U.S. Environmental Protection Agency
Environmental Technology Verification Program
Advanced Monitoring Systems Center

and

Environment Canada
Environmental Technology Verification Program

Joint Verification Protocol for Technologies for Rapid Detection of Whole Soil and Soil Extract Toxicity

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CANADA VERIFIED VÉRIFIÉ

Joint Verification Protocol

for

Verification of Technologies for Rapid Detection of Whole Soil and Soil Extract Toxicity

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This joint verification protocol was developed through a collaboration between the Environmental Technology Verification (ETV) programs of the United States and Canada and is intended for use by both Canada and the United States either individually or together in jointly verifying technologies which will be recognized by both countries. This joint protocol can also be used by a vendor who would like to receive verification from either or both countries. This joint verification protocol expands upon and replaces the generic verification protocol previously prepared by the U.S. Environmental Protection Agency's (EPA's) ETV Advanced Monitoring Systems (AMS) Center (*Generic Verification Protocol for Technologies for Rapid Detection of Soil Toxicity,* April 2007). The AMS Center's generic protocol was prepared in coordination with the EPA's Office of Solid Waste and Emergency Response and National Exposure Research Laboratory and included input from vendors and stakeholders. Peer reviewers for the protocol were Robert Seyfarth, Kenneth Hill, and Amy Juchatz of the Suffolk County (New York) Department of Health Services' Division of Environmental Quality; Dr. John Hayse, I. Hlohoskyj, and Leroy Walston of Argonne National Laboratory's Environmental Science Division; and Dr. Karen Bradham of EPA's National Exposure Research Laboratory. Dr. Lisa Taylor of Environment Canada's Environmental Science and Technology Centre provided significant contribution to the revision of the AMS Center's generic protocol to create the joint protocol. The contributions to this protocol from John Neate of ETV Canada are also gratefully acknowledged. The joint protocol was reviewed by Robert Seyfarth and Leroy Walston, as well as by Deana Crumbling of EPA's Office of Solid Waste and Emergency Response.

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Note: Test/Quality Assurance Plans developed from this Joint Verification Protocol for verifications which will be recognized by both the U.S. and Canada should be distributed to the Verification Program and Verification Organization associated with both countries. Verifications which apply to one country require distribution only to the Verification Program and Verification Organization pertinent to the verifying country.

Verification Program (United States: U.S. EPA ETV, Canada: Environment Canada ETV)

Verification Organization (United States: Battelle AMS Center, Canada: ETV Canada, Verification entity to be determined)

Peer Reviewers

Vendors of Technologies for Rapid Detection of Soil Toxicity

Reference Laboratory

Test Facility, if applicable

Test Collaborators, if applicable

Subcontractors, if applicable

LIST OF ABBREVIATIONS/ACRONYMS

AMS	Advanced Monitoring Systems
COA	certificate of analysis
COC	chain-of-custody
DQI	data quality indicator
EC_{50}	median effective concentration causing 50% inhibition
EPA	U.S. Environmental Protection Agency
ETV	Environmental Technology Verification
LOEC	lowest observed effect concentration
LRB	laboratory record book
MSD	minimum significant difference
NIST	National Institute of Standards and Technology
NOEC	no observed effect concentration
PCB	polychlorinated biphenyl
PD	percent difference
pdf	Adobe portable document format
PE	performance evaluation
QA	quality assurance
QC	quality control
QCS	quality control samples
QMP	quality management plan
RSD	relative standard deviation
SOP	standard operating procedure
TCDD	2,3,7,8-tetrachlorodibenzodioxin
TQAP	test/quality assurance plan
TSA	technical systems audit

SECTION A
PROJECT MANAGEMENT

A1 VERIFICATION TEST ORGANIZATION

This protocol provides generic procedures for implementing a verification test for technologies that rapidly detect soil toxicity in whole soil or soil extracts. This protocol outlines a testing approach which is acceptable under both the U.S. EPA ETV and ETV Canada verification programs. However, acceptability of the testing approach as outlined in this protocol does not imply automatic verification by both the U.S. EPA ETV and ETV Canada verification programs. Each specific round of testing will require preparation of a Test/Quality Assurance Plan (TQAP). The TQAP will specify which verification programs and verification organizations are involved in testing and verifying the technologies involved. This could be jointly with both the U.S. EPA ETV and ETV Canada programs conducting testing and verification together, in which the verification would be recognized by both countries' programs, or individually where either U.S. EPA ETV or ETV Canada conducts testing and verification and the verification is recognized by only one country's program.

Because the organizations involved in testing may vary, specific roles and responsibilities will not be defined here, but must be defined in the TQAP prepared for each round of testing. Information on roles and responsibilities defined in each TQAP should include the following groups or individuals involved in each test:

- Verification program(s)
- Verification organization(s)
- Key testing staff (verification organization program manager, testing leaders, verification coordinators, technical staff, etc.)
- Technology vendors
- Reference laboratories
- Test facilities
- Quality Managers for both the verification program and the verification organization.

A2 BACKGROUND

The purpose of verification programs such as the U.S. EPA and Canadian Environmental Technology Verification (ETV) programs is to provide objective and quality-assured performance data on environmental technologies, so that users, developers, regulators, and consultants can make informed decisions about purchasing and applying these technologies. Stakeholder committees of buyers and users of such technologies recommend technology categories, and technologies within those categories, as priorities for testing. As documented in meeting minutes, technologies for rapidly detecting soil toxicity were identified as a priority technology category through the U.S. EPA ETV Advanced Monitoring System (AMS) Center stakeholder process since these technologies have the potential to make the evaluation of soil toxicity more efficient and timely.

Soil toxicity testing can be used at hazardous waste sites to screen for particular areas of concern or to assist in monitoring the effectiveness of cleanup. Soil toxicity tests do not require knowing the contaminants present at the site; they are typically used as a broad range screen of all potentially toxic compounds that may be present. Traditional soil toxicity tests include evaluations such as seed germination and root elongation, as well as organism-based tests such as earthworm survival.[(1)] Tests such as these can take several weeks to achieve results. This protocol provides procedures for a verification test of rapid analysis technologies that detect toxicity in whole soil and soil extracts. The objective of this soil toxicity technology verification test is to evaluate the technology's ability to detect certain analytes that are particularly toxic to humans by adding them, individually, to a controlled experimental matrix, as well as by testing various "real-world" soil samples where the toxins may be present alone or with various other toxins. This joint protocol outlines testing for a number of contaminants that are common to site cleanups and known to be toxic, but does not include all toxic compounds or testing in all situations which may be encountered in a site cleanup or evaluation situation. Data generated from verification tests based on this joint protocol are intended to provide one set of objective and quality assured performance data on soil rapid toxicity technologies, to assist users, developers, regulators, and consultants in making informed decisions about purchasing and properly applying these technologies.

This verification test will determine the performance characteristics of commercially available technologies that can provide results that make quicker, more efficient soil toxicity determinations than the traditional tests which may take several weeks. Critical characteristics of the soil toxicity technologies that will be assessed during this testing include the following:

- Endpoint
- Precision
- False negative rate
- False positive rate
- Sensitivity
- Matrix effects
- Data completeness
- Operational factors such as ease of use and maintenance
- Field portability.

A3 VERIFICATION TEST DESCRIPTION AND SCHEDULE

A3.1 Summary of Technology Category

Technologies applicable to this technology category can be those designed to directly test the soil, or just the soil extract. These technologies are not intended to be a substitute for chemical analyses for contaminants of interest but rather can be used as a complement that provides an assessment of the biological response of toxicity. Conventional soil toxicity methods often can take weeks to achieve results. Technologies to be evaluated in the verification of rapid soil toxicity technologies include those that produce results within a substantially reduced time. This may be within 24 hours for technologies that test soil extracts to within several days for those involving whole soil. Such rapid soil toxicity technologies have the potential to expedite the decision-making process for regulators. Rapid soil toxicity technologies do not provide a measured concentration of specific toxins; rather, they provide a broad range screen of the toxic nature of the soil. Specific procedures for the operation of each technology will be supplied as part of the verification test. For technologies which operate by extracting an aliquot of soil and then testing the extract, an extract may be added to bacteria, bioluminescent plankton, or other such organisms or compounds which produce a measurable response that varies based on the

toxicity of contaminants in the soil. The specific response may vary by technology, but could include a change in color or light intensity, respiration rate, or other response that is related to the concentration level of the contaminant(s). Similarly, whole soil tests may involve germinating seeds in both contaminated soil and reference soil and comparing the decrease or absence of germination or root growth in the contaminated soil to that which occurs in the reference soil. In this case, the inhibition in germination or root growth is related to contaminant concentration levels.

A3.2 Verification Test Schedule

A verification test following this protocol should take approximately nine months to complete. Test planning and preparation may take place over a period of several months once vendors are committed to the test. Actual testing should be completed within two months. Data review and reporting should be completed within four to five months. Table 1 shows a general schedule of testing and data analysis/reporting activities to be conducted in a verification test that follows this protocol. The test procedures are described in Section B of this protocol. Subsequent to testing, a separate verification report will be drafted for each technology. Each draft report will be peer-reviewed, revised, and submitted for final approval. Technologies for detecting soil toxicity and associated equipment (but not consumables) will be returned to the vendors at the completion of report writing.

A3.3 Test Facility

The test facility should be a location that can accommodate laboratory testing of technologies for detecting toxicity in soil. This could be laboratory facilities at Battelle, Environment Canada, or other such laboratory facilities that routinely test soil. Field portability testing, if applicable, will be conducted by transporting the technology from a laboratory to a non-laboratory area. In addition to a traditional field setting, non-laboratory areas could include warehouses, shipping/receiving areas, storerooms, courtyards, and/or parking lots.

A3.4 Health and Safety

All reference analyses and verification testing will follow the safety and health protocols in place for the test facility. This includes maintaining a safe work environment and a current awareness of handling potentially toxic chemicals. Exposure to potentially toxic chemicals will be minimized, personal protective equipment will be worn, and safe laboratory practices will be followed.

Table 1. General Verification Test Schedule[a]

Month	Testing Activities	Data Analysis and Reporting
1-2	• Prepare draft TQAP and submit for vendor and peer reviews	
2-3	• Revise draft TQAP • Finalize and obtain vendor approval of TQAP • Procure necessary standards and reagents • Vendor to set up technology and train technical staff on technology use	
4-5	• Conduct verification tests • Conduct reference tests and performance evaluation audit of reference methods • Conduct technical systems audit	• Review and compile test data and records as they become available • Review and summarize verification testing staff observations • Begin preparation of report template
6		• Evaluate and analyze data generated during testing • Conduct data quality audits • Complete report template
7		• Complete draft reports and submit for vendor and peer review
8		• Revise draft reports and submit final reports for approval by the verification program
9	• Return equipment to vendors	• Distribute finalized, approved reports • Post reports and verification statements on verification program and verification organization web sites

[a] Verification schedule begins once vendor(s) and collaborators(s) are committed to the verification test.

A4 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

In performing the verification test, the verification organization will follow the technical and QA procedures specified in this protocol and will comply with the data quality requirements

in the verification organization's quality management plan. The objective of this verification test is to evaluate the performance of soil toxicity detecting technologies in their ability to measure the presence of toxins in whole soil or soil extracts under controlled laboratory conditions. This evaluation will assess the capabilities of the soil toxicity technologies to detect toxins added to a controlled experimental matrix, as well as their ability to detect toxins in "real-world" environmental samples. The evaluation will include a comparison of the soil toxicity technology results to known concentrations of toxins in the test samples that will be confirmed as described in Section B4. Additionally, this verification test will rely upon verification testing staff observations to assess other performance characteristics of the technologies. Below is a discussion of the quality objectives and the criteria for measurement data that have been established to ensure that the test objectives are met.

A4.1 Quality Objectives

Data quality objectives assure that the data quality, quantity, and type are appropriate to meet the verification test objectives and specify the minimum acceptance criteria for these parameters. Data quality objectives for this verification test include those related to the reference method performance and those related to the soil toxicity detecting technology performance, as well as those related to documenting verification testing staff observations. Data quality objectives for the reference methods (see Section B4) are presented in terms of data quality indicator (DQI) criteria for the critical measurements associated with the reference methods. The DQI criteria are listed in Table 2 and discussed in Section A4.2. The reference method data quality relies, in part, on proper sample preparation, proper application of the reference method, and proper maintenance of reference method instrumentation. The verification organization will rely on the vendor's data quality objectives for each technology in order to ensure that the technology is performing properly during testing. This will include adhering to each vendor's criteria for calibration and performance of positive and negative control samples. The technology data quality relies on proper operation and maintenance of the technologies and proper sample preparation, as instructed by the vendor. Quantitative data quality objectives for the operator observations have not been defined but are incorporated into documentation requirements and data review, verification, and validation requirements for this verification test.

Table 2. DQIs and Criteria for Critical Measurements for Reference Method

DQI	Method of Assessment	Frequency	Minimum Acceptance Criteria	Corrective Action
Bias and Accuracy of Sample Measurements	Initial Calibration—various levels as specified in reference method	As required in reference method	Refer to reference method criteria	Investigate sources of contamination or changes in instrument parameters; perform instrument maintenance as needed; reanalyze fresh standard or sample, or repeat initial calibration.
	Calibration Check Sample—single-level continuing check of calibration as specified in reference method	As required in reference method	Refer to reference method criteria	
	Method Blank	As required in reference method	Refer to reference method criteria	
	Spiked Samples	As required in reference method	Refer to reference method criteria	
Completeness	Amount of valid data obtained	Overall number of data points collected for reference method	90% of overall data points collected should be valid.	If feasible, analyze additional samples to meet the acceptance criterion.
Method Representativeness	Performance Test Sample	Once, prior to verification testing	Results within ± 10% of expected value for standard solutions, results within certified limits for standard reference materials	Evaluate reference method performance; perform maintenance or recalibration as required, repeat performance test. If performance test criteria cannot be met, consider alternative reference laboratories.

A4.2 Criteria for Measurement Data

Table 2 presents the DQIs and general criteria for the reference method critical measurements. Specific criteria should be added to the TQAP once the reference method is known. The reference method measurement quality will be ensured by adhering to these DQI criteria and monitored by following the calibration procedures and frequency recommended in each respective reference method and by including method blank or spiked samples as indicated in each reference method. Additionally, performance test samples will be sent to each laboratory providing reference method analyses prior to analysis of verification test samples. Performance

test samples will be standard solutions or standard reference materials containing known quantities of the analytes of interest. Each vendor will provide criteria for the soil toxicity technologies for critical measurements related to calibration standards and recommendations for appropriate positive and negative controls and their critical measurements. The verification organization's Quality Manager or designee will perform a TSA at least once during this verification test to review these QA/quality control (QC) requirements. The ETV verification program's Quality Manager (US and/or Canada) also may conduct an independent TSA if desired.

A5 SPECIAL TRAINING/CERTIFICATION

Documentation of training related to technology testing, field testing, data analysis, and reporting should be maintained for all technical staff involved in verification testing. Location of these training records should be documented in the TQAP. Documentation of the expertise and experience of collaborators and/or subcontractors must be similarly available. Any minimum education or experience requirements for testing staff should be specified in the TQAP. The verification organization Quality Manager may verify the presence of appropriate training records prior to the start of testing. If technical staff operate and/or maintain a technology during the verification test, the technology vendor will be required to train those staff prior to the start of testing. The verification organization will document this training with a consent form, signed by the vendor, that states which specific technical staff have been trained on their technology.

A6 DOCUMENTATION AND RECORDS

The records for this verification test will include the TQAP based on this protocol, chain-of-custody (COC) forms, laboratory record books (LRBs), data collection forms, electronic files (both raw data and spreadsheets), and the final verification reports and verification statements. The storage location for these records should be specified the TQAP. The verification program(s) should be notified before disposal of any files. The QA/QC documentation and results of the reference measurements made by the reference laboratory should be submitted to the verification organization immediately upon completion of all sample analyses and maintained with the

records for this test. Table 3 has further details regarding the data recording practices and responsibilities.

Table 3. Summary of Data Recording Process

Data to Be Recorded	Where Recorded	How Often Recorded	By Whom	Disposition of Data
Dates, times, and details of test events, technology maintenance, downtime, etc.	ETV LRBs or data recording forms	Start/end of test procedure, and at each change of a test parameter or change of technology status	Technical staff	Used to organize and check test results; manually incorporated in data spreadsheets as necessary
Technology calibration information	ETV LRBs, data recording forms, or electronically	At technology calibration or recalibration	Technical staff or vendor performing the calibration	Incorporated in verification report as necessary
Technology readings	Either recorded electronically by the technology and downloaded to an independent computer or storage medium, hard copy data printed by the technology and taped into an ETV LRB, or handwritten records into an ETV LRB or on data sheets	Every sample analysis.	Technical staff	Transferred to or manually entered into spreadsheet for statistical analysis and comparisons
Sample preparation and reference method analysis procedures, calibrations, QA, etc.	LRBs, COC, or other data recording forms	Throughout sampling and analysis processes	Technical staff and Reference laboratory	Retained as documentation of reference method performance
Reference method results	Electronically from analytical method or documented in handwritten records	Every sample analysis	Reference laboratory	Transferred to or manually entered into spreadsheets for calculation of results, and statistical analysis and comparisons as needed

All written records must be in ink. Any corrections to notebook entries, or changes in recorded data, must be made with a single line through the original entry. The correction is then to be entered, initialed, and dated by the person making the correction. In all cases, strict confidentiality of data from each vendor's technology, and strict separation of data from different

vendors' technologies, will be maintained. Separate files (including manual records, printouts, and/or electronic data files) will be kept for each technology.

SECTION B
MEASUREMENT AND DATA ACQUISITION

B1 EXPERIMENTAL DESIGN

This joint protocol outlines testing for a number of contaminants that are common to site cleanups and known to be toxic to humans, but does not include all toxic compounds or testing in all situations which may be encountered in a site cleanup or monitoring situation. Data generated from verification tests based on this joint protocol are intended to provide one set of objective and quality assured performance data on rapid soil toxicity technologies, to assist users, developers, regulators, and consultants in making informed decisions about purchasing and properly applying these technologies that would be acceptable for consideration under both the US EPA ETV and ETV Canada verification programs. These technologies do not provide identification or concentration of specific contaminants, but serve as a rapid screening tool to determine whether the soil being tested is toxic. As part of this verification test, the technologies will be subjected to various concentrations of chemicals representing several categories of common contaminants such as commercial solvents, pesticides, persistent pollutants, and metals. At a minimum, the categories listed in Table 4 should be evaluated during verification testing. The specific compounds to be tested may be added or replace compounds in Table 4 (as described in the TQAP) depending upon the capabilities of the technologies being tested. For technologies evaluating soil extracts, each contaminant will be added individually to separate aliquots of sand, and the spiked sand will be analyzed by the technologies. Sand is recommended as the matrix for the spiking experiments because it is inert and it is anticipated will minimally retain the contaminants of interest thereby providing an estimate of technology performance in the case where nearly 100% of the contaminant would be extractable. For technologies evaluating whole soil, each contaminant will be added individually to separate aliquots of artificial soil [e.g., Organization of Economic Cooperation and Development (OECD) artificial soil], and the spiked soil will be analyzed by the technologies. Artificial soil is recommended for the whole soil technologies to optimize the biological response and more

accurately simulate interactions of the chemical with soil properties. The physical properties of the artificial soil may vary depending on the desired characteristics (e.g., water content may be manipulated to optimize the biological response). The exact procedures for sample preparation will be detailed in each TQAP prepared for testing. Additionally, both soil extract and whole soil technologies will be challenged with "real-world" environmental samples of various soil types containing a variety of the contaminants in Table 4 to evaluate the technology performance on samples more representative of those found in practical application of the technologies. These samples are described in Section B1.1.

All of the technologies will be tested in a laboratory. The technologies designed for use in a field location will also be tested at a non-laboratory venue.

The analyses will be performed according to the vendor's recommended procedures as described in the user's instructions or manual, or during training provided to the technical staff. Similarly, calibration and maintenance of the technologies will be performed as specified by the vendor. Results from the technologies being verified will be recorded manually by the operator on appropriate data sheets or captured in an electronic data system and then transferred manually or electronically for further data workup. Qualitative operational characteristics of each technology such as ease of use will be assessed through observations made by the technical staff throughout the verification test. The results from each technology will be reported individually. According to ETV policy, no direct comparison will be made between technologies, but each technology will undergo similar testing and will be reported in a similar manner.

Table 4. Categories and Example Contaminants

Category	Example Contaminant
Commercial solvents	Trichloroethylene Toluene
Carbamate pesticide	Aldicarb
Organophosphate pesticide	Dicrotophos
Metals	Arsenic Lead Mercury Cadmium
Persistent pollutants	Polychlorinated biphenyls (PCBs) (as Aroclor 1254) 2,3,7,8- tetrachlorodibenzodioxin (TCDD) Benzo[a]pyrene

B1.1 Test Procedures

The verification test for technologies that detect toxicity in whole soil and/or soil extracts will focus on a broad range of samples to provide a variety of toxin concentrations. This verification will focus on evaluating dose/response relationships to specific contaminants known to be toxic to humans as well as assessing the technology's ability to provide a toxic response to real-world environmental samples for which known contaminants have been well characterized using standard analytical laboratory methods.

The first sample type will be performance test samples where individual toxins will be added to a clean sand or artificial soil. Sand is recommended for soil extract testing as an inert matrix which will minimally retain the toxins. Use of an inert matrix will eliminate the matrix itself from influencing the lowest detectable concentration of each contaminant and will evaluate technology performance under conditions where the toxin is anticipated to be nearly 100% extractable. For whole soil toxicity testing, an artificial soil is recommended to optimize the biological response and more accurately simulate interactions of the chemical with soil properties. Because the types of technologies anticipated to be tested will provide a broad range screen of all potentially toxic compounds that may be present, the sand/artificial soil selected should be free of any compounds which would cause a toxic response and not just free of the contaminants of interest for the verification test. The sand/artificial soil will be spiked with each contaminant at concentrations ten times screening or remediation goal levels (e.g., EPA Region 9 Superfund Preliminary Remediation Goals) as the highest concentration and will be analyzed in replicate (minimum of three). Subsequent tenfold dilutions (i.e., spiking the sand/artificial soil with a contaminant solution which is tenfold dilute from the starting level) will be prepared and analyzed in replicate (minimum of three) until there is no longer a measurable response indicating toxicity (i.e., inhibition as measured by each technology such as a reduction in light output, change in respiration rate, etc.), up to a maximum of five dilutions below the highest concentration. From these data, the lowest concentration at which the toxicity can be detected can be estimated for each technology with respect to each contaminant. The second sample type will be "real-world" environmental samples and will consist of 5 to 10 soils collected from various cleanup sites or standard reference soils with well documented soil characteristics. These samples will reflect a variety of soil types and will include soils known to contain the

contaminants of interest in this test (individually or in combination with other contaminants) as well as some soils which are known to be free of contaminants, such as American Society for Testing Materials artificial soil or Environmental Resource Associates Semivolatile Blank Soil (Catalog Number 056). The environmental samples will be dried and homogenized (e.g., oven dried using low heat with specifics to be detailed in the TQAP) prior to use in testing to ensure that sample homogeneity is not a significant factor in technology performance. Because the drying and homogenization process has the potential to affect the concentration of contaminants in the samples, the concentration of contaminants will be measured after the drying and homogenization process has taken place to ensure that the measured concentrations of contaminants in the environmental samples accurately reflect the material used in testing. Contaminants in the environmental samples will be measured using the same reference methods that will be used to confirm the concentration of spiked contaminants in the performance test samples. Appropriate soil characteristics such as total organic carbon, grain size distribution, and pH should also be measured once the environmental samples have been dried and homogenized. Information about the soil characteristics may aid in understanding differences in the various environmental samples that will be tested. To the extent possible, each time verification testing is conducted following this joint protocol, attempts should be made to use the same environmental sites, or at a minimum sites with comparable types of soil and types and quantities of contaminants. As with the performance test samples, environmental sample selection should be made with consideration of the fact that the rapid toxicity tests may respond to all toxic compounds and not just the contaminants included in verification testing. Therefore, environmental samples should be as well characterized as possible prior to use in testing. Test results, and in particular assessments of false positive/negatives and matrix effects, should take into consideration whether the technology could be responding to unknown toxic compounds in the samples. The third type of sample will be quality control samples. Quality control samples are discussed further in Section B5.

It should be noted that the technologies covered by this protocol may include test organisms (e.g., microorganisms or invertebrates) which may involve handling, culture or preparatory work and will likely vary with each type of technology. It is expected that each technology vendor will include instructions on proper preparation and handling of any test organisms and that each vendor will outline in detail any procedures necessary to ensure healthy

test organisms (i.e., storage conditions, light, temperature, feeding etc.) and any procedures necessary to ensure an accurate measurement of the biological response (i.e., UV light, spectrophotometer, microscopes etc.). Additionally, each vendor will specify any QA aspects that should be monitored to ensure proper preparation and handling of organisms (i.e. fridge temperature records, calibration records, organism suppliers, taxonomic verification records, health records etc.). Any preparation or QA aspects which will be uniformly applied across all participating technologies will be detailed in the individual TQAPs prepared for tests performed using this protocol.

Also note that inter-unit reproducibility (e.g., test kits from different lots, multiple detectors, etc.) and inter-operator reproducibility are not addressed in this protocol. Should these parameters be desired, the procedures for evaluating them will need to be added to TQAPs prepared for specific tests. In general the same technician will be used to perform all testing where possible. At a minimum, only technicians with equivalent training and experience operating the technology will be used to perform verification testing.

The technologies will be evaluated for the parameters listed in sections B1.1.1 to B1.1.9. If modification of these parameters is required due to the nature of the technologies being tested, any changes will be described in the test-specific TQAP.

B1.1.1 Endpoint

Each technology produces its own unique biological or biochemical endpoint derived from the inhibition data gathered when analyzing various concentrations of contaminants in soil [e.g., median effective concentration causing 50% inhibition (EC_{50}) values]. For each technology, the endpoint used for verification testing will be recommended by the vendor. The endpoint will be used to assess whether or not there was a response to a test sample.

B1.1.2 Precision

Inhibition results (endpoints) specific to each technology from replicates (minimum of three) of each test sample will be evaluated. The average measurement, standard deviation (S), and relative standard deviation (RSD) of the replicate measurements will be calculated and reported in order to evaluate the precision of the technologies. To the extent possible and

appropriate, precision values will be reported with average data values so that measurement uncertainty is understood.

B1.1.3 False Negative Rate

The false negative rate, or frequency of performance test sample inhibitions which are similar to the negative control reported when a contaminant is present in the performance test sample at toxic concentrations, will be calculated. Note that real-world environmental samples could be used for the assessment of false negatives but this would make the evaluation much more complicated due to the possibility of matrix effects.

B1.1.4 False Positive Rate

The false positive rate, or frequency of performance test sample detectable inhibitions which are reported for unspiked samples, will be calculated. Note that real-world environmental samples could be used for the assessment of false positives but this would make the evaluation much more complicated due to the possibility of matrix effects.

B1.1.5 Sensitivity

Various contaminants will be added individually to a controlled experimental matrix at multiple concentration levels and analyzed by the participating technologies to assess their ability to detect the toxicity of these contaminants (performance test samples). After analyzing several concentrations of each contaminant (i.e., ten times the screening or remediation goal level specified in the TQAP and subsequent tenfold dilutions up to a maximum of five dilutions below the highest concentration), a sensitivity assessment will be made. The sensitivity assessment to be used will be described in the TQAP because it may depend on the technologies being tested. Examples of sensitivity assessments that could be used are an evaluation of the lowest tested concentration which gives a response significantly different from the negative control or a calculation of the lowest observed effect concentration (LOEC) and no observed effect concentration (NOEC) along with an associated minimum significant difference (MSD) value.

B1.1.6 Matrix Effects

Five to ten environmental samples representing a variety of soil types and contaminants or mixtures of contaminants will be analyzed. The concentrations of contaminants present in the environmental samples will be measured according to reference methods. The technology's ability to detect contaminants in the environmental samples will be compared with the lowest detectable level of contaminant determined for each technology to assess whether the environmental sample matrix influenced the ability of the technology to detect toxicity.

B1.1.7 Data Completeness

Data completeness will be determined as the number of valid measurements (i.e., useable endpoint measurements with the technology) out of the total number of measurements taken. The cause of any substantial loss of data will be established from technical staff observations or technology records and noted in the discussion of the data completeness results.

B1.1.8 Operational Factors

Operational and sustainability factors such as maintenance needs, calibration frequency, data output, consumables used, ease of use, repair requirements, waste production, and sample throughput will be evaluated based on technical staff observations. An LRB or data sheets will be used to document observations. Examples of information to be recorded include the daily status of diagnostic indicators for the technology, use or replacement of any consumables, the effort or cost associated with maintenance or repair, vendor effort (e.g., time on-site) for repair or maintenance, the duration and causes of any technology downtime or data acquisition failure, quantity and hazardous nature of any waste generated, how to safely dispose of such waste, operator observations about technology ease of use, clarity of the vendor's instruction manual, user-friendliness of any needed software, overall convenience of the technologies and accessories/consumables, and the number of samples that could be processed per hour or per day. These observations will be summarized to aid in describing the technology performance in the verification report on each technology.

B1.1.9 Field Portability

Testing the operation of the technologies in a field setting is a key component of the verification test. Evaluating the performance of each field-portable technology while being used outside the laboratory without the availability of miscellaneous laboratory supplies is important to the buyers and users of these technologies. Technologies will be evaluated in a field setting only if the vendor states that the technology has that capability. For those technologies that are meant to be field-portable, this parameter will be assessed by transporting the technology to a non-laboratory location. In addition to traditional field settings, non-laboratory areas could include warehouses, shipping/receiving areas, storerooms, courtyards, and/or parking lots provided the location meets the criteria that the area is absent of laboratory amenities such as laboratory bench space, power, lighting, temperature control, storage and refrigeration, etc. as would be the case in a traditional field setting. Ideally all of the samples included in the lab-based tests would be repeated in the field; however, at a minimum one performance test sample or environmental sample that had a strong response in the lab-based tests will be analyzed in triplicate in the field. Results obtained in the field will be compared with the results for the same sample obtained in the laboratory by the same technician where possible, or at a minimum by a technician with equivalent training and experience operating the technology to the technician who performed the laboratory analysis. Technical staff will also record observations related to field portability such as requirements for power, space, and ease of use in and transport to a non-laboratory setting.

B1.2 Statistical Analysis

The statistical methods and calculations used for evaluation of the quantitative performance parameters are described in the following sections.

B.1.2.1 Endpoint

Each technology produces its own unique endpoint derived from the inhibition data gathered when analyzing various concentrations of contaminants in soil (e.g., EC_{50} values). For

each technology, these data will be documented and presented with respect to each contaminant and concentration level using the appropriate endpoint for the technology.

B1.2.2 Precision

The standard deviation (*S*) of the results for the replicate analyses of the same sample will be calculated as follows.

$$S = \left[\frac{1}{n-1} \sum_{k=1}^{n} \left(M_k - \overline{M} \right)^2 \right]^{1/2} \quad (1)$$

where n is the number of replicate samples, M_k is the endpoint measurement for the k^{th} sample, and M is the average endpoint measurement of the replicate samples. The technology precision for each sample will be reported in terms of RSD, which will be calculated as follows.

$$RSD(\%) = \left| \frac{S}{\overline{M}} \right| \times 100 \quad (2)$$

The average (M), standard deviation (S) and relative standard deviation (RSD) values for each analyte at each concentration will be listed in data tables in the verification report; however, verification statements and performance summary tables in the verification report will list the range of RSDs obtained for all concentrations of each contaminant tested.

B1.2.3 False Negative Rate

Results will be considered false negative only when a technology is exposed to a contaminant concentration greater than the desired remediation or screening level and the technology does not indicate inhibition greater than the negative control. The rate of false negatives, expressed as a percentage of total samples analyzed for each contaminant, will be calculated by dividing the number of false negative measurements (M_{fn}) by the total number of measurements included in verification testing (M_{total}).

$$FalseNegative(\%) = \frac{M_{fn}}{M_{total}} \times 100 \quad (3)$$

B1.2.4 False Positive Rate

Results will be considered false positive only when an unspiked sample produces inhibition greater than that of the negative control. The rate of false positives, expressed as a percentage of total samples analyzed for each contaminant, will be calculated by dividing the number of false positive measurements (M_{fp}) by the total number of measurements included in verification testing (M_{total}).

$$FalsePositive(\%) = \frac{M_{fp}}{M_{total}} \times 100 \quad (4)$$

B1.2.5 Sensitivity

The sensitivity of the technology for detecting various contaminants in performance test samples (i.e., sand spiked with contaminant) will be assessed. The exact procedures used to assess sensitivity will be detailed in each TQAP developed from this protocol. Sensitivity assessments that could be used are an evaluation of the lowest tested concentration of each contaminant where the average inhibition plus or minus the standard deviation does not overlap with the average inhibition plus or minus the standard deviation of the negative control or determination of the LOEC and NOEC values along with an associated MSD value calculated using an appropriate parametric multiple-comparison method (e.g., Dunnett's test). The sensitivity of the reference method will not be assessed other than it must meet the DQI requirements (Table 2).

B1.2.6 Matrix Effects

The technology's ability to detect each contaminant in the environmental samples will be compared with the technology's lowest detectable contaminant level determined by spiking the contaminant into an inert matrix (i.e., sand) as described in Section B1.2.5. If the contaminant concentration in the environmental sample (measured using reference methods described in Section B4) is above the lowest detectable level in an inert matrix (as determined in Section B1.2.5), but the technology result for the environmental sample is negative, matrix effects will be considered to have contributed to this false negative response. It should be noted that rapid

toxicity technologies are intended to respond to all toxic compounds and not just contaminants of interest in this verification test. As broad screens of toxicity, these technologies may also be susceptible to toxicity potentiation and antagonism. Therefore, the matrix effect results reported should consider the possibility of such toxicity effects and include discussion of such effects in the verification report so that readers can understand matrices where the toxic response may be affected. The number of environmental samples where matrix effects affected results (M_{matrix}) out of the total number of environmental samples tested (M_{total}) will be reported as a percentage using Equation 5.

$$MatrixEffect(\%) = \frac{M_{matrix}}{M_{total}} \times 100 \quad (5)$$

B1.2.7 Data Completeness

Data completeness will be calculated as the percentage of the total possible data by dividing the number of valid data measurements generated by each technology (M_{valid}) by the total number of data measurements included in verification testing (M_{total}).

$$Completeness(\%) = \frac{M_{valid}}{M_{total}} \times 100 \quad (6)$$

The cause of any substantial loss of data will be established from operator observations or technology records and noted in the discussion of the data completeness results.

B1.2.8 Operational Factors

There are no statistical calculations applicable to operational factors. Operational factors will be determined based on documented observations of the technical staff.

B1.2.9 Field Portability

The results obtained from the measurements made on samples in the laboratory and field setting will be compiled independently for each technology and compared to assess the accuracy of the measurements under the different analysis conditions. Means and standard deviations of

the endpoints generated in both locations will be compared and assessed for whether they are statistically different.

B1.3 Reporting

The data obtained in the verification test will be compiled separately for each vendor's technology, and the data evaluations will be applied to each technology's data set without reference to any other. At no time will data from different vendors' technologies be intercompared or ranked. Following completion of the data evaluations, a draft verification report and verification statement will be prepared for each vendor's technology, stating the verification test procedures and documenting the performance observed. For example, descriptions of the data acquisition procedures, use of vendor-supplied proprietary software, consumables used, repairs and maintenance needed, and the nature of any problems will be presented in the draft report. Each report will briefly describe the verification program(s), the verification organization(s), and the procedures used in verification testing. The results of the verification test will be stated quantitatively, without comparison to any other technology tested or comment on the acceptability of the technology's performance. Each draft verification report will be submitted for review by the respective technology vendor, by the verification program(s), and peer reviewers. Comments on the draft report will be addressed in revisions of the report. The peer review comments and responses will be tabulated to document the peer review process. The reporting and review process will be conducted according to the quality procedures set forth by the verification program(s) and the verification organization(s).

B2 SAMPLING REQUIREMENTS

B2.1 Sample Collection, Storage, and Shipment

Environmental samples will be collected for use in a verification test following the TQAP. As much as possible, samples will be obtained from known contaminated sites using the same sampling techniques that are in place at the site for the site evaluation process. Samples may be collected in bulk and shipped to the test facility in plastic buckets or other suitable containers. Shipments will be via a trackable overnight delivery service to the test facility

sample custodian. Samples will be stored refrigerated or frozen as is appropriate for the contaminants expected to be contained in the soil. Environmental samples will be dried (e.g., oven dried using low heat with specifics to be detailed in the TQAP) and homogenized prior to use in testing to ensure that sample heterogeneity is a minimal factor in testing multiple technologies. Because of the sample handling involved, the environmental samples will be homogenized before concentrations of contaminants are measured using the reference methods. Appropriate soil characteristics such as total organic carbon, grain size distribution, and pH should be measured once the environmental samples have been dried and homogenized. Information about the soil characteristics may aid in understanding differences in the various environmental samples that will be tested.

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Sample custody will be documented throughout collection, transport, shipping (if necessary), and analysis using standard COC forms provided by the verification organization or supplied by the reference laboratory, as appropriate. Samples transferred within the verification organization may be documented with internal COC forms. Each COC form will summarize the samples collected and analyses requested. The COC forms will track sample release from the sampling location to the test facility and/or reference laboratory; or release directly from the test facility to the reference laboratory. Each COC form will be signed by the person relinquishing the samples once that person has verified that the COC form is accurate. The original sample COC forms will accompany the samples; the shipper will keep a copy. Upon receipt at the test facility and/or reference laboratory, COC forms will be signed by the person receiving the samples once that person has verified that all samples identified on the COC forms are present. Any discrepancies will be noted on the form; and the sample receiver will immediately contact the verification organization to report missing, broken, or compromised samples. Copies of all COC forms will be delivered to the verification organization and maintained with the test records.

B4 LABORATORY REFERENCE METHODS

Table 5 lists the analytical methods that can be used to determine or measure the concentration of contaminants analyzed during verification tests performed following this protocol. Additional methods may be used provided they are appropriate for the contaminant and matrix and are documented in the TQAP for the verification test.

B5 QUALITY CONTROL

Steps will be taken to maintain the quality of data collected during verification tests conducted under this protocol. This will include analyzing specific quality control samples (QCS) at a regular frequency by the technologies undergoing verification. The QCSs will include negative controls, positive controls, and calibration checks. Negative control samples, consisting of unspiked experimental matrix, will help ensure that no sources of contamination are introduced in the sample handling and analysis procedures. The positive control and calibration check samples, specified by each vendor, will indicate to the technical staff whether or not the technology is functioning properly. The vendor will provide the approximate endpoint that should result with their technology upon analysis of the positive control and calibration check. QCSs producing results that do not meet the anticipated results specified by the vendor will be

Table 5. Example Contaminant Compound Confirmatory Methods

Example Contaminant	Method
Trichloroethylene, toluene	SW-846 8260B[(2)]
Aldicarb	EPA 531.1[(3)]
Dicrotophos	SW-846 8141A[(4)]
Arsenic, lead, mercury, cadmium	EPA 200.8[(5)]
PCBs (as Aroclor 1254)	SW-846 8270C[(6)]
2,3,7,8-TCDD	EPA 1613B[(7)]
Benzo[a]pyrene	SW-846 8270C[(6)]

reanalyzed and corrective action taken if needed to ensure that test sample results are not affected. Corrective actions may include reanalyzing samples to verify that the technology has been operated properly, conducting maintenance, or recalibrating. Positive and negative controls will be analyzed at a frequency of approximately 5% based on the total number of test samples. Calibration checks will be analyzed according to guidance provided by each technology vendor.

As described in Section B4, the reference laboratory will follow standard reference methods for determining the toxins evaluated during verification tests conducted under this protocol. All reference measurements will be expected to meet the reference method QC requirements (such as those listed in Table 2) or, in absence of specific requirements in the reference method, the reference laboratory's standard requirements for QC samples.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

The equipment used by the test facility and/or reference laboratory will be tested, inspected, and maintained as per the SOPs of the test facility and/or reference laboratory and/or the manufacturer's recommendations so as to meet the performance requirements established in the TQAP. When technical staff operate and maintain technologies undergoing testing, they will follow directions provided by the technology vendor. Otherwise, operation and maintenance of the technologies will be the responsibility of the technology vendor.

B7 CALIBRATION/VERIFICATION OF TEST PROCEDURES

Systems used for reference analyses will be calibrated as appropriate before any reference samples are analyzed and recalibrated as needed based on the reference methods and/or reference laboratory SOPs.

Technologies undergoing testing will be calibrated initially by the respective technology vendor prior to shipping the technology to the test facility, or during training, and will be recalibrated according to direction from the vendor. Calibration checks will be performed upon direction of the vendor. In the event that recalibration is necessary, the recalibration will be carried out by the technology vendor or by technical staff under the direction of the vendor. All calibrations will be documented as appropriate by the technical staff or vendor.

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

All materials, supplies, and consumables will be ordered by the verification organization, unless otherwise donated from test collaborators. Where possible, the verification organization will rely on sources of materials and consumables that have been used previously as part of verification testing without problems. The verification organization will also rely on previous experience or recommendations from the verification program(s), stakeholders, test collaborators, subcontractors, or technology vendors. Where possible, materials or supplies will be traceable to the National Institute of Standards and Technology (NIST). Upon receipt of any supplies or consumables, the verification organization will visually inspect and ensure that the materials received are those that were ordered and that there are no visual signs of damage that could compromise the suitability of the materials. Certificates of analysis (COA) or other documentation of analytical purity will be checked for all reagents and standards to ensure suitability for the verification test and will be included with the test files. If damaged, unsuitable, or inappropriate goods are received, they will be returned or disposed of, and arrangements will be made to receive replacement materials.

B9 NON-DIRECT MEASUREMENTS

No non-direct measurements will be used during this verification test.

B10 DATA MANAGEMENT

Various types of data will be acquired and recorded electronically or manually by the verification organization, vendor, test collaborator, and/or subcontractor staff during the verification test. Table 3 summarizes the types of data to be recorded. All maintenance activities, repairs, calibrations, and operator observations relevant to the operation of the technologies will be documented by technical staff in LRBs or on data sheets. Results from the reference methods, including raw data, analyses, and final results, will be compiled by the reference laboratory, preferably in electronic format, and submitted to the verification organization at the conclusion of reference method testing.

Records received or generated by any technical staff during the verification test will be reviewed by a verification organization staff member within two weeks of generation or receipt, before the records are used to calculate, evaluate, or report verification results. This review will be performed by a verification organization technical staff member involved in the verification test, but not the staff member who originally generated the record. The review will be documented by the person performing the review by adding his/her initials and date to the hard copy of the record being reviewed. In addition, any calculations performed by technical staff will be spot-checked by the verification organization QA and/or technical staff to ensure that calculations are performed correctly. Calculations to be checked include any statistical calculations described in this protocol. The data obtained from this verification test will be compiled and reported independently for each technology. Results for technologies from different vendors will not be compared with each other.

Among the QA activities conducted by verification organization QA staff will be an audit of data quality. This audit will consist of a review by the verification organization Quality Manager of at least 10% of the test data. The results of this audit will be compiled in an assessment report. During the course of any such audit, the verification organization Quality Manager will inform the technical staff of any findings and any need for immediate corrective action. If serious data quality problems exist, the verification organization Quality Manager will request that the verification organization Program Manager issue a stop work order. Once the assessment report has been prepared, the verification organization will ensure that a response is provided for each adverse finding or potential problem, and will implement any necessary follow-up corrective action. The verification organization Quality Manager will ensure that follow-up corrective action has been taken.

SECTION C
ASSESSMENT AND OVERSIGHT

C1 ASSESSMENTS AND RESPONSE ACTIONS

Every effort will be made in verification tests conducted under this protocol to anticipate and resolve potential problems before the quality of performance is compromised. One of the major objectives of this protocol is to establish mechanisms necessary to ensure this. Internal QC

measures described in this protocol, which is peer reviewed by a panel of outside experts, will be implemented by the technical staff; these QC measures will give information on data quality on a day-to-day basis. The responsibility for interpreting the results of these checks and resolving any potential problems resides with the verification organization. Technical staff have the responsibility to identify problems that could affect data quality or the ability to use the data. Technical staff will work with the verification organization Quality Manager to resolve any problems that are identified. Action will be taken to control the problem, identify a solution to the problem, minimize losses and correct data, where possible. Independent of any verification program QA activities, the verification organization will be responsible for ensuring that the audits described in the following sections are conducted as part of this verification test.

C1.1 Performance Evaluation Audits

A performance evaluation (PE) audit will be conducted to assess the quality of the reference method measurements made in this verification test. The PE audit of the reference methods will be performed by supplying each reference method a blind sample or standard reference material containing the toxins of interest. The PE audit samples will be analyzed in the same manner as all other samples, and the analytical results for the PE audit samples will be compared with the nominal concentration or certified value. The target criterion for this PE audit is agreement of the analytical result within 25% of the nominal concentration [by percent difference (PD)] or within 25% of the certified value (by PD). If the PE audit results do not meet the tolerances shown, they will be repeated. If the outlying results persist, a change in reference instrument and a repeat of the PE audit may be considered. This audit will be performed once prior to the start of the test and will be the responsibility of the verification organization Quality Manager or designee.

C1.2 Technical Systems Audits

The verification organization Quality Manager or designee will perform a TSA at least once during verification tests conducted under this protocol. The purpose of this audit is to ensure that the verification test is being performed in accordance with the quality plans in place at the verification organization, this protocol, published reference methods, and any SOPs used

by the reference laboratory. In the TSA, the verification organization Quality Manager, or a designee, may review the reference methods used, compare actual test procedures to those specified or referenced in this protocol, and review data acquisition and handling procedures. In the TSA, the verification organization Quality Manager will tour the test facility, observe sample collection if appropriate, inspect documentation of sample COC, and review technology-specific records. He or she will also check standard certifications and technology data acquisition procedures and may confer with the technology vendors, reference laboratory, and technical staff. The verification organization Quality Manager may also visit the reference laboratory to review procedures and adherence to this plan and applicable SOPs. A TSA report will be prepared, including a statement of findings and the actions taken to address any adverse findings. The verification program Quality Manager will receive a copy of the verification organization's TSA report. At the verification program's discretion, verification program QA staff may also conduct an independent on-site TSA during the verification test. The TSA findings will be communicated to technical staff at the time of the audit and documented in a TSA report.

C1.3 Data Quality Audits

The verification organization Quality Manager or designee will audit at least 10% of the verification data acquired in the verification test. The verification organization Quality Manager will trace the data from initial acquisition, through reduction and statistical comparisons, to final reporting. All calculations performed on the data undergoing the audit will be checked.

C1.4 QA/QC Reporting

Each assessment and audit will be documented and submitted in accordance with the verification organization's quality management plan. The results of the TSA will be submitted to the verification program. Assessment reports will include the following:

- Identification of any adverse findings or potential problems
- Response to adverse findings or potential problems
- Recommendations for resolving problems
- Confirmation that solutions have been implemented and are effective
- Citation of any noteworthy practices that may be of use to others.

C2 REPORTS TO MANAGEMENT

The verification organization Quality Manager, during the course of any assessment or audit, will identify to the technical staff performing experimental activities any immediate corrective action that should be taken. If serious quality problems exist, the verification organization Quality Manager is authorized to request that the verification organization Program Manager issue a stop work order. Once the assessment report has been prepared, the verification organization will ensure that a response is provided for each adverse finding or potential problem and will implement any necessary follow-up corrective action. The verification organization's Quality Manager will ensure that follow-up corrective action has been taken. This protocol, any TQAPs based on this protocol, and final verification reports are reviewed by the verification organization's QA staff and the verification organization program management staff. Upon final review and approval, both documents may be posted on the verification organization's and verification program's web site, if applicable.

SECTION D
DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

The key data review requirements for the verification test are stated in Section B10 of this protocol. In general, the data review requirements specify that the data generated during this test will be reviewed by a verification organization technical staff member within two weeks of generation of the data. The reviewer will be familiar with the technical aspects of the verification test, but will not be the person who generated the data. This process will serve both as the data review and the data verification and will ensure that the data have been recorded, transmitted, and processed properly. Furthermore, this process will ensure that the soil toxicity detecting technology data and the reference method data were collected under appropriate testing conditions and that the reference method data meet the specifications of the reference method.

The data validation requirements for this test involve an assessment of the data quality relative to the DQIs and audit acceptance criteria specified for this test. The DQIs listed in Section B5 will be used to validate the quality of the data. The QA audits described within Section C of this document, including the PE audit and audit of data quality, are designed to validate the quality of the data.

D2 VALIDATION AND VERIFICATION METHODS

Data verification is conducted as part of the data review, as described in Section B10 of this protocol. A visual inspection of handwritten data will be conducted to ensure that all entries were properly recorded or transcribed and that any erroneous entries were properly noted (i.e., single line through the entry with an explanation of the error and the initials of the recorder and date of entry). Electronic data from the technologies and other instruments used during the test will be inspected to ensure proper transfer from the datalogging system. Data manually incorporated into spreadsheets for use in calculations will be checked against handwritten data to ensure that transcription errors have not occurred. All calculations used to transform the data will be reviewed to ensure the accuracy and the appropriateness of the calculations. Calculations performed manually will be reviewed and repeated using a handheld calculator or commercial

software (e.g., Excel). Calculations performed using standard commercial office software (e.g., Excel) will be reviewed by inspecting the equations used in calculations and verifying selected calculations by handheld calculator. Calculations performed using specialized commercial software (i.e., for analytical instrumentation) will be reviewed by inspecting and, when feasible, verifying by handheld calculator or standard commercial office software.

To ensure that the data generated from this test meet the goals of the test, a number of data validation procedures will be performed. Section C of this protocol describes the validation safeguards employed for this verification test. Data validation and verification efforts include the completion of QC activities and the performance of TSA and PE audits as described in Section C. The data from this test will be evaluated relative to the measurement DQIs described in Section B5, and the PE audit acceptance criteria given in Section C1.1 of this protocol. Data failing to meet these criteria will be flagged in the data set and not used for evaluation of the technologies, unless these deviations are accompanied by descriptions that adequately demonstrate that data quality was not compromised.

An audit of data quality will be conducted by the verification organization's Quality Manager to ensure that data review, verification, and validation procedures were completed and to assure the overall data quality. The schedule for completing TSA, PE and audits of data quality are included in Table 1.

D3 RECONCILIATION WITH USER REQUIREMENTS

The purpose of a verification test performed following this protocol is to evaluate the performance of commercial technologies for detecting toxicity in soil. In part, this evaluation will include comparisons of the results from the technologies to results from established analytical reference methods. To meet the requirements of the user community, the data obtained in such a verification test should include thorough documentation of the performance of the technologies during the verification test. The data review, verification, and validation procedures described above will ensure that verification test data meet these requirements and are accurately presented in the verification reports generated from the test and that data not meeting these requirements are appropriately flagged and discussed in the verification reports. Additionally, all data generated using reference methods that are used to evaluate technology results during the verification test should meet the QA requirements of the reference methods.

This joint verification protocol and any resulting verification report(s) generated following procedures described in this protocol will be reviewed by participating technology vendors, verification organization staff, test collaborators, the verification program(s), and external expert peer reviewers. These reviews will ensure that this protocol, verification test(s) of technologies for detecting toxicity in soil, and the resulting report(s) meet the needs of potential users and regulators. The final report(s) will be submitted to the verification program(s) in Microsoft Word and in 508 compliant Adobe Portable Document Format (pdf). If applicable, the final report subsequently may be posted on the verification organization's and verification program's web site.

SECTION E

REFERENCES

E1 REFERENCES

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4. SW-846 Method 8141A, "Organophosphorous Compounds by Gas Chromatography: Capillary Column Technique," Revision 1, September 1994.

5. EPA Method 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively-Coupled Plasma Mass Spectrometry," Revision 5.4, 1994.

6. SW-846 Method 8270C, "Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)," Revision 3, 1996.

7. EPA Method 1613, "Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS," Revision B, 1994.