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Environmental Technology Verification Program

Advanced Monitoring Systems Center

Test/QA Plan for Verification of Mobile Mass Spectrometers

TEST/QA PLAN

for

Verification of
Mobile Mass Spectrometers

July 20, 2005

Prepared by

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ETV Advanced Monitoring Systems Center

Test/QA Plan for Verification of Mobile Mass Spectrometers

Version 1

July 20, 2005

VENDOR ACCEPTANCE:

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Company _____

Date _____

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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 daily operations of this verification test will be coordinated and supervised by Battelle personnel, with the participation of the vendors who will provide their mobile mass spectrometers for verification testing. The testing will be conducted at Battelle in Columbus and West Jefferson, Ohio. Staff from Battelle will oversee operation of the mobile mass spectrometers during periods of routine operation. Each vendor will provide one mobile mass spectrometer and training to Battelle staff on the use of the instrument. After the vendor is sufficiently satisfied in the training of Battelle staff, the vendor will sign a consent form. Quality assurance (QA) oversight will be provided by the Battelle Quality Manager. The organization chart in Figure 1 identifies the responsibilities of the organizations and individuals associated with the verification test. Roles and responsibilities are defined in the subsequent section.

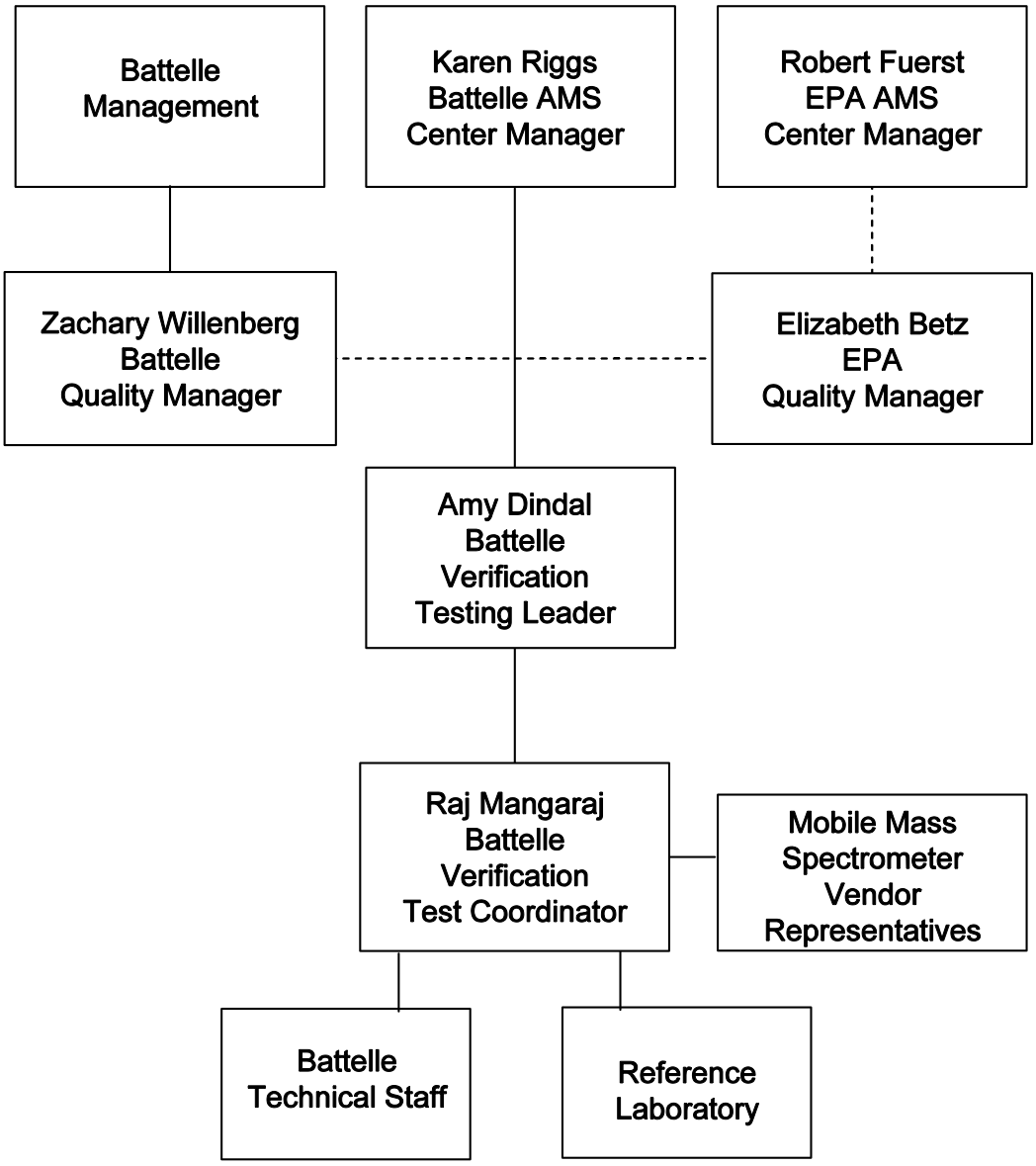

Figure 1. Organization Chart

A4.1 Battelle

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

- Assemble a team of qualified technical staff to conduct the verification test.
- Direct the team in performing the verification test in accordance with this 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 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 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 the Verification Testing Leader for the AMS Center. As such, Ms. Dindal will provide technical guidance and oversee the various stages of verification testing. She will:

- Support Mr. Mangaraj 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.

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.
- Support Mr. Mangaraj in responding to any issues raised in assessment reports and audits.
- Maintain communication with EPA's technical and quality managers.
- Issue a stop work order if Battelle or EPA QA staff discovers adverse findings that will compromise test results.

Battelle Technical Staff will support Mr. Mangaraj in planning and conducting the verification test. The responsibilities of the technical staff will be to:

- Become familiar with the operation and maintenance of the mobile mass spectrometers through instruction by the vendors.
- Assure that test procedures and data acquisition are conducted according to this test/QA plan.
- Assure that the data from each mobile mass spectrometer are recorded and transmitted to the Verification Test Coordinator.
- Provide input on test procedures, instrument operation, and maintenance for the draft verification reports.
- Perform statistical calculations specified in this test/QA plan.
- Provide results of statistical calculations and associated discussion for the verification reports as needed.
- Support Mr. Mangaraj in responding to any issues raised in assessment reports and audits related to statistics and data reduction as needed.

Mr. Zachary Willenberg is Battelle's Quality Manager for the AMS Center. Mr. Willenberg or his designee 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.
- Request that Battelle's AMS Center Manager issue a stop work order if 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 Mobile Mass Spectrometers Vendors

The responsibilities of the mobile mass spectrometer vendors are as follows:

- Review and provide comments on the draft test/QA plan.
- Accept (by signature of a company representative) the EPA-approved test/QA plan prior to test initiation (see page 4).
- Provide one mobile mass spectrometer for evaluation during the verification test.
- Provide all other equipment/supplies/reagents/consumables needed to operate their mobile mass spectrometer for the duration of the verification test.
- Supply a representative to train Battelle staff on the mobile mass spectrometer so that they will be able to operate the technology during the verification test.
- Provide maintenance and repair support for their mobile mass spectrometers, on-site if necessary, throughout the duration of the verification test.
- Review and provide comments on the draft verification report and verification statement for their respective mobile mass spectrometers.

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 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 an external audit.
- Review draft verification reports and verification 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 verification statements.
- Oversee the EPA review process for the test/QA plan, verification reports and verification statements.
- Coordinate the submission of verification reports and verification statements for final EPA approval.
- Notify the Battelle AMS Center Manager if EPA audits indicate a stop work order is necessary.

A4.4 Subcontract Laboratory

Any laboratory that is providing reference measurements will follow the requirements of reference methods as well as the QC requirements as stated in this test/QA plan. The responsibilities of this laboratory will include:

- Proper receipt and handling of sample material.
- Accurate measurement of target analyte.
- Submission of data and any supporting documents.
- Analysis and reporting of performance evaluation (PE) samples.
- Participation in audit by Battelle AMS Quality Manager or EPA AMS Quality Manager.

A5 BACKGROUND

The ETV Program's AMS Center conducts third-party performance testing of commercially available technologies that detect or monitor natural species or contaminants in air, water, and soil. Stakeholder committees of buyers and users of such technologies recommend technology categories, and technologies within those categories, as priorities for testing. Mobile mass spectrometers were identified as a priority water security technology category through the AMS Center stakeholder process. Mobile mass spectrometers are a subset of mass spectrometers that include portable systems (i.e., those able to carried by the user) and field-transportable systems (i.e., systems modified specifically so that they may be able to be taken outside of a fixed laboratory setting).

A6 VERIFICATION TEST DESCRIPTION AND SCHEDULE

A6.1 Summary of Technology Category

The technology category to be tested in this verification test is mobile mass spectrometers to be used in support of drinking water security. The performance of the mobile mass spectrometers will be based on the analysis of target contaminants at levels of concern

using lethal dose (LD_{50}) or maximum contaminant level (MCL) concentrations. Many volatile and semivolatile contaminants in water are typically detected using bench-top mass spectrometers in a traditional laboratory setting. However, the instruments to be verified in this test are portable units designed to be taken outside of the typical laboratory setting for "field" analysis. This portability offers an advantage for first-responders and other users that maybe seeking to obtain chemical information when time, sampling and other limitations require analysis outside of the typical laboratory setting.

Typically, the mobile mass spectrometers are composed of four components: a separation technique, an ion source, a means of ion separation or filtering, and an ion detector. There are a number of different means of ion separation including time of flight, ion trap, quadrupole mass filter, and quadrupole ion trap. The instruments that will be evaluated in this verification test include gas chromatography (GC) as a separation technique prior to detection by the mass spectrometer.

In performing the verification test, Battelle will follow the technical and QA procedures specified in this test/QA plan and will comply with the data quality requirements in the AMS Center QMP[1].

A6.2 Verification Schedule

Table 1 shows the planned schedule of activities in testing and data analysis/reporting in this verification test. As shown in Table 1, the test of mobile mass spectrometers is planned to begin in August 2005. The test will be conducted in three parts. Volatile organic contaminants and pesticides will be tested first. The second part will repeat a portion of the verification test in a field setting. For this test, a field setting will be defined as a setting outside of a laboratory. Finally, testing using dilute solutions of chemical warfare agent (CWA) will be conducted in a chemical surety facility. Following the completion of the testing effort, a separate verification report will be drafted for each mass spectrometer, reviewed, and submitted to EPA for final signature. Concurrent measurements will be conducted using laboratory reference methods where and when applicable.

Table 1. Planned Verification Schedule (Dates Subject to Change)

Month (2005)	Test Activity	
	Verification Activities	**Data Analysis and Reporting**
August	Install mass spectrometers in laboratory Undergo training by vendors Perform tests with volatile organic contaminants (VOC) and pesticides Submit samples for reference analysis	Begin preparation of report template Compile data from VOC and pesticide testing Review and summarize operator observations Compile data packages for reference analyses
September	Repeat portion of testing in field setting Test with CWA Submit samples for reference analysis	Compile data from field testing Compile data fromCWA testing Complete summary of operator observations Finalize results for reference analyses Complete common sections of reports
October	Decontamination of mobile mass spectrometers Return instruments to vendors as soon as items are cleared for release	Complete report sections on reference method comparisons compose draft reports
November/ December		Internal review of draft reports Vendor review of draft reports Revise draft reports Peer review of draft reports
January/ February		Revise draft reports Submit final reports for EPA approval

The test procedures are described in Section B of this test/QA plan. The mobile mass spectrometers will be calibrated per vendors' instructions. The calibrated mobile mass spectrometers will subsequently be challenged with performance testing (PT) standard solutions and various fortified water samples.

A6.3 Test Facility

The verification test will take place at Battelle. A portion of the test will also be conducted in a field (non-laboratory) environment. Testing with CWA will take place at the Hazardous Materials Research Center (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 the demonstrated capability for storing and safely handling CWA, Class A poisons, toxins, agent simulants and other hazardous materials. The HMRC laboratories meet or exceed all requirements for the safe use, storage, decontamination, and accountability of CWA as defined by Army Regulation 50-6 (Chemical Surety)[3]. Operations within the laboratories always are conducted in accordance with Battelle's Chemical Safety Information Program[4].

A7 QUALITY OBJECTIVES

This verification test will evaluate the performance of mobile mass spectrometers for identification and quantification of target contaminants in water. This evaluation will include comparisons to reference methods when available and where applicable. 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 Quality Manager also may conduct an independent TSA, at her discretion.

A8 SPECIAL TRAINING/CERTIFICATION

Documentation of training related to technology testing, field testing, data analysis, and reporting is maintained for all Battelle technical staff in training files at their respective locations. For this test, it should be noted that an operator or operators with prior GC/MS experience will be used. CWA testing will be conducted by staff with the appropriate training, as outlined in HMRC Chemical Hygiene Plan[5]. 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 those staff prior to the start of testing. Battelle will document this training with a vendor training form,

signed by the vendor, that states which specific Battelle staff have been trained on the mobile mass spectrometer.

A9 DOCUMENTATION AND RECORDS

The records for this verification test will be contained in the test/QA plan, chain-of-custody forms, laboratory record books (LRB), data collection forms, electronic files (both raw data and spreadsheets), and the final verification report. All of these records will be maintained in the Verification Test Coordinator's office 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. Section B10 further details the data recording practices and responsibilities.

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. Explanations will accompany all non-obvious corrections. The correction is then initialed and dated by the person making the correction. In all cases, strict confidentiality of data from each vendor's instrument, and strict separation of data from different instruments, will be maintained. 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 test will specifically address the verification of mobile mass spectrometers for identification and quantification of target contaminants in water (listed in Table 2) by evaluating the accuracy, precision, linearity, sensitivity, and stability of each instrument in the analysis of various water matrices. This target analyte list represents the maximum number of contaminants to be tested and each technology will only be tested on target contaminants specified by the vendor. The mobile mass spectrometers will be evaluated for the performance parameters summarized in Table 3 and discussed in detail in the following sections.

Table 2. Target Contaminants and Concentrations of Interest for Verification Test of Mobile Mass Spectrometers

Contaminant	CAS#	LD_{50} (mg/L) Corresponding Concentration[a]	MCL[d] (mg/L)
VX[b]	50782-69-9	2.1	n/a
GB (sarin)[b]	107-44-8	20	n/a
GD (soman)[b]	96-64-0	1.4	n/a
dicrotophos[c]	141-66-2	1400	n/a
2,4-D	94-75-7	n/a	0.07
benzene	71-43-2	n/a	0.005
toluene	108-88-3	n/a	1
ethylbenzene	100-41-4	n/a	0.7
xylenes (total)	1330-20-7	n/a	10

[a] LD_{50} concentrations in water determined using 70 kg individual consuming 250 mL of water
[b] LD_{50} data obtained from USACHPPM MEG, Short-term Water-MEGs (these are cited in the notes for each chemical in Appendix D)
[c] LD_{50} data obtained from Gosselin, R.E., et al., Clinical Toxicology of Commercial Products. 5th ed. Baltimore: Williams and Wilkins, 1984., p. II-291
[d] MCLs specified by EPA National Primary Drinking Water Regulation, 40 CFR Š141

B1.1 Test Procedures

The following sections describe the test procedures that will be used to evaluate each of the mobile mass spectrometer performance parameters listed in Table 3. Performance testing will be conducted on each instrument separately, and will follow the procedures specified by the vendor including analysis of quality control samples. The vendor will define all operational conditions including (but not limited to) chromatographic column, selected ions or mass scanning range, sample preparation method, injection technique, and temperature program.

Table 3. Verification Test Performance Parameters

Performance Parameter	Method of Evaluation
Accuracy	Comparison to nominal concentration of spiked target contaminants (prepared in ASTM Type II water) as determined by reference method
Precision	Determined by relative standard deviation of three replicate measurements
Linearity	Determined by linear regression of performance test standard solutions
Sensitivity	Determined by ability to detect target contaminants at and below concentrations of interest (e.g., LD_{50}, MCL); 10x serial dilutions on PT standards will be performed until no change in instrument response is observed for target contaminant
Instrument Stability	Determined by comparing result of PT standard analyzed at end of the analytical sequence (defined as the entire list of samples to be analyzed on each day of testing) to that of PT standard analyzed at beginning of analytical sequence
Potential Matrix and Interference Effects	Evaluation of performance in various matrices and in the presence of potential interferents
Field Portability	user requirements; site requirements; test will include analysis of "blind" samples
Operational Factors	Operator observations, clarity of instruction manual, user-friendliness of software, records of needed and performed maintenance, use of expendable supplies, and sample throughput

Samples will be prepared daily from stock solutions to minimize loss of target contaminants due to hydrolysis. For PT standards, two separate aliquots will be produced in

ASTM Type II water using the same procedure. One aliquot will be submitted for testing and the other aliquot will be submitted for reference measurement. Reference measurements will be conducted on performance test standards only. For CWA testing, a stock solution containing all of the three target contaminants (GB, GD, and VX) will be prepared in acetone. Stock solutions for the other contaminants will be similarly prepared in solvents that will minimize target contaminant degradation. All matrix samples will be dechlorinated and pH adjusted to pH 7 (± 0.5) to minimize hydrolysis of CWA.

B1.1.1 Accuracy, Precision, Linearity, Sensitivity, and Stability.

Due to concerns regarding the stability of the target contaminants in water matrices, the evaluation of accuracy must be carefully approached so that degradation issues do not negatively impact the assessment of accuracy. As such, reference measurements will be conducted simultaneously or as close as possible to the measurements made with the mobile mass spectrometers. To verify the performance of the mobile mass spectrometers at the concentrations of interest for the target contaminants, PT standards will be prepared in ASTM Type II water. The target contaminant concentrations will be constructed to bracket concentrations of interest that are presented in Table 2. The concentrations of interest are calculated using LD_{50} values assuming a 70 kg individual consuming 250 mL of the contaminated water. When LD_{50} data are not available or feasible for testing, MCL, as defined by EPA National Primary Drinking Water Regulations, are used. See Table 4 for concentrations of the PT standards for the target analytes.

Table 4. Performance Test Standard Solution Concentrations for Target Analytes

Contaminant	PT Standard 1	PT Standard 2	PT Standard 3
VX	0.1 mg/L	1 mg/L	10 mg/L
GB (sarin)	1 mg/L	10 mg/L	100 mg/L
GD (soman)	0.1 mg/L	1 mg/L	10 mg/L
dicrotophos	10 mg/L	100 mg/L	1000 mg/L
2,4-D	0.001 mg/L	0.010 mg/L	0.100 mg/L
benzene	0.0001 mg/L	0.001 mg/L	0.010 mg/L
toluene	0.1 mg/L	1 mg/L	10 mg/L
ethylbenzene	0.1 mg/L	1 mg/L	10 mg/L
xylenes (total)	1 mg/L	10 mg/L	100 mg/L

Three replicate measurements will be made for each sample. Determination of accuracy of the PT standards will be based on agreement with reference measurements that will be performed concurrently with mobile mass spectrometer measurement. Since no reference measurements will be taken for the matrix and interferent-fortified samples, accuracy will be determined by comparison to PT standards. Quantitation of the matrix and interferent-fortified samples will be based on the calibration curve constructed using the theoretical concentrations of the spiked contaminants in the PT standards.

Relative standard deviation (RSD) of three replicate measurements of the PT standards will be determined to assess the precision of all mobile mass spectrometer measurements. Linearity will be determined by linear regression of the instrument response versus the theoretical target contaminant concentrations. This evaluation will not focus on determining instrumental detection limits since the purpose is to ascertain whether the mobile mass spectrometers are sensitive enough to measure the target contaminants at the concentration of interest in the various water matrices. Sensitivity will be assessed by the ability of the mass spectrometer to measure the target contaminant at and below the concentration of interest. Serial dilutions of 10x will be performed on the PT standard 1 until no change in instrument response for the target contaminant is observed. A PT standard will be repeated at the end of the analytical sequence to reflect the degree of calibration stability. Since benzene, toluene, ethylbenzene, and xylene (BTEX) will be

tested prior to CWA testing, the testing will be able to differentiate between instrument stability and any parallel target contaminant degradation that may be suspected in the case of CWA in water.

B1.1.2 Potential Matrix and Interference Effects

To measure the potential matrix effects on the mobile mass spectrometer in selected "real world" applications, the mass spectrometers will be challenged by analyzing samples that are fortified with the target contaminant at the level of PT Standard 2 (as indicated in Table 4) in various matrices including regional drinking water (DW) samples, raw (untreated) surface water, a weakly buffered water sample, and a strongly buffered water sample as shown in Table 5. PT Standard 2 provides a convenient concentration that is at or below the concentration of interest for the most of the target contaminants.

Table 5. Matrix and Potential Interferent Testing

Drinking water samples will be collected from four geographically distributed municipal

Sample Type	Spike Level
DW1	PT standard 2
DW2	PT standard 2
DW3	PT standard 2
DW4	PT standard 2
Raw Surface Water	PT standard 2
Weak Buffer Water	PT standard 2
Strong Buffer Water	PT standard 2
THM Spiked Water	PT standard 2

sources to evaluate the performance of the mobile mass spectrometers with various sample matrices. These finished DW samples will vary in their source, 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 (DW1)
- Chlorinated unfiltered surface water source (DW2)
- Chlorinated groundwater source (DW3)
- Chloraminated filtered surface water source (DW4)

All samples will be collected in 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 (THMs), haloacetic acids, total organic halides, pH, conductivity, alkalinity, turbidity, organic carbon, and hardness (see Table 6).

Table 6. Physio-Chemical Characterization of Drinking Water

Parameter[a]	Method
Turbidity	EPA 180.1[5]
Organic carbon	SM 5310[6]
Specific conductivity	SM 2510[6]
Alkalinity	SM 2320[6]
pH	EPA 150.1[7]
Hardness	EPA 130.2[7]
Total organic halides	SM 5320[6]
Trihalomethanes	EPA 524.2[8]
Haloacetic acids	EPA 552.2[9]

(a) Physio-chemical DW characterization to be performed by the subcontract laboratory

Because free chlorine will degrade many of the contaminants and interferents during storage, the sample will be immediately dechlorinated with sodium thiosulfate pentahydrate (or other dechlorination reagents as per vendor protocol). 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, an additional dechlorinating reagent will be added and the dechlorination confirmation procedure will be repeated.

The effect of ionic strength on the results of the mobile mass spectrometers will be examined. Since natural water salt type and concentration can vary greatly by location, two samples will be fortified at the level of PT standard 2 in 442 Natural Water™ Standard Solution. This solution, manufactured by Myron L Instruments, is produced by adding 40% sodium sulfate, 40% sodium bicarbonate, 20% sodium chloride in deionized water. Two 442 solutions, 442-30 and 442-3000, will be selected to represent 21.8 ppm NaCl and 2027 ppm NaCl, respectively.

The mobile mass spectrometers will also be challenged by the presence of potential interferents. THMs are typically observed at low-levels in drinking water as by-products of the disinfection process. Four THMs (chloroform, bromoform, bromodichloromethane, and dibromochloromethane) will be spiked into the PT standard 2 at 80 ppb total, which is the MCL for THMs as defined in the EPA National Primary Drinking Water Regulations, 40 CRF Š141. Chloroform, bromoform, bromodichloromethane, dibromochloromethane will be spiked so that the concentration in solution will be 50, 5, 15, and 10 ppb, respectively, to represent typical ratios of THMs in finished drinking water.

B1.1.3 Field Portability

For this verification test, field portability is defined as the ability for a user to take and operate the mass spectrometer in a non-laboratory environment for sample analysis. Observations centrally related to field portability will be observed and reported. These considerations will include such items as weight and dimensions of unit, impact on user mobility, start-up time, power requirements, and compressed gas consumption. The mobile mass spectrometers will be operated from within a trailer that will be climate controlled. It will be noted to what extent the mobile mass spectrometers may be carried outside of the trailer by the user. It is in this environment that testing of the mobile mass spectrometers will include the raw surface water matrix. Field testing will include the analysis of contaminants that will be unknown (or “blind”) to the operator. After

performing the vendor-specified calibration procedure, the operator will analyze the blind sample (in three replicates). Due to restrictions, the contaminants will not be CWA, but will consist of a single or combination of the other target contaminants. The blind sample will challenge the identification capability of the library matching function of the mobile mass spectrometer software.

B1.1.4 Operational Factors

Operational factors such as maintenance needs, data output, consumables used, ease of use, repair requirements, sample throughput etc., will be evaluated based on observations recorded by Battelle staff. A separate laboratory record book will be maintained for each mobile mass spectrometer undergoing testing, and will be used to enter daily observations on these factors. Examples of information to be recorded in the record books include the status of diagnostic indicators for the mass spectrometer; 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 instrument down time or data acquisition failure; and operator observations about ease of use of the mobile mass spectrometer. These observations will be summarized to aid in describing mass spectrometer performance in the verification report on each mobile mass spectrometer.

The time required for each sample from the start of sample preparation to reporting of results will define sample time. The number of samples that can be analyzed per unit time will define sample throughput. The sample throughput will be noted for laboratory and field portions of testing. The test samples for the verification test are summarized in Table 7.

Table 7. Summary of Test Samples for ETV Test of Mobile Mass Spectrometers[a]

Sample/ Matrix	Performance Factor	Sample Description	Reps
Performance Test (PT) standards/ ASTM Type II DI Water	Accuracy, Precision, Linearity, and Operational Factors	Blank ASTM Type II DI Water	3
		PT Standard 3	3
		PT Standard 2	3
		PT Standard 1	3
PT Matrix	Sensitivity	Successive 10x serials dilutions of PT Standard 1 will be analyzed until no change in instrument response is observed for target contaminant	1
PT Matrix	Instrument Stability	PT Standard 2	3
Matrix and Potential Interferent Samples/ Drinking Water (DW), Buffered Water (442 Water), and ASTM Type II DI Water	Matrix, Potential Interference Effect, and Operational Factors Matrices will be analyzed as unfortified and fortified (at PT standard 2 level)	DW1	3
		DW2	3
		DW3	3
		DW4	3
		Weak Buffer Water (442-30)	3
		Strong Buffer Water (442-3000)	3
		THM Spiked Water (ASTM Type II Water)	3
Field Portability Sample/ Raw Surface Water; this sample will be "blind" to the operator	Field Portability and Operational Factors Raw surface water will be analyzed as unfortified and fortified	Raw Surface Water (collected from river) will be spiked with target analyte from Table 2 and then submitted to the operator	3

(a) Test samples will be analyzed for each target contaminant mix (BTEX, pesticides, CWAs) except for the field portability samples which will only be performed with one set of non-CWA blind samples.

B1.2 Statistical Analysis

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

B1.2.1 Accuracy

The accuracy of the mobile mass spectrometer measurements with respect to the reference measurements will be assessed as the percent recovery (%R), using Equation 1:

$$\%R = \left(\overline{Y} \div \overline{X}\right) \times 100 \quad (1)$$

where $\overline{Y}$ is the average concentration of the target contaminant as measured by the mobile mass spectrometer and $\overline{X}$ is the target contaminant concentration as measured by the reference method. The ideal accuracy is 100%.

B1.2.2 Precision

The precision of the mobile mass spectrometers will be evaluated from complete measurements of the target contaminant (including three separate sample collections) performed in triplicate. The precision will be defined as the relative standard deviation (RSD) of the triplicate measurements:

$$\%RSD = (s \div Y_i) \times 100 \quad (2)$$

where $\overline{Y}$ is the average concentration *i* of the target contaminant as measured by mobile mass spectrometer, and *s* the standard deviation of the mobile mass spectrometer results. The ideal relative standard deviation is 0%.

B1.2.3 Linearity

Linearity of the analytical response will be assessed by a linear regression analysis using the target contaminant concentration as the independent variable and result from the mobile mass spectrometers being tested as the dependent variable. Linearity will be expressed in terms of slope, intercept, and coefficient of determination (r^2). The ideal value for r^2 is 1.

B1.2.4 Sensitivity

Sensitivity of the mobile mass spectrometers for the target contaminant will be determined by the ability of the instruments to detect at or below the concentrations of interest (see Table 2) in the different matrices including the PT matrix (ASTM Type II water), drinking water matrices, and potential interferent-fortified matrices.

B1.2.5 Instrument Stability

Instrument stability (S) will be determined by analyzing a PT standard at the end of the analytical sequence. The result of the PT standard will be compared to that of the PT standard analyzed at the start of the analytical sequence. Stability will be calculated using Equation 3:

$$\%S_{\mathrm{i}} = \left(Yi2 \div Yi1\right) \times 100 \tag{3}$$

where Y*i*2 and Y*i*1 are the average results for the last and first PT standard, respectively, for target contaminant *i*. The length of the analytical sequence will be noted when reporting stability. The ideal value for S is 100%.

B1.2.6 Potential Matrix and Interference Effects

Potential matrix and interference effects will be assessed by comparison of the analytical results to those of the PT standards. In the absence of instrument drift and target contaminant degradation, it will be assumed that a matrix or interferent effect is responsible for any result with a lower accuracy and/or precision than that of the PT standards.

B1.3 Reporting

The statistical comparisons described above will be conducted separately for each of the mass spectrometers being tested, and information on the operational parameters will be compiled and reported. The data for each mass spectrometer will be kept separate from data for all other mass spectrometers, and no intercomparison of the mass spectrometer data will be performed at any time. A separate verification report will be prepared for each mass spectrometer tested that presents the test procedures and test data, as well as the results of the statistical evaluation of those data. Operational aspects of the mobile mass spectrometers will be recorded by testing staff at the time of observation during the field test, and summarized in the verification report. 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 report. Each verification report will briefly describe the ETV program, the AMS Center, and the procedures used in the verification testing. The results of the verification test will be stated quantitatively, without comparison to any other mobile mass spectrometer tested, or comment on the acceptability of the instrument's performance. Each draft verification report will first be subjected to review by the respective mass spectrometer vendor, then revised and subjected to a review by EPA and other peer reviewers. The peer review comments will be addressed in further revisions of the report, and 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 requirements of the AMS Center QMP.[1]

B2 REFERENCE SAMPLE PREPARATION

An aliquot of the PT standards will be submitted for reference measurement where and when applicable. If the sample volume requirement for the reference method exceeds what will be available after testing, a separate aliquot will be prepared using the same procedure. If a separate aliquot is prepared, it will be prepared as close in time as possible to the original sample to minimize any differences in rate of sample degradation in the matrix.

B3 SAMPLE CUSTODY REQUIREMENTS

Sample custody will be documented throughout collection, shipping, and analysis of the samples from the water utility to Battelle laboratories. 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[11]. The chain-of-custody form summarizes the samples collected and analyses requested. The chain-of-custody form will track sample release from the field 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.

B4 LABORATORY REFERENCE METHODS

Laboratory reference methods (see Table 8) will be used to determine the accuracy of sample preparation, verify the accuracy of the mobile mass spectrometer measurements using the PT standards, and demonstrate the stability of the target contaminants in the PT matrix. These reference methods will be performed by Battelle or a subcontract laboratory. In all cases, the reference analyses will follow the quality control requirements specified in B5 in addition to any QC requirements specified in each reference method.

Table 8. Reference Methods for Target Contaminants

Contaminant	Reference Method
VX	HMRC-IV-118-05[12]
GB (sarin)	HMRC-IV-118-05[12]
GD (soman)	HMRC-IV-118-05[12]
dicrotophos	U.S. EPA 8141[13]
2,4-D	U.S. EPA 515.1[14]
benzene	U.S. EPA 524.2[9]
toluene	U.S. EPA 524.2[9]
ethylbenzene	U.S. EPA 524.2[9]
xylenes (total)	U.S. EPA 524.2[9]

B5 QUALITY CONTROL AUDITS AND REQUIREMENTS

Steps will be taken to maintain the quality of the data collected during this verification test. Reference methods will be required to analyze the PT standards. QC for operation of the mobile mass spectrometers (e.g., daily mass tuning) will be specified by the vendor. Table 9 summarizes the quality control measures used during this test.

Table 9. Quality Control Measures

QC Measures	Addressed By	Required Performance	Corrective Action if Requirements Not Met
Accuracy of Standard Preparation	Performance test (PT) standards prepared from stock solutions will be verified by reference methods	Results must agree within +/- 20% of theoretical concentration	If results do not agree within 20% of theoretical concentration, the concentration as measured by the reference method will be used for linearity calculations
Absence of contaminant in unfortified PT matrix (ASTM Type II water)	Blank measurement; 10% of reference measurements or at least 1 for every 10 reference measurements	Contaminants should be less than reporting limit of reference method	If target contaminant is detected above the reporting limit, blank subtraction of the background contaminant concentration will be performed
Reproducibility of Reference Measurement Value	Duplicate measurement; 10% of reference measurements or at least 1 for every 10 reference measurements	Relative Percent Difference (RPD) must be within +/- 15%	Duplicate analysis will be repeated; if discrepancy still exists, a deviation will be issued and the result will be noted in report
Accuracy of Reference Measurement	Performance evaluation (PE) samples will be submitted for each reference method	Result must agree within +/- 20% of theoretical concentration	At least one PE sample will be submitted for each reference method before the start of the verification testing; if result does not agree within 20% of the theoretical concentration, reference method will be repeated; if discrepancy still exists, a deviation will be issued and the result will be noted in report
Operation of Mobile Mass Spectrometers	Vendor-provided QC	Defined by vendor	Defined by vendor

B6 INSTRUMENT TESTING, INSPECTION, AND MAINTENANCE

The mobile mass spectrometers and associated equipment (e.g., SPME inlet) will be visually inspected before and after each day of testing for any wear and tear that may compromise performance. Maintenance procedures, as outlined by vendors, will be followed during the verification process.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

The mobile mass spectrometers will be calibrated at least once each analytical sequence according to the vendors' instructions. Additional calibration will be performed as outlined by the vendor.

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 mass spectrometer vendors. National Institute of Standards and Technology (NIST)-traceable materials will be utilized where and whenever possible.

B9 NON-DIRECT MEASUREMENTS

Data published previously in the scientific literature 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 staff during this verification test. Table 10 summarizes the types of data to be recorded. All maintenance activities, repairs, calibrations, and operator observations relevant to the operation of the mobile mass spectrometers will be documented by Battelle in laboratory record books. A

separate record book will be provided for each participating mass spectrometer. Results from the laboratory reference method will be compiled in electronic format, and submitted to the Verification Test Coordinator in the form of a sample preparation and analysis report at the conclusion of reference analyses.

Table 10. 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, mass spectrometer maintenance, down time, etc.	ETV laboratory record books or data recording forms	Start/end of test procedure, and at each change of a test parameter or change of instrument status	Battelle	Used to organize and check test results; manually incorporated in data spreadsheets as necessary
Mass spectrometer calibration information	ETV laboratory record books or electronically	At instrument calibration or recalibration	Battelle	Incorporated in verification report as necessary
Mass spectrometer readings	Recorded electronically by each instrument and then downloaded daily	For each sample	Battelle	Converted to spreadsheet for statistical analysis and comparisons
Reference method sample preparation	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 Battelle staff during the verification test will be reviewed by a Battelle staff member within two weeks of generation or receipt, 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, any calculations performed by Battelle 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 mass spectrometer. Results for the mobile mass spectrometers 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 quality control 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, "blind" samples will be submitted to analysts performing the reference measurements. These performance evaluation (PE) samples will assess the accuracy of the reference measurements. These samples will prepared in accordance with the stated detection limits of the reference laboratories. At least one PE sample will be submitted per reference method prior to the start of the verification test and once during the verification test.

The results for the PE samples must be within ± 20% of the expected result. If the PE sample value is not within these limits, the PE sample measurement will be repeated. If the value of the repeated PE sample is outside of the acceptable range, the reference instrument will be

recalibrated, and the PE will be reanalyzed (unless the operator identifies or suspects another cause for the failure). Continued failure of the PE measurement will result in discussion with the Verification Testing Leader and the data being flagged accordingly.

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 standard operating procedures (SOPs) used by Battelle. In the TSA, the Battelle Quality Manager or designee will 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 tour the test site, observe the reference method sample preparation and analysis, inspect documentation; and review instrument-specific record books. He will also check standard certifications and mass spectrometer data acquisition procedures, and may confer with the instrument vendors and 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 or designee 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 is authorized to 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. The test/QA plan and final report are reviewed by EPA AMS Center QA staff and 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 requirements for the verification test are stated in Section B10 of this test/QA plan. All data generated will be reviewed on a daily basis for accuracy and completion. The QA audits described within Section C of this document, including the audit of data quality, are designed to assure the quality of the data.

D2 VALIDATION AND VERIFICATION METHODS

Section C of this test/QA plan provides a description of the validation safeguards employed for this verification test. Data validation and verification efforts include the analysis of QC samples as required in this document, and the performance of TSA and PE audits as described in Section C.

D3 RECONCILIATION WITH USER REQUIREMENTS

This test/QA plan and the resulting ETV verification report(s) will be subjected to review by the mobile mass spectrometer vendors, EPA, and expert peer reviewers. These reviews will assure that this test/QA plan and the resulting verification report(s) meet the needs of potential users of mobile mass spectrometers. Performance data for the mobile mass spectrometers, collected under conditions where the quality control requirements shown in Table 8 were met, will be presented in the final verification report without any further comment. Performance data and reference measurements that do not meet these criteria will be noted and a discussion of the possible impact of the failed requirements on the performance evaluation will be presented in the final verification report. The final verification report(s) will be submitted to EPA in Word Perfect and Adobe pdf format and subsequently posted on the ETV website.

SECTION E

HEALTH AND SAFETY

E1 STANDARD/ TEST SAMPLE PREPARATION

With the exception of field testing, all handling of solid materials and highly concentrated aqueous solutions of target contaminants and potential interferents will be performed 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 sent out through Battelle Waste Operations.

E2 SAMPLE HANDLING DURING VERIFICATION TEST

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

- All containers shall be stored and transported in double containment.
- Safety glasses, nitrile gloves with long cuffs, and a chemical resistant disposable lab coat shall be worn when handling either chemical. Gloves shall be immediately changed if they become contaminated.

E3 TESTING WITH GB, GD, and VX

Verification activities using these CWA will be performed at the HMRC following their internal SOPs for handling and using CWA.

E4 DECONTAMINATION of INSTRUMENTS AFTER CWA TESTING

The return of part or all of the verified instruments is based on current U.S. Army guidelines (which is subject to change). After testing with CWA has been completed, the decontamination of the mobile mass spectrometers will be performed according to HMRC SOP III-007-07[16]. All consumables (e.g., column, inlet liner) that come into contact with liquid CWA or long-term vapor contamination of CWA will be discarded. While all components of the mobile mass spectrometers and associated equipment (including those used in sample preparation) that are in the direct flow path of the dilute agent solution or vapor will be considered for decontamination, an effort to maintain the integrity of the non-consumable parts of the mobile mass spectrometers will be made to the extent allowable by the SOP or the HMRC Bailment Agreement. For example, the detector will not be initially removed from the instrument since the mass spectrometer effectively destroys the CWA during routine analysis.

After removing consumables, the instrument will be bagged or contained for a minimum of 4 hours at 70EF or above. Testing will be conducted to ensure that vapor concentrations of CWA do not exist above the Worker Population Limit (WPL) for each agent inside the bag. If concentrations for CWA in the bag exceed the WPL, the mobile mass spectrometers may be further disassembled and tested until the subject instrument can satisfy the threshold and safely be returned to the vendor.

SECTION F

REFERENCES

1. Quality Management Plan for the ETV Advanced Monitoring Systems Center, Version 5.0, U.S. EPA Environmental Technology Verification Program, Battelle, Columbus, Ohio, March 2004.
2. Environmental Technology Verification Program Quality Management Plan, EPA/600/R-03/021, U.S. Environmental Protection Agency, Cincinnati, Ohio, December 2002.
3. Chemical Surety, United States Army Regulation, AR50-6, June 26, 2001.
4. Battelle Chemical Safety Information Program, Safety and Industrial Hygiene Manual (SIH-PP-005), September 24, 2004.
5. Hazardous Materials Research Center (HMRC) Chemical Hygiene Plan, March 2005.
6. EPA Method 180.1. Turbidity (Nephelometric), Methods for the Determination of Inorganic Substances in Environmental Samples EPA-600-R-93-100. 1993.
7. American Public Health Association, et al. Standard Methods for Examination of Water and Wastewater. 19th Edition. 1997. Washington D.C.
8. EPA 600/4-79-020. Methods for Chemical Analysis of Water and Wastes. March 1983.
9. EPA 600/R-95-131. EPA Method 524.2. Purgeable Organic Compounds by Capillary Column GC/Mass Spectrometry. Methods for Determination of Organic Compounds in Drinking Water, Supplement III. August 1995.
10. EPA 600//R-95-131. EPA Method 552.2. Haloacetic Acids and Dalapon by Liquid-Liquid Extraction, Derivatization and GC with Electron Capture Detector. Methods for the Determination of Organic Compounds in Drinking Water, Supplement III. August 1995.
11. Battelle SOP MDAS.I-009-Draft, Sample Chain-of-Custody.

12. Battelle SOP HMRC-IV-118-05: Standard Operating Procedure for the Determination of CA in Wastewater.

13. EPA SW-846 Method 8141A Organophosphorus Compounds by Gas Chromatography: Capillary Column Technique.

14. EPA 600/R-95-131. EPA Method 515.1. Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector. Methods for Determination of Organic Compounds in Drinking Water, Supplement III. August 1995.

15. Battelle SOP HMRC-III-007-07: Standard Operating Procedure for the 3X and 4X Materials Proof of Decontamination.