory acceptance of field characterization technologies. Current practices in seeking regulatory acceptance for a field characterization technology too often involve repeating the verification process over and over in different states/regions without using earlier experiences (perhaps performed by other organizations) and without a sound statistical basis for the test plan.

3.0 DESCRIPTION OF VERIFICATION TEST

A verification test consists of applying a robust characterization technology to a realistic field application. The verification test should be in accordance with a detailed test plan and quality assurance (QA) plan. The results are compared to a reference measurement method or to an accepted standard to verify the performance of the technology (Reference 2).

3.1 Technology Tested

The field technology used in a verification test should be a readily available commercial product and should be standard and representative of the vendor's normal production of the technology. Use of a specialized or modified unit could create insurmountable obstacles to receiving regulatory approval for the technology.

3.2 Field Test Site

Characterization technologies that are intended for use in the field should be tested with realistic sample matrices and under typical field conditions. Factors that may influence test site selection include possible interferences or matrix effects and availability of an on-site reference method or calibration system.

The characteristics of any test site should include the following (Reference 2):

- The site must be representative of sites where the technology would actually be used;
- The sample matrices available at the site must be representative of the matrices to which the technology would be applied;
- The site should be known to contain target analytes from non-detected to high concentrations (Reference 9)
- If multiple technologies are being tested, the site should not offer a competitive advantage to any one technology;
- The site must be available for the period of time required for verification testing;
- The site must allow a verification test to be carried out in a cost-effective and timely manner;
- The site must not require extensive modification prior to conducting a verification test;
- The site must have sufficient infrastructure (access, space, power, support facilities, etc.) for operation of the technology to be tested.

3.3 Basis of Comparison

A basis of comparison is provided via a standardized reference method of measuring the same target analytes, granting an ability to compare the tested technology results with those from the reference method. This comparison allows the performance of the tested technology to be quantified. However, an accepted standardized method may or may not exist for a particular target analyte. Three situations that may be encountered in determining the appropriate basis of comparison for a verification test include (Reference 2):

- An EPA method exists;
- A generally accepted reference method established by another organization exists;
- No generally accepted method exists, but calibration standards or reference materials can serve as a basis for performance testing.

Selection of the basis of comparison should be based on a review of published methods as well as on input from vendors, stakeholders, regulatory staff, and technology users. The standard basis for comparison should be clearly identified in the test plan and QA plan.

3.3.1 EPA Method

An EPA method should be used as the basis of comparison if one exists that is applicable to the verification test. Examples of EPA methods include the following (Reference 2):

- Standards of Performance for Stationary Sources Appendix A - Test Methods (40 CFR Part 60)
- Ambient Air Monitoring Reference and Equivalent Methods (40 CFR Part 53)
- Methods for the Determination of Metals in Environmental Samples (EPA/600/R-94/111)
- Methods for the Determination of Organic Compounds in Drinking Water (EPA/600/4-88-039)
- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air (EPA/600/4-89-017)

An EPA method used in a verification test must be performed according to its published procedures, including those for calibration and other quality control activities. However, consideration will also be given to the current acceptance of the EPA method; an EPA method that has fallen into disuse or been generally displaced by a non-EPA method may not be the most credible basis for comparison (Reference 2). [\(Give example\)](#)

3.3.2 Other Generally Accepted Method

If no EPA method exists for the verification test, another well accepted reference method established by another reputable organization should be identified. For example, *Standard Methods for the Examination of Water and Wastewater* jointly developed by the American Public Health Association (APHA), the American Water Works Association (AWWA), and the Water Environment Federation, might be considered for reference methods (Reference 2).

Other reference methods can also be used to augment an existing EPA method if the characteristics of the EPA method (e.g., time response) limits the data comparisons that can be made. These results should also be compared to the EPA method results in order to quantify the performance of the other reference method. In other words, all verification technology test results and any additional reference method results are ultimately referenced back to the EPA method results.

In selecting an EPA method or other reference method as the basis of comparison, the following requirements should be met (Reference 2):

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- The method must have sufficient sensitivity, linear range, precision, specificity, etc., to provide a valid basis for comparison to the commercial technologies;
- The method is already in place or can readily be implemented, if necessary;
- The results from the EPA or reference method must be available promptly enough to facilitate comparisons with the commercial technologies;
- The costs of using the EPA or reference method must be acceptable within the context of the verification test.

3.3.3 No Generally Accepted Method

In some cases, neither an EPA method nor a well accepted reference method from a reputable organization will exist for a particular technology. The technology performance could still be quantified by comparison with standards or reference materials from another organization (e.g., National Institute of Standards and Technology).

In cases where a standard or reference sample must be prepared, the non-certified standard should be compared to a certified standard. For example, preparation of standard water samples containing diverse target analytes could be acceptable, provided that some of the target analytes can be verified by comparison to a certified standard (Reference 2).

4.0 ROLES AND RESPONSIBILITIES

Functional responsibilities of those involved in verification testing should be determined and documented. An organizational chart would be helpful to show the division of responsibilities. A clear description of the roles and responsibilities of each participant is necessary. Participants may include the technology vendor, government agency, government agency contractors, and local, state, and Federal regulators. Specific roles may include project management, technical lead, statistician, Safety and Health lead, Quality Assurance lead.

Responsibilities of the organization performing the verification would include (Reference 2):

- Overall organization, budgeting, and coordination of the verification test
- Assuring objectivity in all planning, communication, data analysis, and reporting
- Preparation of the draft and final verification test plan and QA plan
- Definition of the characteristics to be evaluated in the verification test
- Coordination of review of the draft test/QA
- Selection of a test site, and completion of arrangements to use the site
- Communication with the test site regarding the schedule and required support
- Performance of the verification test
- Data analysis
- Preparation of the draft and final verification report

- Coordination of the review of the draft verification report

5.0 VERIFICATION TEST PLAN

The verification test plan should provide all pertinent details for how the verification test will be performed. The document should focus on the specific technology and a specified standard method. Allowing for variations depending on the specific technology being tested, the verification test plan may include the following components (Reference 2):

- Front Material (title page, table of contents, lists of figures and tables, executive summary, abbreviations and acronyms)
- Introduction (nature and purpose of the verification test)
- Roles and Responsibilities (identification of participants and a description of their roles and responsibilities)
- Technology Description (description of the technology, including operating principles and requirements)
- Site Description (location of the site, site history, emissions information, quantitative characteristics such as flows, temperatures, nearby source impacts, etc.)
- Basis of Comparison (description of methods or standards that serve as the basis for the verification)
- Study Design (specify types and numbers of samples to be analyzed or data to be collected, and comparisons or statistical analyses to be made with the data)
- Field Procedures (practical operations to be carried out to obtain needed samples or data, including locations, schedules, collection media, data recording, etc.)
- Quality Assurance (quality control procedures and quality assurance oversight to be implemented in the verification test, including types and number of calibrations and standards, sample custody, data acceptance criteria (e.g., completeness, performance of reference method))
- Emergency Planning and Worker Health and Safety
- Data Reduction and Reporting (data management and organization, confidentiality and separation of data from different technologies, report preparation and review).

6.0 STUDY DESIGN

In order for the final data results to meet the objectives of the verification test, sufficient emphasis needs to be given to the study design. The study design establishes the data to be collected and the data comparisons to be used. The *Guidance for the Data Quality Objectives Process* (see Reference 10) should be used to develop the study design as it will aid in

establishing the type, number, and manner of data to be collected. The data quality objectives (DQO) process has seven steps, as summarized below (Reference 2):

6.1 State the Problem

The first step in the DQO process is to clearly state what problem or question the data are intended to address (e.g., to assess the accuracy, precision, detection limit, etc., of a field characterization technology). Reviewing existing information, such as the results of previous technology demonstrations, can provide assistance in fully understanding the problem.

6.2 Identify the Decision

The next step is to identify the decision by developing a quantitative statistical statement of what decision point is to be reached. Examples include a definition of a range of linearity to be verified, or a degree of uncertainty that is tolerable in assessing accuracy or precision. This step thus provides a quantitative goal for data collection.

6.3 Identify the Decision Inputs

The next step is to identify the decision inputs. This step identifies the data needed to meet the decision criteria established in the previous step, including the types of samples or measurements needed to verify the technology, the approximate number of samples needed, and the concentration ranges. Statistical considerations leading to uncertainty estimates and decision errors are generally incorporated in later steps but may be considered in this step.

6.4 Define the Study Boundaries

The next step is to define the study boundaries, establishing the range of test conditions, sampling locations, sample types or matrices, or sampling environments appropriate for the verification test. The study boundaries can also refer to sampling schedules and the capability of the standard method to provide data for comparison. For example, representativeness may have different meanings for air, water, and soil technologies, as well as different meanings for different technologies within those matrix areas. So while geographic and meteorological factors may determine representativeness in ambient air sampling, target analyte levels, hydrological factors, or matrix composition may determine representativeness for water sampling and analysis.

6.5 Develop a Decision Rule

The next step is to develop a decision rule. Select statistic tests that will be used to verify the technology performance. For example, linearity may be assessed by using a mathematical test or accuracy relative to a standard method may be assessed by comparison of mean values at some standard or typical concentration, or by the slope of a regression of data at multiple points. Decision rules should be selected from those commonly accepted and readily understood, so that the meaning of the verification results is clear.

6.6 Specify Tolerable Limits on Decision Errors

The next step is to specify tolerable limits on decision errors. Apply acceptable uncertainty limits to the decisions made in previous steps and reevaluate the types, numbers, ranges, etc. of the data to be obtained. If the sampling estimates made previously cannot provide acceptable error limits for the comparisons to be made, then more or different samples, or added QC efforts, may be needed. The DQO process is iterative so previous steps need to be revised until uncertainty limits are acceptable.

6.7 Optimize the Design

The final DQO step is to optimize the design. Consider the data collection guidance from the previous steps with respect to the real-world limitations of the verification test. Is the cost of sampling and analysis within budget constraints? Is it feasible to obtain all the data suggested? Is sufficient time available to perform the verification test? Does the test site have specific restrictions to be considered? Revisit the previous steps as necessary to find compromises among the various factors.

The result of this DQO process is a study design that specifies what data collection activities are to be done, how many samples of what kinds are to be collected, and what comparisons are to be made with the data.

6.8 Conversion to Field Procedures

The study design developed through the DQO process can then be used to develop the procedures to be used in implementing the verification test. Take the study design results showing what is to be done and spell out how it will be done: specific characterization or sample collection procedures and schedules, instructions for collection of other data, and procedures for sample handling and analysis.

7.0 QUALITY ASSURANCE AND QUALITY CONTROL (QA/QC)

A well developed QA Plan outlines the steps necessary to ensure that data resulting from the verification test is of known quality and that a sufficient number of critical measurements are taken. Careful adherence to the QA plan will ensure that data generated from the verification test will meet the desired performance objectives and will provide sound analytical results (Reference 3).

Important elements of a QA Plan are discussed below (Reference 4).

7.1 Field Operations

7.1.1 Site Training

Describe the training that will be required and/or provided prior to the verification testing. This training may include field site orientation, safety information, emergency procedures, and logistics of the verification test.

7.1.2 Documentation

A flawlessly executed verification test will not provide the basis needed to receive regulatory approval for the technology without proper documentation. Field activities should be thoroughly documented. Field documentation may include field logbooks, photographs, data sheets, and chain-of-custody forms. Field notes should be kept in a bound logbook with each page sequentially numbered and completed pages should be signed and dated by the person making the entries. Any deviations from the verification test plan should be thoroughly documented in the field logbook including reasons for the deviations and subsequent results. Operating conditions during the verification testing should be monitored and documented. For example, if humidity can affect the measurements being collected, humidity readings should be taken and documented during the verification test. Documentation should also address any unforeseen events and document the measures taken in response to these events.

7.2 Performance and System Audits

The following types of audits should be considered to ensure the quality of the verification process and the resulting data:

- On-site surveillance of the technical systems during the verification test
- An audit of the analytical laboratory during the analysis of the samples to confirm that the laboratory is following its procedures
- An audit during the verification testing to observe the operation of the field technology, such as observing the operations, photo-documenting the test site activities, surveying calibration procedures, and reviewing sample data

Any findings from these audits or other monitoring activities should be documented along with the corrective actions taken to resolve the findings. Any occurrence that causes discrepancies from the verification test plan should also be noted.

7.3 Data Validation

Validation determines the quality of the results relative to the end use of the data. Many of the characterization technologies will be compared to results generated by a reference laboratory. The findings of the data validation should be documented. As appropriate, describe instances

of failure to meet quality objectives and the potential impact on data quality. Data validation includes a review of the following (Reference 4):

7.3.1 Completeness of Laboratory Records

A review of the completeness of laboratory records verifies that all of the samples that were sent to the laboratory were analyzed and that all of the applicable records and relevant results are included in the data package.

7.3.2 Holding Times

Sample holding times will vary depending on the analyte and matrix being analyzed. Therefore, holding times should be reviewed against the specifications in the verification plan or in the contractual arrangement with the laboratory.

7.3.3 Correctness of Data

Laboratory data that contains transcription errors, calculation errors, or interpretation errors should be corrected as necessary to avoid biasing the assessment of the technology's performance. Any changes must be justified and documented in the validation records.

7.3.4 Correlation Between Samples within a Concentration Set

Questionable sample analytical results may include the following:

- Data result was reported with a flag by the reference laboratory
- Reported result does not correspond to the known concentration of the sample (e.g., spiked sample)
- Replicate results from a homogenous sample set do not correspond

In these cases, criteria may be established to determine if data is suspect. For example, data sets could be considered suspect if the standard deviation of the replicate results was greater than 30 ppm and the percent relative standard deviation was greater than 50%. These criteria would indicate imprecision in the sample replicate set. These data should be flagged so as not to bias the assessment of the technology's performance.

Precision and accuracy evaluations may be made with and without these suspect values to represent the best and worst case scenarios. If both the reference laboratory and the vendor(s) report erratic results and it is suspected that the erratic results are due to a sampling error, the data may be discarded.

7.3.5 Evaluation of QC Results

The purpose of QC samples is to indicate whether or not the batch of samples were analyzed properly. Quality control samples include laboratory control samples, matrix spikes, matrix spike duplicates, surrogate recoveries, field blanks, trip blanks, field duplicates, etc., and should be analyzed with each batch of samples, as appropriate. The results from QC samples should be compared to the acceptable QC results specified in either the reference laboratory's procedures or in the verification plan.

7.3.6 Evaluation of Spiked Sample Data

Spiked samples are homogenous samples containing certified concentrations of known analyte(s). The performance of the reference laboratory can be evaluated using the results of the spiked samples as they represent the best estimate of accuracy and precision for verification testing.

7.4 Data Quality Analysis

The data obtained during verification testing must be of sufficient quality for the appropriate conclusions to be drawn. Data quality can be evaluated based on the following:

- Precision
- Accuracy
- Representativeness
- Completeness
- Comparability

As applicable, these data quality parameters can be used to assess both the technology and the reference laboratory. The statistics used to evaluate each of these parameters should be described in the verification plan, where necessary.

7.4.1 Precision

Precision, in general, refers to the degree of mutual agreement among measurements of the same materials and contaminants. In environmental applications, precision is often specified as a percentage of contaminant concentration.

7.4.2 Accuracy

Accuracy is a measure of how close measured values are to true values. Inaccuracies or biases are the result of systematic differences between these values. The incorporation of blanks, replicates, and spiked samples in the study design will enable a determination of the technology's accuracy during the verification test.

7.4.3 Representativeness

Representative samples, in general, are samples that contain a reasonable cross-section of the “population” over which they are to be used to make inferences. Representativeness may also express the degree to which the sample data represent the capability of the technology.

7.4.4 Completeness

Completeness refers to the amount of data collected from a measurement process expressed as a percentage of the data that would be obtained using an ideal process under ideal conditions. The completeness objective, which is usually 95% or better, should be discussed in the technology-specific verification plan.

7.4.5 Comparability

Comparability refers to the confidence with which one data set can be compared to another. If possible, the field technology will be compared in some way to a reference or baseline method, as discussed in Section 3.3 of this document.

7.4.6 Other Characteristics

Other data characteristics that could be applicable for technologies that give a quantitative measurement, include the following (Reference 2):

- Detection limit
- Linear range
- Reliability
- Interferences
- Matrix effects
- Response stability
- Ease of use
- Maintainability
- Safety measures
- Use of consumables
- Cost

7.5 Qualitative Technologies

Some technologies may produce qualitative rather than quantitative data. The technology may provide a yes/no indication or categorize an environmental parameter, rather than provide a numerical result. Verification of such a technology should consist of determining performance measures such as false positive and false negative frequency, response threshold, and equivalence of duplicate results. The QA plan should state the procedure used to compare the qualitative results with quantitative data from the standard method or reference material and also state the statistical procedures used to quantify the predictive power or uncertainty associated with each method (Reference 2).

8.0 DATA REDUCTION AND DOCUMENTATION OF RESULTS

8.1 Data Reduction

Data reduction refers to the process of converting the raw results into a concentration which will be used for evaluation of performance. The procedures to be used will be technology dependent, but should include a consistent concentration unit, if applicable. Also, if the result is below detection, the concentration should be reported as less than the reporting limits of the technology. A result reported as “0” is not acceptable.

8.2 Documentation Of Results

The structure of the report documenting the results of the verification test should be similar to the verification test plan (see Section 5) and include the following additional elements (Reference 1):

- concise description of the technology
- underlying scientific, engineering, and operating principles
- evidence that the technology is based on sound scientific and engineering principles as documented in the peer-reviewed scientific literature, or documented in reports written for review by technical experts, or determined to be sound on the basis of professional judgment
- description of the necessary components
- range of conditions for operation
- environmental conditions under which the technology operates
- limitations and restrictions of the technology
- description of the methodology employed in the evaluation, including a thorough discussion of the verification test plan and any deviations made from the plan
- discussion of the statistical methods used to assess the quality of the data results as well as the comparison to the reference method
- description of the findings, conclusions, and the basis for the conclusions
- data and other documentation on which these conclusions and recommendations are based

Verification reports for field characterization technologies that have gone through a verification process are available from various sources including EPA’s ETV and SITE Programs and California Environmental Technology Certification Program. A table showing known verifications of field characterization technologies is included as Attachment 2. (Table will be filled in as this guidance goes through review stages.)

9.0 EXAMPLE VERIFICATION PROCESS FOR A SPECIFIC TECHNOLOGY

This section provides a description of the verification process that was used for a specific technology under the Environmental Protection Agency's (EPA) Environmental Technology Verification (ETV) program. This example for the Niton X-ray Fluorescence (XRF) XL Spectrum Analyzer is intended to show how the verification process can be applied to an actual characterization technology. Further information can be found in the ETV Report *Field Portable X-ray Fluorescence Analyzer-Niton XL Spectrum Analyzer* (Reference 11).

9.1 Purpose and Objectives

The purpose of the demonstration, conducted under the Monitoring and Measurement Technologies Program (a component of EPA's SITE program), was to fairly and thoroughly evaluate the performance of field portable XRF analyzers to identify and quantify metals in soils. The XRF analyzer was primarily evaluated for:

- accuracy and precision relative to conventional analytical methods
- influence of sample matrix variations (texture, moisture, heterogeneity, and chemical composition) on performance
- logistical and economic resources needed to operate these analyzers

The performance of the XRF analyzer was compared to standard analytical methods and assessed relative to measurements of standard reference materials, performance evaluation samples, and other quality control samples.

9.2 Reference Methods

EPA SW-846 Methods 3050A/6010A were chosen as the reference methods. Method 3050A is the standard acid extraction method for determining the concentration of metals in soil samples and method 6010A is the standard method used to analyze these extracts.

The analytical laboratory used was based on their costs, ability to meet the demonstration's QA Plan requirements, and ability to perform all the analyses in the required timeframe.

The reference methods were evaluated using precision, accuracy, representativeness, completeness, and comparability to establish the quality of data generated and to ensure that the comparison of the XRF analyzer to reference data was acceptable.

9.3 Site Selection

Forty-six potential sites were screened against the following selection criteria:

- site owner agrees to allow access for the demonstration
- site has soil contaminated with some or all of the target heavy metals
- site is accessible to two-wheel drive vehicles
- site has sand, clay, and/or loam soil textures
- site has surface soil contamination

- selected sites have to have different climates

The list of 46 potential sites was developed by contacting numerous state, regional, and federal regulatory agencies as well as using the Record of Decision Scan database to search for appropriate sites. The two final sites chosen were hazardous waste sites in Iowa and in Washington State as they met most of the selection criteria. They both exhibit a wide range of concentrations for most of the target analytes, are located in different climatological regions of the United States, and combined they exhibit three distinct soil textures: sand, loam, and clay.

9.4 Pre-demonstration Sampling

Sampling was performed at both of the selected sites prior to the demonstration of the technology in order to:

- provide data on, or verify, the extent of surface contamination at each site
- locate optimum sampling areas for the demonstration
- allow the developers to analyze samples from the demonstration sites in advance of the demonstration, and if necessary, refine and recalibrate their technologies and revise their operating instructions
- evaluate samples for the presence of any unanticipated matrix effects or interferences that might occur during the demonstration
- check the QA and QC procedures of the reference laboratory

One hundred soil samples, representing a wide range of metal concentrations and soil textures, were analyzed on each site by the XRF analyzer during the pre-demonstration sampling activities. In addition, 39 samples were sent to the analytical laboratory for analysis by the reference method while 29 of these samples were split and sent to the developers. To assess the proposed sample homogenization procedures, 9 field duplicates were collected and analyzed by the reference method. One performance evaluation sample (with known metal concentrations) was purchased and analyzed by the reference method to provide an initial check of the laboratory's accuracy. Finally, three samples (of low, medium, and high concentrations) were collected at each site, dried, ground, and analyzed by six independent laboratories to create site-specific performance evaluation samples.

9.5 Experimental Design

Complete information about the design of the demonstration plan can be found in the *Final Demonstration Plan for Field Portable X-ray Fluorescence Analyzers* (Reference 12). A brief summary is included here.

Approximately 100 samples were collected from each of clay, loam, and sand soil textures to allow for a determination of the effect of soil texture on data comparability. These samples

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were split for analysis by the XRF and by the reference method. Attachment 3 illustrates the process used for sample analysis. The following types of samples were analyzed:

- *in situ* - unprepared: XRF analyzer took 1 *in situ* measurement in each sample area (4" x 4") that was prepared by clearing all vegetation, debris, and gravel larger than 2 mm in diameter (replicate measurements taken at 4% of sample locations)
- *in situ* - prepared: the 4"x4" sample area was removed to a depth of 1", homogenized, and placed in a 1" deep petri dish prior to the XRF analyzer taking 1 measurement from each sample (replicate measurements taken on same 4% of samples)
- intrusive - unprepared: the sample material was then passed through a No. 10 mesh sieve (2-mm openings) and approximately 10 grams placed in a sample cup for analysis in an intrusive mode by the XRF analyzer (replicate measurements taken on same 4% of samples); sample material from this preparation step was also sent to the laboratory for analysis by the reference method
- intrusive - prepared: a portion of the sample material from the intrusive - unprepared step was then dried and ground to pass a No. 40 sieve (0.425-mm openings) and analyzed in an intrusive mode by the XRF analyzer (replicate measurements taken on same 4% of samples)

9.6 Qualitative Factors

Factors that can impact the data collected but that are difficult to quantify are the qualitative factors. The qualitative factors considered for this demonstration included:

- operator training – The person that would operate the XRF analyzer during the demonstration was trained by the developer of the technology on how to operate it. Based on this training and the field experience gained during the demonstration, the operator completed a subjective evaluation to assess the training and technology operation
- operator effects – Individual differences in sample preparation or operator technique can have a significant effect on the data results. To reduce the potential for errors from the evaluation, the same operator was used for the XRF unit at both sites, the same personnel were used to prepare samples, and only one reference laboratory was used to analyze the samples

9.7 Quantitative Factors

Factors that could affect the quantitative evaluations and the procedures used to evaluate their influences included:

- heterogeneity: For *in situ* - unprepared measurements, heterogeneity was partially controlled by restricting measurements within a 4-by-4-inch area. For measurements

after the initial point-and-shoot preparation, heterogeneity was minimized by sample homogenization. This effect was evaluated through the sample preparation data.

- particle size: The effect of particle size was evaluated using the two intrusive sample preparation procedures. Theoretically, precision and accuracy should increase as particle size decreases and becomes uniform.
- moisture content: It has been suggested that major shifts in sample moisture content can affect a sample's relative fluorescence. This effect could not be evaluated as thoroughly as planned because of the small difference in sample moisture content observed at the two sites.
- interferences: Interferences result from overlapping spectra of metals that emit X-rays with similar energy levels. The reference method analysis provided data on the concentration of potential interferants in each sample.

9.8 Evaluation of Analyzer Performance

The concentrations for each target metal analyte measured by the XRF analyzer were compared to the reference data from the analytical laboratory, and to other QA/QC sample results. These measurements were used to determine an analyzer's accuracy, data quality level, method precision, and comparability to reference methods. Performance evaluation and standard reference material samples were used to assess analyzer accuracy. Relative standard deviations on replicate measurements were used to determine analyzer precision. These data were also used to help determine the data quality of the XRF analyzer's output. The data comparability and quality determination was primarily based on a comparison of the analyzer's data and the reference data. Linear regression and a matched pairs t-test were the statistical tools used to assess comparability and data quality. A detailed description of how the statistical tools were used to assess the data can be found in the *Final Demonstration Plan for Field Portable X-ray Fluorescence Analyzers* (Reference 12).

The results of the performance evaluation of the Niton XL Spectrum Analyzer include the following (Reference 11):

- Detection limits: Precision-based detection limits were determined by collecting 10 replicate measurements on site-specific soil samples with metals concentrations 2 to 5 times the expected method detection limits (MDL). The results were 130 milligrams per kilogram (mg/kg) or less for all of the reported target analytes except chromium, which was determined to be 900 mg/kg.
- Throughput: Average throughput was 20 - 25 analyses per hour using a live count time of 60 seconds. This rate only represents the analysis time since different personnel were used to prepare the samples.
- Drift: This was evaluated using the results of an analysis of a standard reference material (SRM) calibration check sample which contained quantifiable levels of arsenic, copper, lead, zinc, and iron. Over the course of the demonstration, this sample

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was analyzed approximately 100 times. The mean recovery for these analytes was between 85 and 140 percent. The drift relative standard deviation (RSD) for the mean recovery of these analytes was less than 8 percent.

- **Completeness:** The XL Spectrum Analyzer produced results for 1,258 of the 1,260 samples for a completeness of 99.8 percent. The two lost data points were a consequence of operator error.
- **Blank results:** More than 100 lithium carbonate blanks were analyzed during the demonstration. None of the reported analytes were observed above the method detection limits.
- **Precision:** The goal of the demonstration was to achieve RSDs less than 20 percent at analyte concentrations of 5 to 10 times the method detection limits. The RSD value for arsenic was 9.2 percent, 13.2 percent for copper, 6.5 percent for lead, and 11.2 percent for zinc. Chromium was not reported due in part to the short 60 live-second count time.
- **Accuracy:** Intra-method accuracy was assessed using site-specific soil PE samples and soil SRMs. The data showed that 18 of 28 or 64.2 percent of the PE sample analytes had recoveries within the quantitative acceptance range of 80 - 120 percent. For the soil SRMs, 11 of 16 (68.7 percent) of the results were within the 80 - 120 percent recovery range.
- **Comparability:** This demonstration showed that the XL Spectrum Analyzer produced data that exhibited a log₁₀ - log₁₀ linear correlation to the reference data. The coefficient of determination (r^2) which is a measure of the degree of correlation between the reference and field data was 0.82 for arsenic, 0.50 for chromium, 0.92 for copper, 0.96 for lead, and 0.89 for zinc.
- **Data quality levels:** Using the demonstration derived precision RSD results and the coefficient of determination as the primary qualifiers, the XL Spectrum Analyzer produced definitive level data for lead and data of quantitative screening level for arsenic, copper, and zinc. Since a precision RSD value was not determined for chromium, no data quality level can be assigned.

Based on the performance of the XL Spectrum Analyzer, this demonstration found it to be an effective tool for characterizing the concentration of metals in soil samples. As with all field portable XRF analyzers, unless a user has regulatory approval, confirmatory (reference) sampling and data correction is recommended when using this technology for site characterization or remediation monitoring.

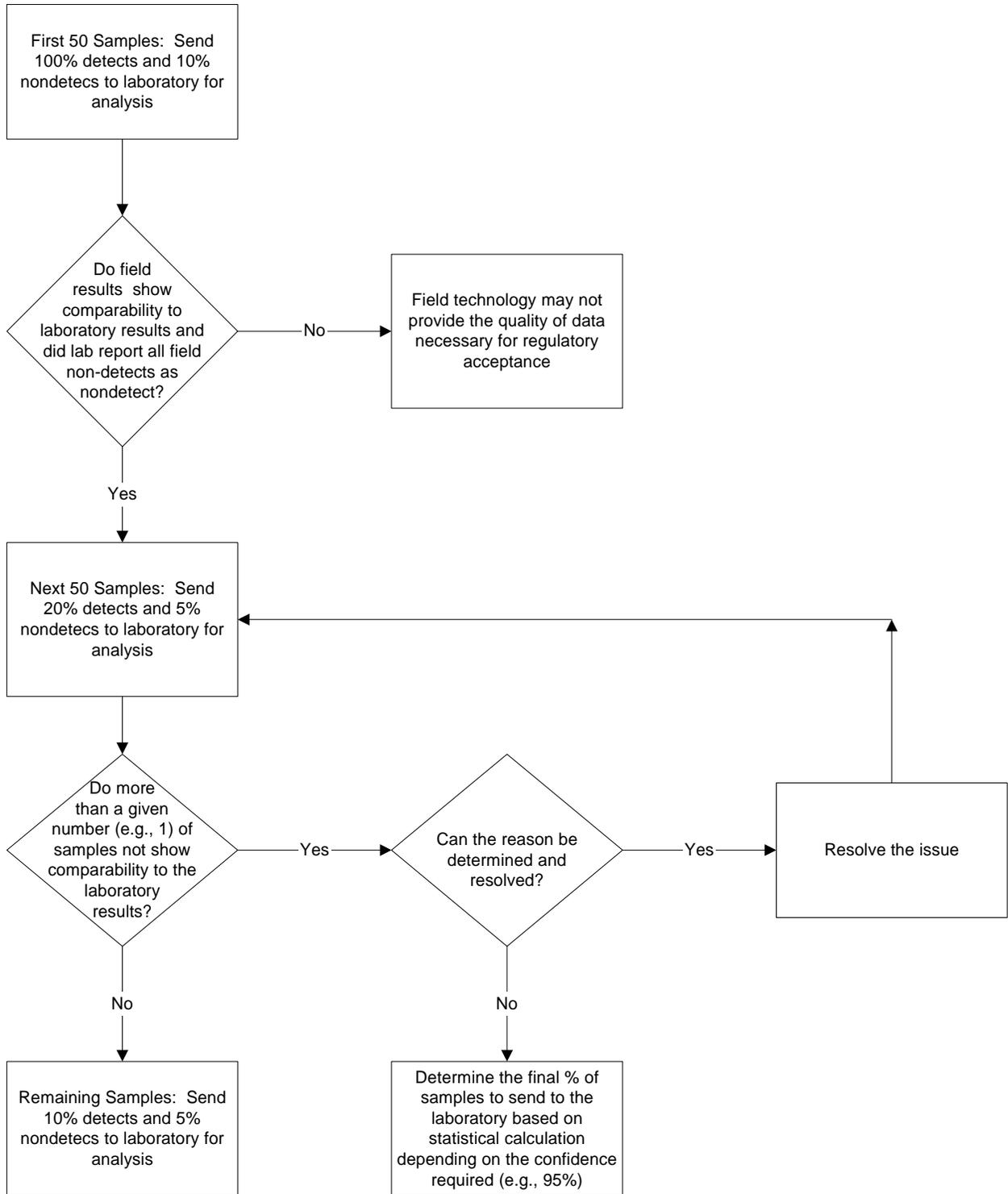
10.0 REFERENCES

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12. Final Demonstration Plan for Field Portable X-ray Fluorescence Analyzers, PRC Environmental Management, Inc. 1995.

ATTACHMENT 1
Example of a Sampling & Analysis Process To Gain Regulatory Acceptance of Field Characterization Technology

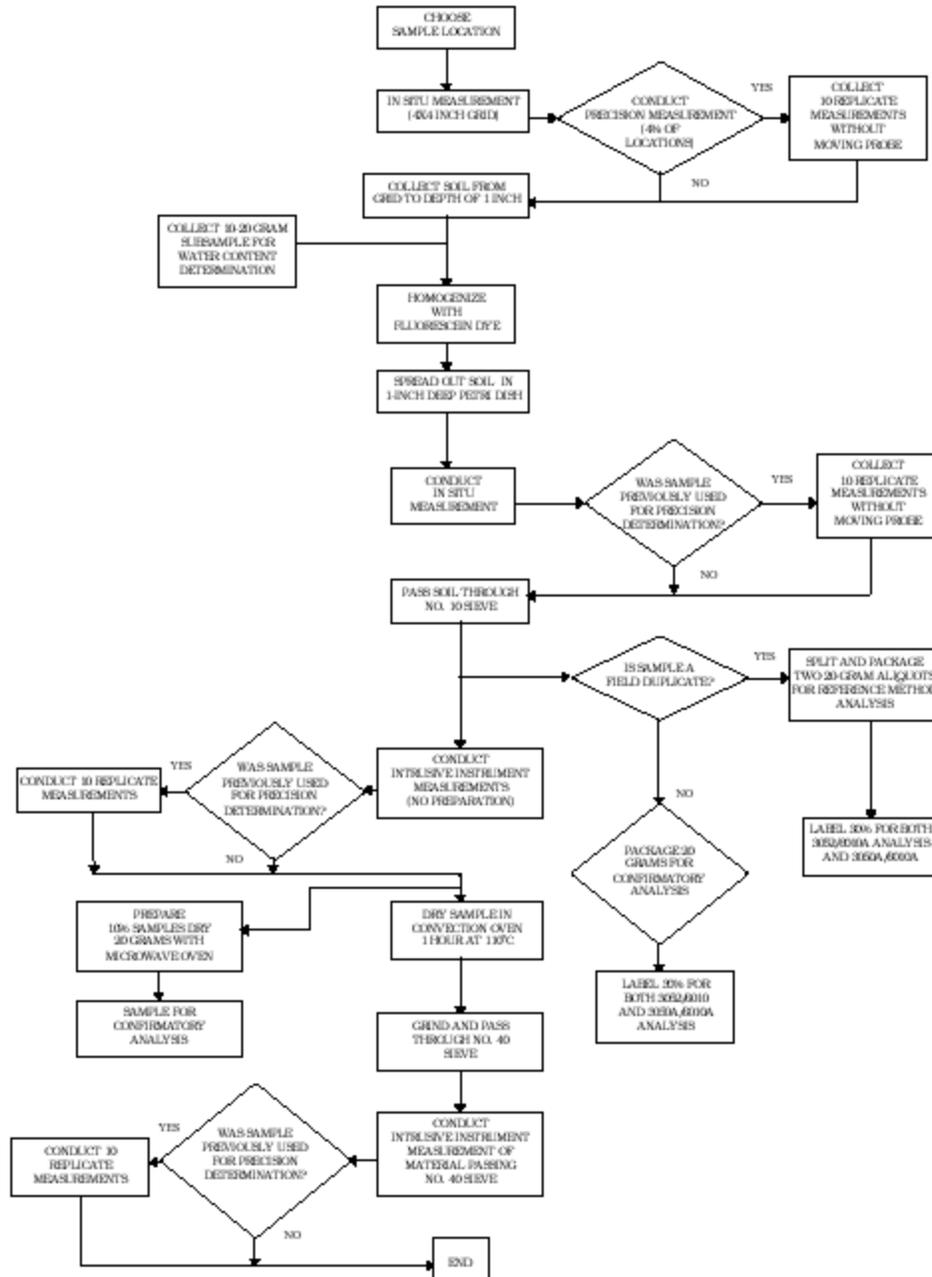


ATTACHMENT 2
Verifications of Field Characterization Technologies

Field Technology Type	Technology Name	Company/Manufacturer	Contaminant	Reference Method	Verification
X-Ray Fluorescence	MAP Spectrum Analyzer	EDAX Portable Products	Metals	EPA SW-846 Method 6010A	EPA ETV Verification Report
	SEFA-P Analyzer	HNU Systems, Inc.			EPA ETV Verification Report
	X-MET 920-P and X-MET 940	Metrorex, Inc.			EPA ETV Verification Report
	X-MET 920-MP	Metrorex, Inc.			EPA ETV Verification Report
	XL Spectrum Analyzer	Niton Corporation			EPA ETV Verification Report
	TN 9000	TN Spectrace			EPA ETV Verification Report
	TN Pb Analyzer	TN Spectrace			EPA ETV Verification Report
PCB Field Analytical					
Explosives Detection					
Portable Gas Chromatograph- Mass Spectrometer					
TPH measurement devices	Synchronous Scanning Luminoscope	Environmental Systems Corporation	Total petroleum hydrocarbons	EPA SW-846 Method 8015B (modified)	EPA SITE Verification Report
	EnSys Petro Test System	Strategic Diagnostics Inc.			EPA SITE Verification Report

ATTACHMENT 3

Sample Preparation & Analysis Process Used for the Demonstration of XRF Technology



(Source: Final Demonstration Plan for Field Portable X-ray Fluorescence Analyzers, PRC Environmental Management, Inc. 1995)