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Environmental Technology Verification Program

Advanced Monitoring Systems Center

Test/QA Plan for Verification of Enzymatic Test Kits

TEST/QA PLAN

for

Verification of Enzymatic Test Kits

September 21, 2005

Prepared by

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ETV Advanced Monitoring Systems Center

Test/QA Plan for Verification of Enzymatic Test Kits

Version 1

September 21, 2005

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SECTION A

PROJECT MANAGEMENT

A4 VERIFICATION TEST ORGANIZATION

The verification test will be conducted under the auspices of the U.S. Environmental Protection Agency (EPA) through the Environmental Technology Verification (ETV) Program. It will be performed by Battelle, which is managing the ETV Advanced Monitoring Systems (AMS) Center through a cooperative agreement with EPA. The scope of the AMS Center covers verification of monitoring technologies for contaminants and natural species in air, water, and soil.

The day to day operations of this verification test will be coordinated and supervised by Battelle personnel, with the participation of the vendors who will be having the performance of their enzymatic test kits verified. The testing will be conducted at Battelle in Columbus and West Jefferson, Ohio. Battelle staff will operate the test kits during the verification test. Each vendor will provide sufficient supplies to allow for completion of the test (as described in this document) and training to Battelle staff on the use of the enzymatic test kit. Quality assurance (QA) oversight will be provided by the Battelle Quality Manager and the EPA AMS Center Quality Manager at his/her discretion. The organization chart in Figure 1 identifies the responsibilities of the organizations and individuals associated with the verification test. Roles and responsibilities are defined further below.

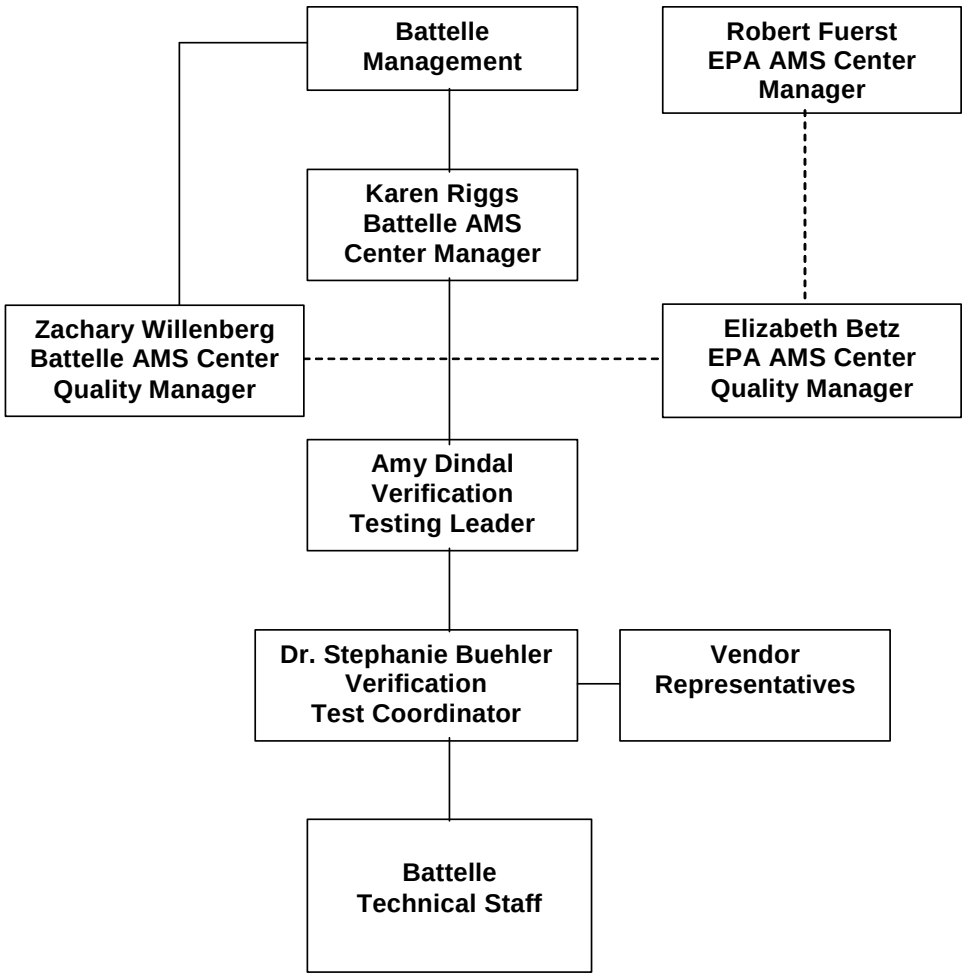

Figure 1. Organization Chart

A4.1 Battelle

Dr. Stephanie Buehler is the AMS Center's Verification Test Coordinator for this test. In this role, Dr. Buehler will have overall responsibility for ensuring that the technical, schedule, and cost goals established for the verification test are met. Specifically, Dr. Buehler will:

- Assemble a team of qualified technical staff to conduct the verification test.
- Direct the team in performing the verification test in accordance with the test/QA plan.
- Ensure that all quality procedures specified in the test/QA plan and in the AMS Center Quality Management Plan[1] (QMP) are followed.
- Prepare the draft and final test/QA plan, verification reports, and verification statements.
- Revise the draft test/QA plan, verification reports, and verification statements in response to reviewers' comments.
- Respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary.
- Serve as the primary point of contact for vendor representatives.
- Coordinate distribution of the final test/QA plan, verification reports, and verification statements.
- Establish a budget for the verification test and manage staff to ensure the budget is not exceeded.
- Ensure that confidentiality of sensitive vendor information is maintained.

Ms. Amy Dindal is a Verification Testing Leader for the AMS Center. Ms. Dindal will provide technical guidance and oversee the various stages of verification testing. She will

- Support Dr. Buehler in preparing the test/QA plan and organizing the testing.
- Review the draft and final test/QA plan.
- Review the draft and final verification reports and verification statements.
- Support Dr. Buehler in responding to any issues raised in assessment reports and audits.

Ms. Karen Riggs is Battelle's manager for the AMS Center. Ms. Riggs will

- Review the draft and final test/QA plan.
- Review the draft and final verification reports and verification statements.
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the verification test.
- Ensure that confidentiality of sensitive vendor information is maintained.
- Maintain communication with EPA's project and quality managers.
- Facilitate a stop work order if Battelle or EPA QA staff discovers adverse findings that will compromise test results.

Battelle Technical Staff will conduct the testing of the enzymatic test kits during the verification test. The responsibilities of the technical staff will be to:

- Assist in the collection, receipt, and storage of drinking water samples.
- Maintain and operate the test kits after proper training is provided by the vendor.
- Perform the verification testing as described in the test/QA plan, including the preparation of all test samples.
- Make qualitative observations about the operation of the enzymatic test kits.
- Record the results for each enzymatic test kit and transmit them to the Verification Test Coordinator on a daily basis.
- Perform reference method measurements indicated in this test/QA plan.
- Troubleshoot any problems with the enzymatic test kits and communicate them to the Verification Test Coordinator immediately.

Mr. Zachary Willenberg is Battelle's Quality Manager for the AMS Center. Mr. Willenberg will:

- Review the draft and final test/QA plan.
- Conduct a technical systems audit at least once during the verification test, or designate other QA staff to conduct the audit.
- Audit at least 10% of the verification data.
- Prepare and distribute an assessment report for each audit.

- Verify implementation of any necessary corrective action.
- Notify Battelle's AMS Center Manager to issue a stop work order if self audits indicate that data quality is being compromised.
- Provide a summary of the QA/QC activities and results for the verification reports.
- Review the draft and final verification reports and verification statements.
- Assume overall responsibility for ensuring that the test/QA plan is followed.

A4.2 Vendors

The responsibilities of the vendor representatives are as follows:

- Review the draft test/QA plan.
- Approve the test/QA plan prior to test initiation.
- Provide enough off-the-shelf enzymatic test kits for evaluation of all test samples during the verification test.
- Provide all other equipment/supplies/reagents/consumables needed to operate their test kits for the duration of the verification test.
- Provide training to Battelle staff in the operation of their enzymatic test kit.
- Provide written instructions for operation of enzymatic test kits.
- Review and provide comments on the draft verification report and statement.

A4.3 EPA

EPA's responsibilities in the AMS Center are based on the requirements stated in the "Environmental Technology Verification Program Quality Management Plan" (EPA QMP)[2]. The roles of the specific EPA staff are as follows:

Ms. Elizabeth Betz is EPA's AMS Center Quality Manager. For the verification test, Ms. Betz will:

- Review the draft test/QA plan.
- Perform at her option one external technical system audit during the verification test.
- Notify the EPA AMS Center Manager of the need for a stop work order if external audit indicates that data quality is being compromised.

- Prepare and distribute an assessment report summarizing results of external audit.
- Review draft verification reports and statements.

Mr. Robert Fuerst is EPA's manager for the AMS Center. Mr. Fuerst will:

- Review the draft test/QA plan.
- Approve the final test/QA plan.
- Review the draft verification reports and statements.
- Oversee the EPA review process for the verification reports and statements.
- Coordinate the submission of verification reports and statements for final EPA approval.
- Contact the AMS Center Manager to issue a stop work order if an EPA assessment indicates that data quality is being compromised.

A4.4 Subcontract Laboratory

Any laboratory providing reference measurements will follow the requirements of the reference methods as well as the QC requirements as stated in this test/QA plan. A subcontract laboratory will provide reference measurements for the drinking water characterization as well as confirmation of the interferent solution concentrations. The responsibilities of this laboratory will include:

- Proper receipt and handling of sample material.
- Accurate measurement of the target analyte(s) or target parameter(s).
- Submission of data and any supporting documents to Battelle.
- Participation in audit by Battelle AMS Quality Manager or EPA AMS Quality Manager, if requested.

A5 BACKGROUND

The ETV Program's AMS Center does third-party verification testing of commercially available technologies that detect natural species and contaminants in air, water, and soil. A stakeholder committee of buyers and users of such technologies recommend the technology

categories and technologies within those categories as priorities for testing. Enzymatic test kits for detection of chemical agents were identified as a priority technology category through the AMS Center stakeholder process.

A6 VERIFICATION TEST DESCRIPTION AND SCHEDULE

A6.1 Summary of Technology Category

Enzymatic test kits are qualitative technologies that can detect the presence of chemical agents, carbamate pesticides, and/or organophosphate (OP) pesticides. These kits are generally designed to be handheld and portable. This verification test will assess the ability of the enzymatic test kits to detect these compounds in water.

Enzymatic test kits generally rely on the presence or absence of color or a color change to indicate that a chemical agent, carbamate pesticide, or OP pesticide has been detected. These test kits cannot determine the specific contaminant that is present, nor can they determine the amount of contaminant(s) present. However, some technologies can indicate the concentrations range of chemical agent, carbamate pesticide, or OP pesticide that is present (e.g., high, medium, or low) through a sliding color scale.

To detect chemical agents, carbamate pesticides, and OP pesticides, enzymatic test kits rely on the reaction of the cholinesterase enzyme. Under regular conditions, the enzyme reacts normally with other chemicals that might be present in the test kit. When a chemical agent, carbamate pesticide, or OP pesticide is present, the activity of the enzyme is inhibited by these compounds. The effects of this inhibition will then generally lead to a color change in the test kit, indicating the presence of a chemical agent, carbamate pesticide, or OP pesticide. For some kits, a color change may indicate the absence of the contaminant.

This verification test will assess the performance of each enzymatic test kit relative to key verification parameters including accuracy, precision, and false positive and negative rates in detecting selected chemical agents, carbamate pesticide and OP pesticide in American Society of Testing and Materials (ASTM) Type II deionized (DI) water, in the presence of possible interferents added to ASTM Type II DI water, and in drinking water obtained from a variety of geographically dispersed U.S. water utilities that use various water treatment processes.

Qualitative characteristics of each enzymatic test kit, such as ease of use, sample throughput, and cost, will also be assessed and reported. While most of the testing will occur in a laboratory, the enzymatic test kits that are designed for field use will be tested outside of the laboratory. In performing the verification test, Battelle will follow the technical and QA procedures specified in this test/QA plan and will comply with the quality requirements in the AMS Center QMP[1].

A6.2 Verification Schedule

The verification test of enzymatic test kits will begin in September 2005 and last through October 2005. At the close of testing, individual reports will be drafted for each technology, reviewed, and submitted to EPA for final signature. All reports will be submitted to EPA in electronic (Word and Adobe portable document format [pdf]) and hard copy formats.

A6.3 Test Facility

The verification test will take place at Battelle facilities in Columbus and West Jefferson, Ohio. A portion of the test (field portability) will also be conducted in a non-laboratory environment. Testing with chemical agents will take place at the Hazardous Materials Research Facility (HMRC). The HMRC, located in the North area of Battelle's West Jefferson Campus, is an ISO 9001 certified facility. The HMRC and its personnel have demonstrated the capability for storing and safely handling chemical agents, Class A poisons, toxins, agent simulants, and other hazardous materials.

The HMRC research laboratories meet or exceed all requirements for the safe use, storage, decontamination, and accountability of chemical agents as defined by Army Regulation 50-6 (Chemical Surety).[3] Operations withing the laboratories always are conducted in accordance with Battelle's Chemical Safety Information Program.[4] Any non-disposable equipment that is part of a particular technology and that comes into contact with chemical agent(s) will not be able to be returned to the vendor because of restrictions on the release of such equipment.[5]

The pesticides to be evaluated in this test are not required to be used in a specially designed facility as are the chemical agents. Thus, testing of these chemicals will take place in qualified laboratories in the Battelle Columbus Operations facilities.

A7 QUALITY OBJECTIVES

This verification test will evaluate the performance of enzymatic test kits for the identification of chemical agents, carbamate pesticides, and OP pesticides in water. Only qualitative results (e.g., presence/absence of contaminant at specified levels) will be considered for each technology. In the instance where semi-quantitative (e.g., percent inhibition) measures are used in determining the results for a particular technology, a qualitative result will be reported (i.e., presence or absence of the contaminant of interest) as with the other technologies, and the semi-quantitative measure used to determine that result will also be reported for that sample but will not be used in any other data analyses as described in Section B1.2.

Method blank and positive and negative control samples will be included as QC samples for each technology. The quality of the reference measurements will be monitored using QC samples and procedures, as described in the testing laboratory's procedures or the method. These requirements are further discussed in Section B. Performance evaluation audit samples will also be used to confirm the accuracy of the reference solution concentrations (see Section C1.1).

The Battelle Quality Manager or his designee will perform a technical systems audit (TSA) at least once during this verification test and will audit at least 10% of the verification data acquired. The EPA AMS Center Quality Manager also may conduct an independent TSA, at her discretion.

A8 SPECIAL TRAINING/CERTIFICATION

Battelle staff will have appropriate training and experience. Documentation of training is maintained for all Battelle technical staff in training files at their respective locations. Chemical warfare agent testing will be conducted by staff with the appropriate training that will be documented in their training record. Any use of respirators will be conducted by staff with the appropriate training. The Battelle Quality Manager will verify the presence of appropriate training records prior to the start of testing. The technology vendor will be required to train Battelle technical staff in the operation of his/her technology prior to the start of testing. Battelle will document this training with a consent form, signed by the vendor, that states which specific Battelle staff have been trained to use the enzymatic test kit.

A9 DOCUMENTATION AND RECORDS

The records for this verification test include the test/QA plan, the protocols, chain of custody forms, laboratory record books (LRB), data collection forms, electronic files (both raw data and spreadsheets), and the final verification report and statement. All of these records will be maintained in the Verification Test Coordinator's office or at the testing locations during the test and will be transferred to permanent storage at Battelle's Records Management Office at the conclusion of the verification test. All Battelle LRBs are stored indefinitely, either by the Verification Test Coordinator or Battelle's Records Management Office. EPA will be notified before disposal of any files. The results from the reference measurements made by the Battelle or subcontractor laboratory will be submitted to Battelle after making the measurement and obtaining the results of the analyses. Section B10 further details the data recording practices and responsibilities. QA documents generated over the course of this verification test include audit and assessment reports and will be maintained by the Battelle Quality Manager.

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 the raw data from each vendor's technology, and separation of data from different technologies, will be maintained throughout the verification test. Separate files (including manual records, printouts, and/or electronic data files) will be kept for each instrument.

SECTION B
MEASUREMENT AND DATA ACQUISITION

B1 EXPERIMENTAL DESIGN

This verification test will specifically address verification of enzymatic test kits for the detection of GB, GD, and VX (chemical agents); aldicarb (carbamate pesticide); and dicrotophos (OP pesticide) in drinking water. The contaminants were selected based on the capabilities of the technologies being tested and the importance of these contaminants. The enzymatic test kits participating in this test will be evaluated on qualitative results (as noted in Section A7), indicating only the presence or absence of the contaminants within a specified concentration interval (see Section B1.1). The performance of the enzymatic test kits will be verified based on the parameters identified in Table 1. These parameters are discussed in detail in Section B1.2.

Table 1. Verification Test Performance Parameters

Performance Parameter	Method of Evaluation
Accuracy	Frequency of positive responses in the presence of nominal concentrations of each contaminant in ASTM Type II DI water
Precision	Determined by repeatability of response from triplicate measurements of each test sample
False Positive/Negative Rates	Determined by response in the presence or absence of a contaminant in various matrices
Potential Matrix and Interference Effects	Evaluation of performance in various matrices and in the presence of potential interferents
Portability	Evaluation of operational factors obtained in a non-laboratory setting
Operational Factors	Observational factors such as operator observations, ease of use, and sample throughput

B1.1 Test Procedures

The following sections describe the test procedures that will be used to evaluate each of the enzymatic test kit performance parameters listed in Table 1. Performance testing will be conducted on each kit separately, and will follow the operational procedures specified by the vendor, including analysis of quality control (QC) samples.

This verification test will determine the performance capabilities of enzymatic test kits to detect GB, GD, VX, aldicarb, and dicrotophos in three types of samples—performance test (PT), drinking water (DW), and QC. Each sample type is described in detail in the following sections. Contaminants will be tested individually on each technology. Stock solutions of each contaminant will be prepared in the ASTM Type II DI water or appropriate reagents from certified standards. Samples will then be prepared in the appropriate matrix using these stock solutions. Samples will be prepared and analyzed on the same day, with samples being prepared as close in time to their use with a test kit as possible. To minimize the loss of analytes to hydrolysis, contaminant stock solutions prepared in DI water will be made on a daily basis unless the stability of the analyte in water has been previously demonstrated. Aliquots of each stock solution will be diluted to the appropriate concentration using volumetric glassware and volumetric or calibrated pipettes. In some cases, reference solutions will be prepared in ASTM Type II DI water using the stock solutions to determine their concentration; otherwise, the concentration of the stock solutions will be confirmed. Reference methods for confirming solution concentrations are discussed in Section B4 (see Tables 4 and 5). All DW samples will be dechlorinated prior to their use to minimize the hydrolysis of the contaminants. The DW samples will also be characterized for typical water quality parameters (see Sections B1.1.2 and B4) prior to use.

B1.1.1 PT Samples

PT sample types will be prepared in ASTM Type II DI water. The first type of PT sample will consist of ASTM Type II DI water spiked with a contaminant at five concentrations. Concentrations will include the lethal dose concentration given in Table 2 for each contaminant along with dilutions at approximately 10, 100, 1,000, and 10,000 times less than the lethal dose. The lethal dose of each contaminant was determined by calculating the concentration at which

250 mL of water is likely to cause the death of a 70-kg person based on human oral LD_{50} data.[6,7] Human oral LD_{50} data was not available for aldicarb, so rat oral LD_{50} data was used instead.[8] Each concentration level for the PT samples will be analyzed in triplicate.

Table 2. Lethal Dose of Target Contaminants

Contaminant (common name)	Oral Lethal Dose Concentration	Contaminant Class
VX	2.1 milligrams (mg)/Liter (L)	Chemical agent
GB (sarin)	20 mg/L	Chemical agent
GD (soman)	1.4 mg/L	Chemical agent
Aldicarb	260 mg/L	Carbamate pesticide
Dicrotophos	1400 mg/L	Organophosphate pesticide

The second type of PT sample will be potential interferent samples. Three replicates of each interferent PT sample will be analyzed to determine each enzymatic test kit's susceptibility to these commonly found interferents in DW. There will be two types of interferent PT samples. One will contain calcium and magnesium from carbonates spiked into ASTM Type II DI water, and the other will contain humic and fulvic acids obtained from the International Humic Substances Society spiked into ASTM Type II DI water. Each of these interferent mixtures will be prepared at two different concentration levels. One concentration will be near the upper limit of what would be expected in drinking water (250 mg/L total concentration for calcium and magnesium, 5 mg/L total concentration for humic and fulvic acids) and one concentration at a mid-low range of what would be expected (50 mg/L total concentration for calcium and magnesium, 1 mg/L total concentration for humic and fulvic acids). These spiked interferent levels will be confirmed through analysis of aliquots by the subcontract laboratory. Also, each contaminant will be added to these samples along with the potential interferent, at a concentration consistent with a 10x dilution of the lethal dose, and analyzed in triplicate.

B1.1.2 Drinking Water (DW) Samples

Drinking water samples will be collected from four geographically distributed municipal sources (Ohio, New York, California, and Florida) to evaluate the performance of the enzymatic test kits with various DW matrices. These samples will vary in their source and treatment and disinfection process. All samples will have undergone either chlorination or chloramination disinfection prior to receipt. Samples will be collected from water utility systems with the following treatment and source characteristics:

- Chlorinated filtered surface water source
- Chlorinated unfiltered surface water source
- Chlorinated filtered groundwater source
- Chloraminated filtered surface water.

All samples will be collected in new, pre-cleaned high density polyethylene (HDPE) containers. After sample collection, to characterize the DW matrix, an aliquot of each DW sample will be sent to a subcontract laboratory to determine the following water quality parameters: concentration of trihalomethanes, haloacetic acids, total organic halides, calcium, magnesium, pH, conductivity, alkalinity, turbidity, organic carbon, and hardness. The DW samples will be dechlorinated prior to their use with sodium thiosulfate pentahydrate to prevent the degradation of some of the target contaminants by chlorine. Also, some test kits require dechlorination of DW prior to analysis. The dechlorination of the DW will be qualitatively confirmed by adding a diethyl-p-phenylene diamine (DPD) tablet to an aliquot of DW. If the water does not turn pink, the dechlorination process will be determined to be successful. If the water does turn pink, additional dechlorinating reagent will be added and the dechlorination confirmation procedure repeated. Each DW sample will be analyzed without adding any contaminant, as well as after fortification with each individual contaminant at a single concentration level (10x dilution of the lethal dose). Aliquots of each contaminant stock solution will be diluted with DW samples to the appropriate concentration. Each sample will be tested in triplicate.

B1.1.3 Quality Control (QC) Samples

QC samples will include method blank (MB) samples consisting of ASTM Type II DI water, and positive and negative controls, if applicable and as provided by the vendor. All of the MB QC samples will be exposed to identical sample preparation and analysis procedures as the test samples. Positive and negative controls will be provided by the vendor or prepared and used according to the protocol provided by the vendor. The MB samples will be used to ensure that no sources of contamination are introduced in the sample handling and analysis procedures. At least 10% of the test samples (7 samples for each contaminant) will be MB samples. The vendor provided control samples will indicate to the operator whether the enzymatic test kit is functioning properly and will be added to each set of samples analyzed by the technology, as applicable, or added per batch of samples as indicated by the vendor. The test samples and MB samples will be analyzed blindly by the operator in that the samples used for analysis will be marked with a non-identifying number.

B1.1.4 Portability Samples and Operational Factors

Those enzymatic test kits that are designed to be field portable will be tested outside of the laboratory by a non-technical operator. A non-technical operator is considered to be someone with little to no laboratory experience who would be representative of a first responder. The non-technical operator will be trained in the use of the enzymatic test kit by the vendor or by another Battelle staff person who was trained by the vendor. Because many of the contaminants being tested are highly toxic and unsafe to be handled outside of a special facility, method blank samples and non-toxic positive and negative control samples will be analyzed as portability samples. The non-toxic positive and negative control samples will be provided by the vendor or prepared and used according to the vendor's protocol.. Because no samples spiked with the contaminants of interest will be used, only the operational aspects of each enzymatic test kit will be evaluated as part of the portability testing. As these technologies are anticipated to be used by first-responders, their performance in a non-laboratory setting will be evaluated under simulated first response conditions by having the operator dressed in a Level B protective suit, butyl gloves, boots, and either a self contained breathing apparatus (SCBA) or an air purifying respirator,

depending on the availability of personnel trained in the operation of such devices. The operator will have prior experience in working in personal protective equipment (PPE). One set of MB samples will be tested without the use of PPE. Ease of use from the perspective of the operator will be documented both with and without the PPE.

Operational factors such as ease of use and sample throughput will be evaluated based on observations recorded by Battelle staff. Operational factors will be noted for both the laboratory and non-laboratory portions of the verification test. These observations will be summarized to describe the operational performance of each enzymatic test kit in the verification reports.

Table 3 provides a summary of all of the samples to be tested for each enzymatic test kit and each contaminate of interest.

Table 3. Summary of Test Samples for Enzymatic Test Kits Verification

Performance Test (PT) Samples	Performance Factor Evaluated	Sample Description	Reps
Contaminant Only	Accuracy, Precision, False Negative	Contaminant PT sample @ lethal dose	3
		Contaminant PT sample @ 10 times less than the lethal dose	3
		Contaminant PT sample @ 100 times less than the lethal dose	3
		Contaminant PT sample @ 1,000 times less than the lethal dose	3
		Contaminant PT sample @ 10,000 times less than the lethal dose	3
Interferent	Interference Effects, Precision, False Positive/Negative Responses	Fulvic and humic acids @ a total concentration of 1 mg/L	3
		Fulvic and humic acids @ a total concentration of 1 mg/L + contaminant @ 10 times less than the lethal dose	3
		Fulvic and humic acids @ a total concentration of 5 mg/L	3
		Fulvic and humic acids @ a total concentration of 5 mg/L + contaminant @ 10 times less than the lethal dose	3
		Calcium and magnesium @ a total concentration of 50 mg/L	3

		Calcium and magnesium @ a total concentration of 50 mg/L + contaminant @ 10 times less than the lethal dose	3
		Calcium and magnesium @ a total concentration of 250 mg/L	3
		Calcium and magnesium @ a total concentration of 250 mg/L + contaminant @ 10 times less than the lethal dose	3
Portability Samples	**Performance Factor Evaluated**	**Sample Description**	**Reps**
Method Blank Samples	Operational factors	Analysis in PPE by non-technical operator	3
		Analysis not in PPE by non-technical operator	3
Drinking Water (DW) Samples	**Performance Factor Evaluated**	**Sample Description**	**Reps**
Filtered chlorinated surface water	Potential Matrix and Interference Effects, Precision, False Positive/Negative Responses	Unspiked	3
		Spiked with contaminant @ 10 times less than the lethal dose	3
Unfiltered chlorinated surface water		Unspiked	3
		Spiked with contaminant @ 10 times less than the lethal dose	3
Filtered chlorinated groundwater		Unspiked	3
		Spiked with contaminant @ 10 times less than the lethal dose	3
Filtered chloraminated surface water		Unspiked	3
		Spiked with contaminant @ 10 times less than the lethal dose	3
Quality Control (QC) Samples	**Performance Factor Evaluated**	**Sample Description**	**Reps**
Method Blank	Quality Check	DI water - 10% of all non-QC samples	7
Positive Control		Provided by vendor	Var[(a)]
Negative Control		Provided by vendor	Var[(a)]
Approximate total number of samples per contaminant			**76**

(a) Number of positive and negative controls will vary based on the protocol provided by the vendor.

B1.2 Statistical Analysis

The technologies participating in this verification test will only be evaluated for qualitative results (i.e., presence or absence of the contaminant of interest). All data analyses will be based on these qualitative results. The statistical methods and calculations used for evaluation of the qualitative performance parameters are described in the following sections. The performance parameters will be determined for each target analyte (see Table 2) individually.

When semi-quantitative measures (e.g., percent inhibition) are used in determining the presence or absence of a contaminant for a particular technology, the semi-quantitative measure will be used to determine a qualitative result for the enzymatic test kit (i.e., presence or absence of contaminant). A qualitative result will then be reported as with the other technologies and used for the data analyses described in the following sections. The semi-quantitative measure will also be reported for that sample but will not be used in any other data analyses.

B1.2.1 Accuracy

The accuracy will be assessed by evaluating how often the enzymatic test kit result is positive in the presence of a concentration above the LOD. Contaminant-only PT samples will be used for this analysis. An overall percent agreement will be determined by dividing the number of positive responses to the overall number of analyses of contaminant-only PT samples above the kit's LOD (see Equation 1). If the LOD for a technology is not known or available, then all analyzed contaminant-only PT samples above the concentration level where consistent negative results are obtained will be used in estimating the accuracy for that enzymatic test kit.

$$Accuracy\ (\%\ Agreement) = \frac{\#\ of\ positive\ contaminant\ only\ PT\ samples}{total\ \#\ of\ contaminant\ only\ PT\ samples} \times 100 \qquad (1)$$

B1.2.2 False Positive/False Negative Rates

A false positive response will be defined as an enzymatic test kit response indicating the presence of a contaminant when the ASTM Type II DI water (including interferent samples) or

drinking water sample is not spiked at all. A false positive rate will be reported as the number of false positive results out of the total number of unspiked samples (see Equation 2).

A false negative response will be defined as an enzymatic test kit response indicating the absence of a contaminant when the sample is spiked with a contaminant at a concentration greater than the enzymatic test kit's LOD. Spiked PT (contaminant and interferent) samples and spiked DW samples will be included in the analysis. Contaminant-only PT samples above the technology's LOD or the level at which consistent negative responses are obtained (when the LOD is not known) will be included in the analysis. A false negative rate will be evaluated as the number of false negative results out of the total number of spiked samples for a particular contaminant (see Equation 3).

$$\textit{False Positive Rate} = \frac{\textit{\# of positive results}}{\textit{total \# of unspiked samples}} \tag{2}$$

$$\textit{False Negative Rate} = \frac{\textit{\# of negative results}}{\textit{total \# of spiked samples}} \tag{3}$$

B1.2.3 Precision

Precision measures the repeatability and reproducibility of enzymatic test kit responses. The precision of the three replicates of each sample set will be assessed. Responses will be considered inconsistent if one or more of the three replicates differs from the response of the other samples in the replicate set. The overall precision for each enzymatic test kit will be assessed by calculating the overall number of consistent responses for all the sample sets. The results will be reported as the percentage of consistent responses out of all replicate sets (see Equation 4).

$$\textit{Precision (\% Consistent results)} = \frac{\textit{\# of consistent responses of replicate sets}}{\textit{total \# of replicate sets}} \times 100 \tag{4}$$

B1.2.4 Potential Matrix and Interferent Effects

The potential effect of the DW matrix on the enzymatic test kit performance will be evaluated qualitatively by comparing the results for the spiked and unspiked DW samples to those for the PT samples spiked with the contaminant at 100 times less than the lethal dose. Similarly, the potential effect of interferent PT samples will be evaluated. The results indicating the correct or incorrect reporting of the presence of a contaminant will be evaluated. The findings will be reported and discussed.

B1.3 Reporting

The statistical comparisons described above will be conducted separately for each enzymatic test kit being tested, and information on the additional performance parameters such as ease of use and sample throughput will be compiled and reported. Separate verification reports will be prepared for each kit that was tested. No intercomparison of the enzymatic test kit data will be performed at any time. For each enzymatic test kit, the verification report will present the test procedures and test data, as well as the results of the statistical evaluation of those data.

All use of the test kits by Battelle staff, including all operational aspects observed during laboratory and field testing, will be documented at the time of the test and reported. The verification report will briefly describe the ETV program, the AMS Center, and the procedures used in verification testing. These sections will be common to each verification report. The results of the verification test will then be stated, without comparison to any other technology tested or comment on the acceptability of the technology's performance.

B2 REFERENCE SAMPLE PREPARATION AND ANALYSIS

An aliquot of the stock or reference solution (that will be prepared from the stock solution) will be submitted for reference measurement (see Section B4 for reference methods). Reference or stock solutions for each contaminant (see Table 2) will be prepared separately. Interferent stock

solutions will contain multiple analytes in the same solution (calcium and magnesium or fulvic and humic acids together). The concentration of the contaminant and interfering compound stock solutions will be verified with standard analytical methods as described in Section B4. Up to four aliquots of each stock or reference solution will be analyzed over the course of the verification test. Aliquots to be analyzed by confirmatory methods will be preserved or extracted and stored as prescribed by the standard method to be used. Reference solutions for the pesticides and chemical agents will be prepared as close in time as possible to the test samples to minimize any differences in the rate of sample degradation. A reference sample for these compounds may also be submitted for reference measurement at the end of each day of testing to assess the stability of the analyte throughout the testing day. Reference solutions known or suspected to undergo hydrolysis will be preserved or extracted, according to the reference method, on the day of preparation. The time of preparation, extraction or preservation, and analysis will be noted. Reference or stock solutions (after extraction or preservation if prescribed in the reference method) will be stored according to the reference method or laboratory protocols.

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Sample custody will be documented throughout collection, shipping, and analysis of the samples from the water utilities to Battelle. Similar documentation will be recorded for shipping and analysis of samples to the subcontract laboratory. Sample chain-of-custody procedures will be in accordance with the Battelle SOP MDAS.I-009-Draft, Sample Chain of Custody.[9] The chain-of-custody form summarizes the samples collected and analyses requested. The chain-of-custody form will track sample release from the DW utilities to the Battelle laboratory, and from the Battelle laboratory to the subcontract laboratory. Each chain-of-custody form will be signed by the person relinquishing samples once that person has verified that the chain-of-custody form is accurate. The original sample chain-of-custody forms accompany the samples; the shipper will keep a copy. Upon receipt at the sample destination, chain-of-custody forms will be signed by the person receiving the samples once that person has verified that all samples identified on the chain-of-custody forms are present in the shipping container. Any discrepancies will be noted on the form and the sample

receiver will immediately contact the Verification Test Coordinator to report missing, broken, or compromised samples. Reference samples shipped to the subcontract laboratory will be analyzed within the holding time identified in the applicable reference method.

B4 LABORATORY REFERENCE METHODS

Table 4 provides the standard laboratory methods that will be used for the sample preparation and reference analyses during this verification test. Table 5 provides the reference methods that will be used for the analysis of the interferent solutions. Table 6 provides the methods that will be used for determining the physio-chemical characteristics of the drinking water matrices. Instrumentation used for each reference method is given in each table. All of the reference methods for the target analytes will be performed by Battelle. Reference methods for the physio-chemical characterization of the drinking water and the interferent solutions will be performed by a subcontract laboratory. In all cases, laboratories will follow the QC requirements specified in B5. A subcontract laboratory will be responsible for providing calibrated instrumentation, performing all method QA/QC, and providing calibration records for any instrumentation used.

Table 4. Reference Methods for Target Analytes

Target Analyte	Reference Method	Instrumentation[a]
VX	HMRC-IV-118-05[10]	GC/MS
GB (sarin)	HMRC-IV-118-05[10]	GC/MS
GD (soman)	HMRC-IV-118-05[10]	GC/MS
aldicarb	SOP for Analysis of Water Sample Extracts For Type 1 Analytes by Liquid Chromatography/ Mass Spectrometry[11]	LC/MS
dicrotophos	SOP for Extracting and Preparing Water Samples For Analysis of Dicrotophos, Mevinphos, And Dichlorovos [12]	GC/MS

[a] Gas chromatography (GC)/ mass spectrometry (MS)
Liquid chromatography (LC)/mass spectrometry (MS)

Table 5. Reference Methods for Interferents

Interferents	Reference Method	Instrumentation[a]
Ca and Mg	EPA 200.8[13]	Simultaneous ICP
Humic and Fulvic Acids	SM 5310[14]	Combustion infrared NDR

[a] Inductively coupled plasma (ICP)
Negative differential resistance (NDR)

Table 6. Reference Methods for the Physio-Chemical Characterization of Drinking Water

Parameter	Reference Method	Instrumentation[a]
Turbidity	SM 2130[14]	Turbidimeter
Organic carbon	SM 5310[14]	Combustion infrared NDR
Specific conductivity	SM 2510[14]	Conductivity meter
Alkalinity	SM 2320[14]	pH meter
pH	EPA 150.1[15]	pH meter
Hardness	EPA SM 2340[14]	pH meter
Total organic halides	SM 5320[14]	Microcoulometric combustion analyzer

Trihalomethanes	EPA 524.2[16]	GC/MS
Haloacetic acids	EPA 552.2[16]	GC/ECD

[a] Negative differential resistance (NDR)
Gas chromatography (GC)/ mass spectrometry (MS)
Gas chromatography (GC)/ Electron capture detection (ECD)

B5 QUALITY CONTROL AUDITS AND REQUIREMENTS

Steps will be taken to maintain the quality of the data collected during this verification test. When confirmation analyses of the stock or reference solutions of both contaminant and potential interfering compounds are performed, QC measures as noted in each laboratory's operating procedures or the reference method will be followed. Similarly, appropriate QC measures as stated in the reference method or the laboratory's operating procedures will be followed for the physio-chemical characterization of the DW. The reference methods to be followed for this verification test are listed in Tables 4 through 6. A summary of the QC samples and acceptance criteria associated with each method is presented in Table 7. The QC measures for these reference methods will at least include the analysis of a method blank sample with the analyses of the reference or stock solution. Method blank samples will be analyzed to ensure that no sources of contamination are present. If the analysis of a method blank sample indicates a concentration above the MDL for the confirmatory instrument, contamination will be suspected. Any contamination source(s) will be corrected, and proper blank readings will be achieved, before proceeding with the analyses. In general, a matrix spike or laboratory fortified spike sample will also be analyzed. Average acceptable recoveries for these samples are between 70-150%. Samples outside of the expected range will generally be flagged and rerun once the QC acceptance criteria have been met. QC samples will be run with every batch of 1-20 samples. Specific QC samples and acceptance criteria associated with each method can be found in the appropriate reference (see Tables 4-6).

Quality control samples as provided with each enzymatic test kit will also be run per the vendor's instructions (see Section B1.1.3). Method blank samples will also be run as part of the verification test (see Section B1.1.3).

Table 7. Quality Control Measures for Reference Methods

Reference Method	QC Samples	Frequency	Acceptance Criteria[a]
HMRC-IV-118-05	Duplicate	Once in every 10 samples	±10% RPD
	Blank	Once in every 10 samples	< MDL
	Matrix spike	Once in every 10 samples	Depends on matrix
SOP for Analysis of Water Sample Extracts For Type 1 Analytes by Liquid Chromatography/ Mass Spectrometry	Duplicate	Once in every 10 samples	±20% RPD
	Fortified blank	Once in every 10 samples	50-100% recovery
	Method blank	Once in every 10 samples	<1 ng/mL
	Spike	Once in every 10 samples	80-120% recovery
SOP for Extracting and Preparing Water Samples For Analysis of Dicrotophos, Mevinphos, And Dichlorovos	Duplicate	Once in every 10 samples	±50% RPD
	Method blank	Once in every 10 samples	<50 ppb
	Spike	Once in every 10 samples	70-150% recovery
EPA 200.8	Initial performance check	First, before anything else is run	95-105% recovery
	Continuing performance check	After every 10 samples	90-110% recovery
	Quality check standards	Once in every 10 samples	95-105% recovery
	Blank	At the start of a run and after every 10 samples	± the PQL
	Matrix spike	Once in every 10 samples	85-115% recovery
	Duplicate	Once in every 10 samples	±20% RPD if > 5x the PQL
SM 5310	Blank	At the start of a run and after every 10 samples	< RDL
	RDL	Once per run	70-130% recovery
	Matrix spike	Once in every 10 samples	75-125% recovery
	Duplicate	Once in every 10 samples	±20% RPD
SM 2130	Check standards	First, before anything else	90-110% recovery

	Duplicate	Once in every 10 samples	±10% RPD
SM 2510	Check standards	At the start of a run and after every 10 samples	±10% of the true value
	Blank	At the start of a run and after every 10 samples	< 2 UHMO
	Duplicate	At the start of a run and after every 10 samples	±20% RPD
EPA 150.1	Buffer	At the start of a run and after every 10 samples	±1% of the true value
	Duplicate	Once in every 10 samples	±10% RPD
SM 2320	Check standards	At the start of a run and after every 10 samples	±10% of the true value
	Blank	At the start of a run and after every 10 samples	< RDL
	Duplicate	Once in every 10 samples	±10% RPD
	Matrix spike	Once in every 10 samples	±10% of the true value
SM 2340	Check standards	At the start of a run and after every 10 samples	±10% of the true value
	Blank	At the start of a run and after every 10 samples	< RDL
	Duplicate	Once in every 10 samples	±10% RPD
	Matrix spike	Once in every 10 samples	±10% of the true value
EPA 524.2	Check standards	Every 12 hours	70-130% recovery
	LCS	Every 24 hours	70-130% recovery
	Blank	Every 12 hours	70-130% recovery
	Duplicate	Every 24 hours	70-130% recovery
EPA 552.2	Blank	Once in every 10 samples	70-130% recovery
	Matrix spike	Once in every 10 samples	70-130% recovery
	Duplicate	Once in every 10 samples	70-130% recovery
SM 5320	Blank	Once per batch not to exceed 20 samples	< RDL
	Matrix spike	Once per batch not to exceed 20 samples	55-139% recovery

	Duplicate	Once per batch not to exceed 20 samples	90-131% recovery

[a] Relative percent difference (RPD)
Method detection limit (MDL)
Practical quantitation limit (PQL)
Reporting detection limit (RDL)

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

The instruments used for the reference analyses (see Tables 4-6) will be tested and inspected as per the standard operating procedures of Battelle or the subcontract laboratory or per the standard methods being used to make each measurement. Instruments to be used in the reference analyses for this test include (but are not limited to) gas chromatography (GC)/mass spectrometry (MS), liquid chromatography/MS, pH electrodes, inductively-coupled plasma/MS, and GC/electron capture detection. Any discovered deficiencies with a particular instrument will be resolved per the protocol of the laboratory in a timely manner. Any maintenance required on components of the enzymatic test kits will be the responsibility of the vendor.

B7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

The instruments used for the reference analyses will be calibrated per the standard reference methods being used to make each measurement or the standard operating procedures of the analysis laboratory. Instruments to be used in the reference analyses for this test include (but are not limited to) gas chromatography (GC)/mass spectrometry (MS), liquid chromatography/MS, pH electrodes, inductively-coupled plasma/MS, and GC/electron capture detection. If any component of an enzymatic test kit requires calibration, the vendor will provide Battelle technical staff with instructions on how to properly maintain such components. All calibrations performed will be documented by Battelle in the project laboratory record book. Calibration of spectrometry instruments will generally involve a 4-8 point calibration curve covering the range of concentrations of the reference solutions to be analyzed. Calibration of each reference instrument will be performed as frequently as required in the reference method guidelines. Calibration procedures for each method

are summarized in Table 8.

Calibration of the applicable enzymatic test kit components will be done as often as suggested by the vendor. This calibration will use National Institute of Standards and Technology (NIST)-traceable standards if applicable or calibration solutions and/or devices supplied by the vendor. Pipettes used during solution preparation will be maintained and calibrated as required by Battelle standard operating procedures (i.e., minimum of every 6 months). Pipettes will be checked and either recalibrated or replaced if they are dropped accidentally over the course of testing.

Table 8. Calibration Procedures for Reference Methods

Reference Method	Calibration Procedures
HMRC-IV-118-05	5 point curve performed once with each set of samples. Calibration check also performed.
SOP for Analysis of Water Sample Extracts For Type 1 Analytes by Liquid Chromatography/ Mass Spectrometry	At least a 4 point curve performed once with each set of samples. Continuing calibration blanks and verification standards run also.
SOP for Extracting and Preparing Water Samples For Analysis of Dicrotophos, Mevinphos, And Dichlorovos	4 point curve performed once with each set of samples
EPA 200.8	3 point curve performed once with each set of samples. Performance check standards also run.
SM 5310	6 pint curve performed once with each set of samples or once every 24 hours
SM 2103	5 point curve performed quarterly each year. Continuing calibration checks and check standards also performed.
SM 2510	N/A. Performance check standards run.
EPA 150.1	Calibration performed once before each set of samples
SM 2320	Calibration performed once before each set of samples. Performance check standards also run.
SM 2340	Calibration performed once before each set of samples. Performance check standards also run.
EPA 524.2	7 point curve performed once before each set of samples. Performance check standards also run.
EPA 552.2	5-6 point curve performed once before each ste of samples.

SM 5320	5 point curve performed once before each set of samples. Recalibrated if the performance check standards fail.

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

All materials, supplies, and consumables will be ordered by the Verification Test Coordinator or designee. Where possible, Battelle will rely on sources of materials and consumables that have been used previously as part of ETV verification testing without problems. Battelle will also rely on previous experience or recommendations from EPA advisors, host facility staff, or enzymatic test kit vendors. NIST-traceable materials will be used whenever possible. All items will be compared with their Certificates of Analysis, or similar documentation. Items that don't match will be sent back to their supplier.

B9 NON-DIRECT MEASUREMENTS

Data published previously in the scientific literature relating to enzymatic test kits' performance will not be used during this verification test.

B10 DATA MANAGEMENT

Various types of data will be acquired and recorded electronically or manually by Battelle technical staff during this verification test. Table 9 summarizes the type of data to be recorded. All data and observations for the operation of the test kits will be documented by Battelle technical staff on data sheets or in laboratory record books. If a subcontract laboratory is used, results from the subcontract laboratory reference instruments will be compiled by the subcontractor's staff in electronic format and submitted to Battelle upon obtaining the results. Results from any Battelle-performed reference methods will be similarly compiled and submitted.

Table 9. 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	ETV laboratory record books or data recording forms	Start/end of test procedure, and at each change of a test parameter	Battelle	Used to organize and check test results; manually incorporated in data spreadsheets as necessary
Sample preparation (dates, concentrations, etc.)	ETV laboratory record books	When each solution is prepared	Battelle	Used to confirm the concentration and integrity of the samples analyzed
Enzymatic test kit procedures and sample results	ETV data sheets and laboratory record book	Throughout test duration	Battelle	Manually incorporated into data spreadsheets for statistical analysis and comparisons
Reference method sample preparation	ETV laboratory record book	Throughout sample preparation	Battelle	Used to demonstrate validity of samples submitted for reference measurements
Reference method procedures, calibrations, QA, etc.	Laboratory record books or data recording forms	Throughout sampling and analysis processes	Battelle or subcontract laboratory	Retained as documentation of reference method performance
Reference method analysis results	Electronically from reference analytical method	Every sample analysis	Battelle or subcontract laboratory	Converted to spreadsheets for calculations

Records received by or generated by any of the Battelle technical staff during the verification test will be reviewed by a Battelle staff member within two weeks of receipt or generation, respectively, before the records are used to calculate, evaluate, or report verification results. If a Battelle staff member generated the record, this review will be performed by a Battelle 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, data calculations performed by Battelle technical staff will be spot-checked by Battelle technical staff to ensure that calculations are performed correctly. Calculations to be checked include any statistical calculations described in this test/QA plan. The data obtained from this verification test will be compiled and reported independently for each enzymatic test kit. Results for the enzymatic test kits from different vendors will not be compared with each other.

Among the QA activities conducted by Battelle QA staff will be an audit of data quality. This audit will consist of a review by the Battelle Quality Manager of at least 10% of the test data. During the course of any such audit, the Battelle Quality Manager will inform the technical staff of any findings and any immediate corrective action that should be taken. If serious data quality problems exist, the Battelle Quality Manager will request that Battelle's AMS Center Manager issue a stop work order. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem, and will implement any necessary follow-up corrective action. The Battelle 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 this verification test to anticipate and resolve potential problems before the quality of performance is compromised. One of the major objectives of this test/QA plan is to establish mechanisms necessary to ensure this. Internal QC measures described in this test/QA plan, which is peer reviewed by a panel of outside experts, implemented by the technical staff and monitored by the Verification Test Coordinator, 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 Test Coordinator. Technical staff have the responsibility to identify problems that could affect data quality or the ability to use the data. Any problems that are identified will be reported to the Verification Test Coordinator, who will work with the Battelle Quality Manager to resolve any issues. Action will be taken to control the problem, identify a solution to the problem, and minimize losses and correct data, where possible. Independent of any EPA QA activities, Battelle will be responsible for ensuring that the following audits are conducted as part of this verification test.

C1.1 Performance Evaluation Audits

When possible, the concentration of the standards used to prepare the samples fortified with contaminants and potential interfering compounds will be confirmed by analyzing standards prepared in ASTM Type II DI water from two separate commercial vendors using the reference methods noted in Tables 4 and 5. The standards from one vendor will be used during the verification test, while the standards from the second vendor will be used exclusively to confirm the accuracy of the displayed concentration of the first vendor. Agreement of the standards within 25% (percent difference) is required for the measurements to be considered as acceptable. Failure to achieve this agreement will trigger a repeat of the performance evaluation (PE) comparison.

Failure in the second comparison requires obtaining another set of standards, and repeating the PE audit.

Given the security requirements and lack of alternate sources for some of the chemical agents used in this verification test, PE audits will not be performed for these contaminants. PE audits will be done for all remaining compounds when more than one source of the contaminant or potential interfering compounds are available. PE audits will only be performed on compounds used to prepare test samples and will not be performed on any solutions supplied as part of an enzymatic test kit.

C1.2 Technical Systems Audits

The Battelle Quality Manager will perform a technical systems audit (TSA) at least once during this verification test. The purpose of this audit is to ensure that the verification test is being performed in accordance with the AMS Center QMP[1], this test/QA plan, published reference methods, and any SOPs used by Battelle. In the TSA, the Battelle Quality Manager, or designee, may review the reference methods used, compare actual test procedures to those specified or referenced in this plan, and review data acquisition and handling procedures. In the TSA, the Battelle Quality Manager will observe testing in progress, observe the reference method sample preparation and analysis (when available), inspect documentation, and review technology-specific record books. He will also check standard certifications and may confer with other Battelle staff. A TSA report will be prepared, including a statement of findings and the actions taken to address any adverse findings. The EPA AMS Center Quality Manager will receive a copy of Battelle's TSA report. At EPA's discretion, EPA 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 Battelle Quality Manager will audit at least 10% of the verification data acquired in the verification test. The Battelle 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 in accordance with Section 3.3.4 of the AMS Center QMP[1]. The results of the TSA will be submitted to EPA. 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 Battelle 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 Battelle Quality Manager will notify the AMS Center Manager to authorize a stop work order. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem and will implement any necessary follow-up corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken. The test/QA plan and final report are reviewed by EPA AMS Center QA staff and the EPA AMS Center program management staff. Upon final review and approval, both documents will then be posted on the ETV website (www.epa.gov/etv).

SECTION D

DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

The key data review, validation, and verification requirements for the verification test are the collection of QC samples as outlined in the test/QA plan, a comparison of field data sheet comments against final data to flag any suspect data, and a review of final data (as described in Section B10). The QA audits, as described in Section C, are designed to assure the quality of this data.

D2 VALIDATION AND VERIFICATION METHODS

Data verification is conducted as part of the data review as described in Section B10 and C1.2 of this test/QA plan. This verification will include a visual inspection of handwritten data 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 error code and the initials of the recorder and data of entry). Data manually incorporated into spreadsheets for use in calculations will be checked against the handwritten data to ensure no transcription errors occurred. Calculations will be spot-checked 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 (i.e., Excel). Calculations performed using standard commercial software (i.e., Excel) will be reviewed by inspection of the equations used for the calculations and verification of selected calculations by handheld calculator.

A number of data validation procedures will also be performed. Section C of this test/QA plan provides a description of the validation safeguards employed for this verification test. Data validation efforts include the completion of QC activities, and the performance of TSA and PE audits as described in Section C.

An audit of data quality will be conducted by the Battelle Quality Manager to ensure that

data review, verification, and validation procedures were completed, and to assure the overall quality of the data.

D3 RECONCILIATION WITH USER REQUIREMENTS

This purpose of this verification test is to evaluate the performance of enzymatic test kits. To meet the requirements of the user community, the data obtained in this verification test should include thorough documentation of the performance of each enzymatic test kit during the verification test. The data review, verification, and validation procedures described in previous sections will assure that data meet these requirements, are accurately presented in the verification reports generated from this test, and that data not meeting these requirements are appropriately flagged and discussed in the verification reports. The QC acceptance criteria described in Section B4 will be used to ensure that the reference methods provide accurate measurements of the reference or stock solutions and that the levels of contaminants tested are accurately presented in the verification reports.

The data from this verification test will be compiled into an ETV verification report. The report will be submitted to EPA in Word Perfect and Adobe pdf format and subsequently posted on the ETV website. This test/QA plan and the resulting ETV verification report(s) will be subjected to review by the enzymatic test kit vendors, Battelle staff, EPA, and expert peer reviewers. The reviews of this test/QA plan will assure that this verification test and the resulting reports meet the needs of potential users of the enzymatic test kits.

SECTION E

HEALTH AND SAFETY

E1 STANDARD/TEST SAMPLE PREPARATION

All handling of solid and highly concentrated aqueous solutions of contaminants and possible interferences will be done inside of a laboratory hood with hood sash set to the lowest height that still allows for safe manipulation of materials. The following guidelines should be adhered to:

- Personal protective equipment shall include safety glasses with side shields, a laboratory coat, and nitrile lab gloves. Gloves shall be immediately changed if they become contaminated.
- All contaminated waste shall be handled as hazardous waste and disposed of according to facility regulations.

E2 SAMPLE HANDLING DURING VERIFICATION TESTING

Laboratory handling of any solutions used during the verification test will be accomplished by taking the following precautions:

- All containers shall be stored and transported in double containment.
- Safety goggles or glasses, nitrile gloves with long cuffs, and a chemical resistant disposable lab coat, or other safety equipment specifically called for in facility guidelines, shall be worn when handling all chemicals. Gloves shall be immediately changed if they become contaminated.

E3 TESTING OF VX, GB, AND GD

Use of these contaminants in the verification of enzymatic test kits will be done following the safety procedures required at the HMRC facility.

SECTION F

REFERENCES

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