

**Environmental Technology  
Verification Program  
Advanced Monitoring  
Systems Center**

Quality Assurance Project Plan  
for Verification of  
Underground Storage Tank  
Automatic Tank Gauging  
Leak Detection Systems



Verification of  
Underground Storage Tank  
Automatic Tank Gauging  
Leak Detection Systems

**August 9, 2011**

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**SECTION A**  
**PROJECT MANAGEMENT**

**A1 VENDOR APPROVAL PAGE**

ETV Advanced Monitoring Systems Center

Quality Assurance Project Plan  
for Verification of  
Underground Storage Tank  
Automatic Tank Gauging  
Leak Detection Systems

APPROVAL of this QAPP and  
participation in the following tests  
as indicated with a 'yes' or a 'no':

1. \_\_\_\_\_ Water ingress detection of continuous water ingress with a splash or without a splash (Continuous),
2. \_\_\_\_\_ Water ingress detection of a quick water dump, then a fuel dump (Quick Dump),
3. \_\_\_\_\_ Water ingress and fuel leak detection during water ingress and fuel egress (Water Ingress + LD), and
4. \_\_\_\_\_ Fuel leak detection (LD).

Name \_\_\_\_\_

Company \_\_\_\_\_

Date \_\_\_\_\_

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## A4 LIST OF ACRONYMS AND ABBREVIATIONS

ADQ	Audit of Data Quality
AMS	Advanced Monitoring Systems
ASTM	American Society for Testing and Materials
ATG	automatic tank gauging
COC	chain of custody
CCV	continuing calibration verification
CFR	Code of Federal Regulation
D	difference
DQO	data quality objective
DVR	digital video recorder
E0	100% gasoline
E10	fuel that is 10% ethanol and 90% gasoline, by volume
E15	fuel that is 15% ethanol and 88% gasoline, by volume
E85	fuel that is 85% ethanol and 15% gasoline, by volume
EISA	Energy Independence and Security Act
EPA	Environmental Protection Agency
ETV	Environmental Technology Verification
gal/hr	gallon/hour
k	tolerance coefficient
JHA	job hazard analysis
LD	leak detection
LRB	laboratory record book
MLC	minimum water level change
NIST	National Institute of Standards and Technology
NWGLDE	National Work Group on Leak Detection Evaluations
OUST	Office of Underground Storage Tanks
PEA	performance evaluation audit
PDF	portable document format
PO	Project Officer
QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
QM	Quality Manager
QMP	Quality Management Plan
RCRA	Resource Conservation and Recovery Act
RMO	Records Management Office
SD	standard deviation
SOP	standard operating procedure
SRM	standard reference material
TL	tolerance limit
TSA	technical systems audit
$\bar{x}$	mean

UST        underground storage tank  
Var        variance  
VTC        Verification Test Coordinator



## **A5 VERIFICATION TEST ORGANIZATION**

Oversight of the verification test will be provided by the U.S. Environmental Protection Agency (EPA) through the Environmental Technology Verification (ETV) Program. It will be performed by Battelle, which manages 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, with the participation of the vendors who will be supplying automatic tank gauging (ATG) technology for performance verification. The verification test will be performed using laboratory facilities (i.e., Battelle's West Jefferson Campus in Ohio) under highly-controlled conditions, selected field sites (e.g., existing distribution stations) under partially-controlled conditions, or a combination of both. Verification tests will require the participation of technology vendors and Battelle staff. Vendors, expert peer reviewers, EPA AMS Center management review the quality assurance project plan (QAPP), verification reports, and verification statements. The QAPP and verification statements are approved by the EPA AMS Center Management.

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. Quality Assurance (QA) oversight will be provided by the Battelle Quality Manager (QM) and also by the EPA AMS Center Quality Manager (EPA QM), at his/her discretion. This verification test is Quality Category II which requires a quality assurance (QA) review of 25% of the test data (See section C1).

### **A5.1 Battelle**

Ms. Anne Gregg is the AMS Center's Verification Test Coordinator (VTC) for this test. In this role, Ms. Gregg will have overall responsibility for ensuring that the technical, schedule, and cost goals established for the verification test are met. Specifically, Ms. Gregg will:

- Prepare the draft QAPP, verification reports, and verification statements;

- Establish a budget for the verification test and manage staff to ensure the budget is not exceeded;

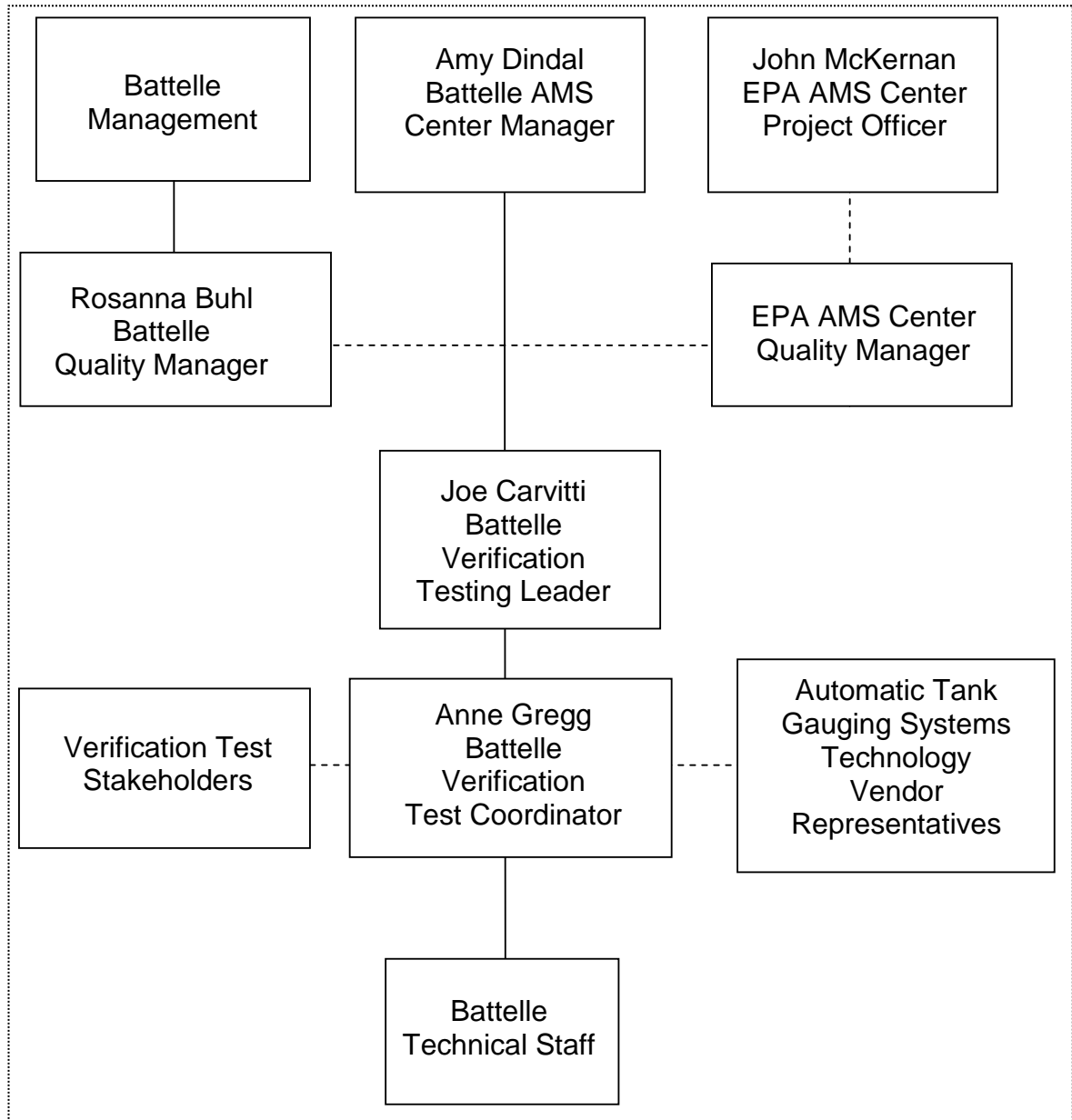


Figure 1. Organization Chart for the Verification Test

- Revise the draft QAPP, verification reports, and verification statements in response to reviewers' comments;
- Assemble a team of qualified technical staff to conduct the verification test;
- Direct the team in performing the verification test in accordance with this QAPP;
- Hold a kick-off meeting approximately one (1) week prior to the start of the verification test to review the critical logistical, technical, and administrative aspects of the verification test. Responsibility for each aspect of the verification test will be reviewed to ensure each participant understands his/her role;
- Ensure that all quality procedures specified in this QAPP and in the AMS Center Quality Management Plan<sup>1</sup> (QMP) are followed;
- Serve as the primary point of contact for vendor representatives;
- Ensure that confidentiality of sensitive vendor information is maintained;
- Assist vendors as needed during verification testing;
- Become familiar with the operation and maintenance of the technologies through instruction by the vendors, if needed;
- Respond to any issues raised in assessment reports, audits, or from test staff observations, and institute corrective action as necessary;
- Coordinate distribution of the final QAPP, verification reports, and verification statements; and
- Respond to QAPP deviations and any issues raised in assessment reports, audits, or from test staff observations, and institute corrective action as necessary.

Mr. Joe Carvitti is the Verification Testing Leader for this test. Mr. Carvitti will:

- Support Ms. Gregg (Verification Test Coordinator) in preparing the QAPP;
- Review the draft and final QAPP;
- Attend the verification test kick-off meeting;
- Ensure that confidentiality of sensitive vendor information is maintained;
- Support Ms. Gregg in responding to issues raised in assessment reports and audits;  
and
- Review the draft and final verification reports and verification statements.

Ms. Amy Dindal is Battelle's Manager for the AMS Center. As such, Ms. Dindal will oversee the various stages of verification testing. Ms. Dindal will:

- Review the draft and final QAPP;
- Attend the verification test kick-off meeting;
- 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 Ms. Gregg in responding to any issues raised in assessment reports and audits;
- Maintain communication with EPA's technical and quality managers; and
- Issue a stop work order if Battelle or EPA QA staff discover any situation that will compromise test results.

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

- Assist in planning for the test and making arrangements for the receipt of and training on the technologies;
- Attend the verification test kick-off meeting;
- Assist vendor staff as needed during technology receipt and training;
- Independently acquire technology for verification testing, if necessary;
- Arrange for and/or acquire adequate fuel supplies, equipment, and facilities/locations for performing verification tests and disposing of generated wastes;
- Conduct verification testing using the vendor's or other acquired technology;
- Collect the data and samples during verification testing;
- Conduct analytical methods to determine the ethanol and water content of the fuel;
- Perform statistical calculations specified in this QAPP on the technology data as needed;
- Provide results of statistical calculations and associated discussion for the verification reports as needed;

- Support Ms. Gregg in responding to any issues raised in assessment reports and audits related to statistics and data reduction as needed; and
- Conduct and observe verification testing on-site, as appropriate. Immediately report deviations to this QAPP to the VTC.

Ms. Rosanna Buhl is the Battelle QM for the AMS Center. Ms. Buhl will:

- Review the draft and final QAPP;
- Attend the verification test kick-off meeting and lead the discussion of the QA elements of the kickoff meeting checklist;
- Prior to the start of verification testing, verify the presence of applicable training records, including any vendor training on test equipment;
- Conduct a technical systems audit at least once during the verification test.
- Conduct audits to verification data quality;
- Prepare and distribute an audit report for each audit;
- Verify that audit responses for each audit finding and observation are appropriate and that corrective action has been implemented effectively;
- Provide a summary of the quality assurance/quality control (QA/QC) activities and results for the verification reports;
- Review the draft and final verification report(s) and verification statement(s);
- Communicate to the VTC and/or technical staff the need for immediate corrective action if an audit identifies QAPP deviations or practices that threaten data quality;
- Delegate QA activities to other Battelle quality staff as needed to meet project schedules;
- Review and approve QAPPs, QAPP amendments, deviations and audit reports;
- Work with the VTC and Battelle's AMS Center Manager to resolve data quality concerns and disputes; and
- Recommend a stop work order if audits indicate that data quality or safety is being compromised.

## **A5.2 Technology Vendors**

The responsibilities of the technology vendors, which includes representatives designated by the technology vendor or technology owners who have submitted the technology for testing, are as follows:

- Review and provide comments on the draft QAPP;
- Accept (by signature of a company representative) the final QAPP prior to test initiation to confirm that the conditions of the test are understood. The vendor has the option to participate in any or all of the four main tests presented in this QAPP. Provide technology for evaluation during the verification test;
- Supply training on the use of the technology and provide written consent and instructions for test staff to carry out verification testing, including written instructions for routine operation of their technology;
- Provide maintenance and repair support for the technology, on-site if necessary, throughout the duration of the verification test; and
- Review and provide comments on the draft verification report and statement for the respective technology.

## **A5.3 EPA AMS Center**

EPA's responsibilities in the AMS Center are based on the requirements stated in the "Environmental Technology Verification Program Quality Management Plan" (ETV QMP)<sup>2</sup>.

The roles of specific EPA staff are as follows:

For the verification test, the EPA's AMS Center QM will:

- Review the draft QAPP;
- Perform at his/her option one external technical systems audit during the verification test;
- Notify the EPA AMS Center Project Officer (PO) of the need for a stop work order if the external audit indicates that data quality is being compromised;
- Prepare and distribute an assessment report summarizing results of the external audit;
- Perform audits of data quality;

- Notify the EPA AMS Center PO of the need for a stop or modify work order if the audit of data quality indicates that data quality is being compromised; and
- Review draft verification reports and verification statements.

Dr. John McKernan is EPA's PO for the AMS Center. Dr. McKernan, or designee will:

- Review the draft QAPP;
- Approve the final QAPP;
- Review the draft verification reports and verification statements;
- Oversee the EPA review process for the QAPP, verification reports, and verification statements;
- Coordinate the submission of verification reports and verification statements for final EPA approval;
- Post the QAPP, verification reports, and verification statements on the ETV web site; and
- Be available during the verification test to review and authorize any QAPP deviations by phone and provide the name of a delegate to the Battelle AMS Center Manager should he not be available during the testing period.

#### **A5.4 Verification Test Stakeholders**

A Technical Panel of stakeholders was specifically assembled for the preparation of this QAPP. Appendix A presents a list of participants in the Technical Panel. The panel includes representatives from industry associations, state and federal governments, and users, including representatives of the National Work Group on Leak Detection Evaluations (NWGLDE). A Vendor Panel was separately formed to acquire input from technology vendors. Appendix A also presents a list of participants in the Vendor Panel. These groups represent the Verification Test Stakeholders for this evaluation. The responsibilities of verification test stakeholders and/or peer reviewers include:

- Participate in technical panel discussions (when available) to provide input to the test design;
- Review and provide input to the QAPP; and
- Review and provide input to the verification report(s)/verification statement(s).

Finally, this QAPP and the verification report(s) and verification statement(s) based on testing described in this document will be reviewed by experts in the fields related to underground storage tank (UST) leak detection (LD) and statistics. The following experts have agreed to provide peer review:

- Randy Jennings, Tennessee Department of Agriculture,
- Samuel Gordji, University of Mississippi and SSG Associates, and
- James Weaver, EPA, National Exposure Research Laboratory.

## **A6 BACKGROUND**

The ETV Program's AMS Center conducts third-party performance testing of commercially available technologies that monitor, sample, detect, and characterize contaminants or naturally occurring species across all matrices. The purpose of ETV is to provide objective and quality assured performance data on environmental technologies so that users, developers, regulators, and consultants can make informed decisions about purchasing and applying these technologies. Stakeholder committees of buyers and users of such technologies recommend technology categories, and technologies within those categories, as priorities for testing.

According to U.S. EPA Office of Underground Storage Tanks (OUST), an estimated 600,000 USTs are operated in the United States, all of which are required to utilize some type of LD technology that meets federal performance requirements. The current EPA protocols for evaluating these technologies were developed in the early 1990's before biofuel use became widespread. This QAPP is not replacing the existing ATG systems protocol<sup>3</sup>. Rather, the QAPP expands the existing protocol to incorporate the relevant fuel blends of today. Currently ethanol is blended into 90% of all gasoline consumed in the United States at percentages ranging from 10% (E10) to 85% (E85) ethanol<sup>4</sup>. E85 is also commonly called flex fuel; however, in this document, it is referred to as E85. Biofuel consumption is expected to increase in response to Energy Independence and Security Act (EISA) requirements for biofuel production and use. Because petroleum and ethanol have very different chemical and physical characteristics, LD technologies that operate based on density, conductivity, refractive index, or other properties may not function properly in the new ethanol blend environment. Questions have also been raised about the long-term performance of new and existing LD devices due to the corrosive nature of ethanol, although long-term material compatibility will not be evaluated in this QAPP.



It is important to understand whether deficiencies exist in current LD protocols that do not account for the physical and chemical properties of ethanol-blended fuels. Several different LD technologies are used to monitor USTs for possible fuel leaks. One of the most common types is ATG technology. The purpose of this QAPP is to specify procedures for a verification test applicable to commercial ATG LD technologies for USTs containing ethanol blends. The purpose of the verification test is to evaluate the performance of ATG technologies by challenging them under combinations of variables. The variables are related to establishing the test condition desired by changing different variables (ethanol content, fuel height, etc). Future QAPPs are planned to evaluate other LD technologies such as pipeline LD systems or interstitial monitors.

### **A6.1 Technology Need**

LD technologies are required by federal regulation to ensure that USTs are not leaking the fuel product into the environment. EPA LD requirements for USTs are found in 40 Code of Federal Regulation (CFR) 280.40 and require written determination of LD equipment performance. According to this regulation, the equipment must be able to detect a leak from any portion of the tank or underground piping that routinely contains product and be installed, calibrated, operated, maintained, and checked for operability in accordance with the vendor's instructions. In addition, certain release detection methods such as ATGs must detect a 0.20 gallon per hour (gal/hr) fuel leak rate and the presence of water in the tank. Thus, the conditions of performance certification and the equipment vendor's maintenance requirements become compliance requirements. For leak detection and water ingress detection, this QAPP addresses the following requirements. ATGs are expected to:

- be capable of detecting a leak of 0.20 gal/hr with a probability of (at least) 95%, while operating at a false alarm rate of 5% or less; and
- measure any water in the bottom of the tank at least once a month to the nearest 1/8 inch.

Prior to being offered commercially, water detection systems must establish the minimum water level that the system can detect and determine the smallest change in the water level that the system can reliably measure.

## A6.2 Technology Description

An ATG system consists of a sensing probe and a display/recording console. The QAPP addresses evaluation of the sensing probe, but the console is also needed to make the LD system fully operational. Table 1 lists the various types of ATG systems in use and their principles of operation. According to discussions with the Verification Test Stakeholders, the magnetostrictive probe is the most widely used in the industry. In addition, the capacitance probe is no longer produced or sold; however, it is reportedly still in use.

**Table 1. Automatic Tank Gauging Technologies and LD Operating Principles**

Technology Type	Operating Principle
Magnetostrictive Probe	A wire sensor inside a stainless steel rod detects the presence of a magnetic field, which indicates the height of a float.
Ultrasonic/Acoustic Methods (speed)	A sensor detects sound wave echoes reflected from an interface of water/fuel or fuel/air to calculate the liquid level based on the speed of sound in the media.
Mass Buoyancy/Measurement System	The buoyancy of a probe is detected on a load cell and compared to the tank geometry to calculate the liquid level.
Capacitance Probe	Detection is based on the dielectric property of the stored liquid.
Water Level Float (part of the magnetostrictive technology)	Buoyancy of float allows the signal generated (e.g., magnetic field) to coincide with the top of the liquid layer based on the liquid density in comparison to float density.

## A7 VERIFICATION TEST DESCRIPTION AND SCHEDULE

This verification test will assess the performance of ATG systems relative to key verification parameters including accuracy, probability of false alarm, and precision. These performance parameters will be evaluated using multiple variables that will challenge the ATG's ability to detect fuel leaking out of or water entering into an UST. In performing the verification test, Battelle will follow the technical and QA procedures specified in this QAPP and will comply with the data quality requirements in the AMS Center QMP<sup>1</sup>. This verification test is an EPA designated QA Level II verification.

## A7.1 Verification Test Description

Specific procedures described herein are based on input received from the Verification Test Stakeholders, the procedures described in the current EPA protocol for ATG systems<sup>3</sup>, and the performance requirements found in 40 CFR 280. The ATG technology evaluation is organized as four main tests. The vendors have the option to commit to participating in one of, or up to all four of the tests. The tests to be conducted will be determined as part of the vendor's approval of this QAPP (see Page 3). Each test evaluates the performance of the ATG to operate under different experimental environments. The four tests are:

1. Water ingress detection of continuous water ingress with a splash or without a splash (Continuous);
2. Water ingress detection of a quick water dump, then a fuel dump (Quick Dump);
3. Water ingress and fuel leak detection during water ingress and fuel egress (Water Ingress + LD); and
4. Fuel leak detection (LD).

The water ingress detection portions of the evaluation are much more extensive than the original protocol<sup>3</sup> due to the complex interactions between gasoline, ethanol, and water. The water ingress tests (Tests 1 through 3) will be performed in a laboratory test vessel that simulates the tank environment in a controlled manner. The fuel and water interactions and the ATG responses will be video recorded, and the fuel properties will be either controlled or monitored. The test to evaluate LD capabilities only (Test 4) will be conducted in the field in an UST at a service or blending station.

The performance of the ATG technologies will be verified based on the following performance parameters.

- Water Ingress Detection Ability
  - Accuracy
  - Sensitivity
  - Precision
  - Phase differentiation
  - Operational factors
- Fuel LD Ability
  - Accuracy

- Precision
- False alarm rate
- Operational factors

The responses for these parameters will be collected from the technologies as either a “detect” or “non-detect” or if determined by the technology, as a nominal leak rate for the water ingress and fuel leak run results. An independent comparison to metered rates will be used to confirm the true water ingress rates and fuel leak rates established during testing.

The tests will be performed with the technologies operating in accordance with the vendor’s recommended procedures as described in the user’s instructions/manual or during training provided to the operator. Similarly, calibration and maintenance of the technologies will be performed as specified by the vendor. Results from the technologies being verified will be recorded electronically by the technology display/recording console and/or manually in laboratory record books (LRBs) and test data sheets. Appendix B presents an example test data sheet.

A verification report describing the results obtained during the evaluation will be drafted for each vendor participating in the verification test. A verification statement summarizing the results will be drafted for each technology tested in the verification test. Each report and verification statement will be reviewed by the participating vendor, EPA, and the peer reviewers. In performing the verification tests, Battelle will follow the technical and QA procedures specified in this QAPP and comply with the data quality requirements in the AMS Center QMP<sup>1</sup>.

Quality procedures include a Technical Systems Audit (TSA) and Audits of Data Quality (ADQ). The Battelle QM or her designee will perform the TSA. The first batch of data will be delivered to the vendors and EPA within 30 days of test initiation. Because of the testing options, it is unknown how many tests will be performed during the first month of testing; all data collected during the first 2 weeks of testing will be considered the first batch of data. Unaudited data will include the disclaimer “has not been reviewed by Battelle QM.” The first ADQ will review the first batch of data delivered. A second ADQ will be performed once all data are collected, and a final ADQ will be performed on the reports and verification statements. More detail is provided in Section C.

## **A7.2 Verification Test Schedule**

Table 2 shows a general schedule of testing and data analysis/reporting activities to be conducted in this verification test.

## **A7.3 Test Site Descriptions**

As presented in Section B1, test conditions for some of the tests will involve use of gasoline/ethanol fuel blends that are just beginning to be dispensed into vehicles. In addition, tests designed to verify the functionality of water detection technology may produce a mixture that is not suitable for use as a motor fuel. Although accommodations could be made to simulate the desired test conditions in an actual field storage situation, the fact that some of the conditions are not observed in USTs (e.g., E15 fuel blend) suggests that the tests are best performed in a research facility under highly-controlled conditions.

The optimum means to achieve the desired results stated above is to structure the tests so that visual results can be obtained and conditions that influence ATG performance are controlled. Therefore, the laboratory test vessel used for this evaluation will have a diameter similar to an UST and the ability to visualize the testing and withdrawal of samples from the test vessel. USTs have diameters ranging in size from approximately 4 feet (approximately 1000-gal capacity) to 10.5 feet (approximately 25,000-gal capacity).

**Table 2. General Verification Test Schedule**

Approximate Months after Start Date	Testing Activities	Data Analysis and Reporting
0 to 3	<ul style="list-style-type: none"> <li>Fabricate test equipment</li> <li>QAPP revision (if necessary for technology- or site-specific considerations) and approval</li> <li>Conduct pre-test checks and dry runs</li> </ul>	<ul style="list-style-type: none"> <li>Not Applicable</li> </ul>
3	<ul style="list-style-type: none"> <li>Coordinate for technologies and testing supplies to be delivered to testing sites</li> <li>Install necessary equipment and technology</li> <li>Technology training by vendor or coordination with vendor representative</li> </ul>	<ul style="list-style-type: none"> <li>Prepare report template</li> </ul>
3 to 5	<ul style="list-style-type: none"> <li>Perform Performance Evaluation Audit (PEA)</li> <li>Complete PEA report</li> <li>Conduct verification testing</li> <li>Perform TSA</li> <li>Perform initial ADQ (1<sup>st</sup> batch, see Section B10)</li> <li>Complete verification testing</li> <li>Perform second ADQ (25% of all data)</li> </ul>	<ul style="list-style-type: none"> <li>Compile PEA results</li> <li>Compile data</li> <li>Review and summarize data</li> <li>Perform data analysis</li> <li>Begin draft reports</li> </ul>
5	<ul style="list-style-type: none"> <li>Prepare draft verification report(s) and statement(s)</li> <li>Perform third ADQ of report(s) and statement(s)</li> </ul>	<ul style="list-style-type: none"> <li>Complete draft verification report(s) and statement(s)</li> <li>Complete internal review of draft report(s) and verification statement(s)</li> </ul>
6 to 7	<ul style="list-style-type: none"> <li>Coordinate reviews of draft verification report(s) and statement(s)</li> </ul>	<ul style="list-style-type: none"> <li>Complete peer review and vendor review of draft report(s)</li> </ul>
8	<ul style="list-style-type: none"> <li>Prepare final verification report(s) and statement(s)</li> </ul>	<ul style="list-style-type: none"> <li>Revise draft verification report(s) and statement(s)</li> <li>Submit final verification reports(s) and statement(s) for EPA approval</li> </ul>

The full length of a typical tank need not be constructed, as adequate mixing and ATG probe behavior can occur in a shorter section, approximately 3 to 5 feet in length, provided that the actual diameter is maintained. This approach will yield several advantages to testing: 1) the volume of waste generated when water mixes with ethanol or gasoline during the operation will be minimized when using a less-than-full-length test vessel, 2) tilt angle can be controlled in the laboratory, thus eliminating error due to varying product or water height within the length of the test vessel, 3) fuel temperature and vibration can be controlled to eliminate reading errors from these external influences, and 4) the laboratory test vessel adds the advantage that testers can visually observe the physical behavior of a dense phase that has been reported to confound tank and ATG equipment operators and vendors.

These laboratory tests are anticipated to be performed in Battelle facilities in West Jefferson, Ohio. When verification tests are performed under field conditions, they will be performed at fueling stations or blending sites where the ATG technologies to be evaluated have been installed or can be installed.

#### **A7.4 Health and Safety**

Battelle will conduct all verification testing and OP measurements following the safety and health protocols in place for the locations used for testing. In addition, a job hazard analysis (JHA) will be performed to describe the specific hazards associated with gasoline and ethanol, as well as the use of engineering controls and other procedures required to reduce the possibility of potential mishaps. These include maintaining an explosion-proof work environment, providing secondary containment for all storage vessels, and promoting a current awareness of safe chemical and waste handling methods. Proper personal protective equipment will be worn, and safe laboratory practices will be followed. Standard Battelle JHA forms will be completed once the test locations and hazardous activities are defined. The JHA forms will discuss the following topics, in addition to others:

- Fuel handling and safety procedures;
- Ventilation procedures;
- Waste handling and labeling; and
- Use of explosion-proof equipment.

The JHA forms will be physically present at the location where verification testing is being conducted. All test participants will be required to review and understand the JHA forms prior to initiating laboratory or field work and adhere to its procedures during conduct of all verification tests.

### **A8 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA**

In performing the verification test, Battelle will follow the technical and QA/QC procedures specified in this QAPP and comply with the data quality requirements in the AMS Center QMP.<sup>1</sup> Data quality objectives (DQOs) have been established as test conditions to ensure that this verification test provides suitable data for a robust evaluation of performance. The DQOs are used to develop the testing variable tolerances required to meet the objectives of the

verification test. The DQOs for this verification test were established to assess the performance of the ATG technologies and their abilities to detect water ingress and fuel leaks in USTs containing ethanol-blended fuels. For this verification test, the DQOs are evaluated by the acceptance criteria determined in Section B5 to detect fuel leakage and water ingress. The DQOs of this verification test are to:

- Evaluate the ability of ATGs to detect the presence of water entering an UST according to the requirements for USTs found in 40 CFR 280.40 when in use with ethanol-blended fuels.
- Evaluate the ability of ATGs to detect a fuel leak out of an UST according to the requirements for USTs found in 40 CFR 280.40 when in use with ethanol-blended fuels.

Assessing the DQOs is also a key component of the ETV PEA process. PEAs will be used to independently confirm the accuracy of the analytical measurements. PEAs will also check the calibration for equipment used to deliver the fuel or water into the test vessel. Section C1.1 describes the PEA.

The Battelle QM or her designee will perform a TSA at least once during this verification test to verify that testing and analysis were performed according to the QAPP. The EPA QM also may conduct an independent TSA, at his/her discretion.

## **A9 SPECIAL TRAINING/CERTIFICATION**

Documentation of training related to technology testing, analytical method analysis, operation of ancillary equipment used to collect supporting data, routine laboratory procedures, and reporting is maintained for all Battelle technical staff in training files at their respective Battelle location. The Battelle QM will verify the presence of appropriate training records prior to the start of testing. The vendors will train the Battelle technical staff prior to the start of testing. Battelle will document this training with a consent form, signed by the vendor or designated representative, which identifies Battelle technical staff who have been trained to use their technologies and can train other staff. In the event that other staff members are required to use the technologies, they will be trained by staff trained by the vendors. All technical staff will have a minimum of a bachelor's degree in science/engineering or equivalent work experience (e.g., experience or training using ATGs).



Battelle will conduct all verification testing using the engineering controls and safety procedures described in the JHA forms and will document that all testing staff and vendors have read, understood, and agreed to adhere to the procedures described in the JHA prior to and during work on the test.

## **A10 DOCUMENTATION AND RECORDS**

The documents for this verification test will include the QAPP, vendor instructions, verification report(s), verification statement(s), and audit reports. Project records will include: laboratory record books (LRBs); data collection forms; supporting laboratory records, training records, electronic files (both raw data and spreadsheets), JHA forms, and QA audit files. Table 3 summarizes the types of data to be recorded. The raw and final results from the variable measurements will be collected by Battelle, and technology data will be downloaded and/or printed from the display/recording console. Section B10 details the data recording practices and responsibilities. Documentation of Battelle staff training by vendors and copies of other project specific training will also be included in the project files. All of these records will be maintained at the test site or in the VTC's office during the test and then transferred to permanent storage at Battelle's Records Management Office (RMO) at the conclusion of the verification test.

All data generated during the conduct of this project will be recorded directly, promptly, and legibly in ink. All data entries will be dated on the date of entry and signed or initialed by the person entering the data. Any changes will be made so as not to obscure the original entry, dated and signed or initialed at the time of the change, and indicate the reason for the change. Project-specific data forms will be developed prior to testing to document critical information in real time. The draft forms will be provided to the Battelle QM for review.

**Table 3. Summary of Records to be Collected and Maintained<sup>(a)</sup>**

<b>Record/Data</b>	<b>Where Recorded</b>	<b>How often recorded</b>	<b>Disposition of Data</b>
Dates, times of test events, technology model and console	LRBs or data collection sheets	Start/end of test run, and at each change of a variable	Use to organize/check test results; manually incorporate in data spreadsheets
Test variables	LRBs, data collection sheets, or video recording	When set or changed, or as needed to document notable details during testing	Use to organize/check test results; manually incorporate in data spreadsheets, or visually record test
Technology data	Electronically within and on thermal paper in the ATG console	During each test run	Use to document and interpret performance of the ATG technology
Field data (Test 4 only)	LRBs or data collection sheets	During each field test run	Use to interpret performance of the ATG technology
PEA records	LRBs or data collection sheets	Before testing begins	Use to verify the performance of the procedures to leak fluid out and into the test vessel
Analytical method sample analysis, chain of custody, and results	LRBs, chain of custody forms, data collection sheets, or data acquisition system, as appropriate	When test samples are aliquoted for the analysis and throughout sample handling and analysis process	Use to organize/check test results; manually incorporate in data spreadsheets; transfer to spreadsheets/agreed upon report; project files; retain for documentation of analytical method performance

a. Battelle is responsible for collecting and maintaining all specified records.

## SECTION B

### MEASUREMENT AND DATA ACQUISITION

#### **B1 EXPERIMENTAL DESIGN**

This verification test is designed to evaluate the functionality of the ATG systems when in ethanol-blended fuel service. The characteristics of independent variables have been selected and will be established during the runs to determine the response of the dependent variables. Performance parameters will be evaluated based on the responses of the dependent variables and used to characterize the functionality of the selected ATG systems. All technologies will be tested simultaneously to ensure the testing conditions are the same and to minimize the wasted fuel.

**Dependent Variable Responses--**The ATGs will be evaluated with respect to their ability to properly respond to the presence or absence of a leak. Detection of a leak, either water ingress or a fuel leak, thus represents the dependent variable included in the test. The actual dependent variable considered will depend on the test procedure being evaluated.

**Independent Variable Levels--**The levels of the independent variables will be established to simulate conditions expected to be found in operating USTs. The water ingress detection tests and the fuel LD test will consider different independent variables.

One to four tests will be performed to evaluate the ATG technology performance with respect to its ability to detect water leaking into (water ingress detection) and fuel leaking out (fuel LD) of the UST. The independent variables included in the tests and the levels for each variable will depend on the environment the test is simulating. These variables will be varied to achieve different conditions for the ATG systems to operate within. All water ingress tests will be performed in the laboratory. Laboratory tests will be performed in a laboratory test vessel that is between 3 and 5 feet in length, thus preserving important physical tank features that impact ATG technology response. The test to evaluate the fuel LD of the ATG systems will employ a UST at a gasoline filling or blending station.

Common to all four tests is the fuel used for testing, more specifically, the ethanol content of the fuel. The ethanol content variable will be 0% (E0), 15% (E15), or 85% (E85). The E0 fuel will serve as an operational baseline for the ATGs. The fuel ethanol content is designed to low and high ethanol contents. The low end is represented by E15 as a result of the

EPA E15 Waiver (<http://www.regulations.gov/#!documentDetail;D=EPA-HQ-OAR-2010-0448-0001>). The E85 represents an existing high-end blend in use.

Prior to testing, the percent ethanol will be verified analytically using ASTM D4815<sup>5</sup> for E0 and E15 and ASTM D5501<sup>6</sup> or an equivalent method for E85. As per the ASTM method, measured sample levels must be within 10% of the nominal concentration before each test run (for example, the acceptable percent ethanol of E15 is 13.5 to 16.5%). For the water ingress tests, the water content of the fuel and the ethanol-water mixture (dense phase) will be determined using either Karl-Fischer titration methods 203 or 1064.

The vendor has the option to choose any or all of the four main tests for this evaluation. The four tests are described in detail as follows.

**1. Water ingress detection of continuous water ingress with a splash or without a splash (Continuous).**

The water ingress tests are focused on the mixing method and rate of water addition into the test vessel. In the first test, a continuous stream of water will be introduced into the laboratory test vessel to produce a splash on the surface of the fuel or to not produce a splash by trickling the water along a surface of the tank or riser pipe to slowly meet the surface of the fuel. The independent variables and levels for the continuous water ingress test are:

- Fuel ethanol content (3 levels): E0, E15, and E85;
- Water ingress method/rate (2 levels): with splash and without splash; and
- Fuel height (2 levels): 25% and 90% full.

The water ingress method/rate was selected to establish conditions that impact the degree of mixing that occurs in a tank using the three ethanol blends. In these runs, the vendor-stated amount of water that is needed to trigger a response by the water detection technology (absent any adsorption) will be calculated based on the curvature of the test vessel. This calculated volume represents the threshold height of water that theoretically should be detectable by any of the technologies. An initial volume of water equal to approximately 75% of this calculated amount will be placed in the vessel prior to beginning the run to allow a response to be observed in less time than if the entire calculated volume had to be added after the test begins. The amount introduced as a water ingress rate will then be varied as follows and will be specifically determined during the pre-checks and dry runs.

- A continuous water ingress rate that causes a splash on the surface of the fuel. The rate will be established such that vendor-stated threshold height of water that can be detected (absent any adsorption) will be produced within approximately 1 hour (after having an initial height of 75% of the threshold height). This water addition rate will be continued beyond 1 hour until a response in the water detection technology is observed. If no response is observed in 3 hours, the test will be terminated. With this rate of water ingress, some mixing may occur due to splash mixing (depending on the height of fuel in the vessel) and some mixing may occur by diffusion. The extent of mixing by these two mechanisms may be influenced by independent variables and may cause adsorption of water into the ethanol along with subsequent phase separation of the mixture.
- A continuous water ingress rate that follows along the inside wall of the test vessel or the riser pipe with minimal agitation to the surface of the fuel. The rate will be established using the same procedure as above, except it will occur over approximately 2 hours. The test condition will be maintained until a response in the water detection technology is observed, or terminated after 3 hours if there is no response. With this rate of water ingress, most of the mixing is expected to occur by diffusion. The run termination times are established to be the same, because it is expected that this time interval encompasses the potential for the technology to detect the water with both ingress rates.

To address the second requirement of water detection, once the water detection technology has reacted to the minimum water height, the smallest increment in water height that can be measured will be determined. The ingress rate will be increased to produce a calculated height increase at the bottom of the tank of  $1/16^{\text{th}}$  of an inch in 10 minutes. After 10 minutes the technology reading and the height of the water level will be measured and recorded. Ten 10-minute increments will be measured for each run of Test 1 (to produce approximately 100 measurements). This same flow rate will be used for all runs regardless of the initial flow rates of with or without splashing. The true increase of the water level will be measured and recorded.

Two fuel height levels are specified to establish several possible splash mixing regimes and diffusion columns. The lower fuel height will yield the greater splash mixing potential, but the shorter diffusion columns through which the water can flow. Conversely, the higher fuel

height will yield the lower splash mixing potential, but the higher diffusion column. The fill height will be established to  $\pm 1\%$  of the target height of either 25% or 90%. Data will be gathered under every combination of levels between all variables. For the E85 runs, the ratio of fuel to water will be calculated at the end of the 25% full tests. The same ratio or greater will need to be established in the 90% level, and calculations may yield a resulting volume that is greater than the capacity of the test vessel. In this case, the fill height for the 90% full runs will be reduced or the two 90% full runs will be removed from the E85 test design. In Table 4 below, runs 1 to 12 represent all combinations of the variables and runs 13 to 16 are duplicate runs. Duplicate runs were chosen to encompass the combinations of test conditions and to minimize the fuel waste.

**Table 4. Summary of Continuous Water Ingress Runs<sup>(a)</sup>**

Runs	Fuel Type	Fuel Level (%)	Water Ingress Method/Rate
1	E0	25%	With splash
2	E0	25%	Without splash
3	E0	90%	With splash
4	E0	90%	Without splash
5	E15	25%	With splash
6	E15	25%	Without splash
7	E15	90%	With splash
8	E15	90%	Without splash
9	E85	25%	With splash
10	E85	25%	Without splash
11	E85	90%	With splash
12	E85	90%	Without splash
<b>Duplicate Runs</b>			
13	E0	90%	Without splash
14	E15	25%	Without splash
15	E15	90%	With splash
16	E85	25%	With splash

a. Run numbers do not reflect the order in which runs are performed. Run order will be established during testing.

Data collected or calculated with each run will include, but are not limited to:

- Did the ATG technology detect water?
  - a. If so,
    - i. What was the independently-measured height of the water at the bottom of the tank at the beginning of the run and when the technology detected the water?
    - ii. How much time elapsed since the water ingress began?

- iii. How much water was added to the tank before the technology detected the water? (Sum of water volume added before the run to achieve 75% of vendor-stated height and the water volume added during the run.)
  - iv. What water height was reported by the technology?
- b. If not,
- i. What was the independently-measured height of the water at the bottom of the tank at the beginning of the run and when the run was aborted?
  - ii. How much time elapsed since the water ingress began?
  - iii. How much water was added to the tank before the run was aborted? (Sum of water volume added before the run to achieve 75% of vendor-stated height and the water volume added during the run.)
- Did the technology register increases in water after the initial water detection?
  - If so, for every increase (approximately 10 increases for each test run),
    - c. What was the volume of water added and what is the time interval between the initial response and the first detected increase? For later increases, what was the volume of water added and the time intervals between the most recent increase and the previous increase?
    - d. What was the water height of the increases as determined by the technology?

## 2. **Water ingress detection of a quick water dump, then a fuel dump (Quick Dump)**

This second test focuses on the potential to detect phase separation in an UST. A quick water ingress rate with a high degree of mixing will simulate addition of water in a manner that might occur if the spill bucket is dumped into the tank at a 25% fill height, then fuel will be dumped to fill the tank to a 90% fill height. This test is mainly observational in that the test vessel will be disturbed quickly with water then fuel and the response of the technology will be recorded throughout the test. There will be three runs, one for each of the fuel types being evaluated in this test. The E0 run will be run first and used as the baseline for the technology responses to establish the minimum wait time for the other two runs with E15 and E85. The stepwise approach is listed below.

1. Fill test vessel to the 25% height with the fuel product.
2. Dump 2 gallons of water into the test vessel through the riser pipe.
3. Observe and record results.

- a. If phase separation occurs and/or the water is detected by the technology, observe the separation for a wait time to be determined and then perform step.
  - b. If phase separation does not occur after a wait time to be determined based on the E0 response time, repeat step 2.
4. Add fuel product to the 90% height.
  5. Observe and record results for 2 hours or adjust wait time as needed to capture the interactions in the test vessel.
  6. Test complete.

Data collected or calculated with each run will include, but are not limited to:

1. Was phase separation observed after water dumps and before additional fuel ingress?
    - a. If so, how much total water and time was added before phase separation was observed?
  2. Was phase separation observed after the fuel dump?
    - a. If so, how long after the fuel dump was it observed?
    - b. If so, what other behaviors of the phase separation were observed after how much time?
- 3. Water ingress and fuel leak detection during water ingress and fuel egress (Ingress + LD)**

This test combines water ingress with fuel leak egression to challenge the technology to detect both situations simultaneously. Before this test begins, the stability of the laboratory test vessel must be established. The ATG technology will need to pass three tank tightness tests when the test vessel is at a 'zero' leak rate, with no water ingress. The water ingress methods/rates will be similar to the first test with a continuous stream to produce a splash or a continuous stream that follows along a surface in the test vessel that does not splash. The fuel height will be set at the 25% height for all runs during this test. The two ingress methods will be varied in combination with three different fuels, and three different leak rates. The independent variables and levels for these runs are:

- Fuel ethanol content (3 levels): E0, E15 and E85;
- Water ingress method/rate (2 levels): with splash and without splash; and
- Fuel leak rate (3 levels): 0 gal/hour, two other levels to be determined.



The water ingress method/rate was selected to establish conditions that impact the degree of mixing that occurs in a tank using the three fuel ethanol content levels. As in the previous tests an existing water bottom will be present to shorten the length of the tests. The amount of water that is needed to produce the vendor-stated water detection threshold will be calculated based on the curvature of the test vessel. This calculated volume represents the threshold height of water that was determined to be detectable by the ATG technology prior to vendor deployment. An initial volume of water equal to approximately 75% of this calculated amount (or 75% of the vendor-stated threshold level for the water detection technology) will be placed in the test vessel prior to beginning the test to allow a response to be observed in less time than if the entire calculated volume had to be added after the test begins. The amount introduced as a water ingress rate will be specifically determined during the pre-checks and dry runs and varied as follows.

- The splashing water ingress rate will be established such that vendor-stated threshold height of water that can be detected (absent any adsorption) will be produced within 1 hour (after having an initial height of 75% of the threshold height). This water addition rate will be continued beyond 1 hour until a response in the water detection technology is observed. If no response is observed in 3 hours, the test will be terminated. With this rate of water ingress, some mixing may occur due to splash mixing (depending on the height of fuel in the tank) and some mixing may occur by diffusion. The extent of mixing by these two mechanisms may be influenced by independent variables and may cause adsorption of water into the ethanol along with subsequent phase separation of the mixture.
- The non-splashing ingress rate will be established using the same procedure as the splashing ingress rate, except it will occur over 2 hours. The potential energy available for splash mixing is expected to be less, and a slow ingress should flow down the vessel wall. Ultimately, this rate is expected to produce less mixing. The test condition will be maintained until a response in the water detection technology is observed, or terminated after 3 hours if there is no response. With this rate of water ingress, most of the mixing is expected to occur by diffusion.

Finally, three leak rates are specified as 0 gal/hr and two other levels that will be experimentally determined in the pre-test checks. The leak rates will be determined to elicit

specific net changes in volume for the leak and/or water detection technologies to be able to detect. The desired net volume changes will be specified with input from the vendor at the time of the pre-test checks. Both sets of rates should be established in bench tests and dry runs to produce the desired net effect for the detection of both to be challenged independent of the regulation levels. Table 5 presents the basic and duplicate runs for this test. Duplicate runs were chosen to encompass the combinations of test conditions and to minimize the fuel waste.

**Table 5. Summary of Water Ingress + Leak Detection Runs<sup>(a,b)</sup>**

Run	Fuel Type	Water Ingress Method/rate	Fuel Leak Rate <sup>c</sup>
1	E0	With Splash	0
2	E0		TBD1
3	E0		TBD2
4	E15		0
5	E15		TBD1
6	E15		TBD2
7	E85		0
8	E85		TBD1
9	E85		TBD2
10	E0	Without Splash	0
11	E0		TBD1
12	E0		TBD2
13	E15		0
14	E15		TBD1
15	E15		TBD2
16	E85		0
17	E85		TBD1
18	E85		TBD2
<b>Duplicate Runs</b>			
19	E0	With Splash	0
20	E15	Without Splash	TBD1
21	E15	Without Splash	TBD2
22	E85	With Splash	TBD2

- a. Run numbers do not reflect the order in which runs are performed. Run order will be established during testing so as to minimize the amount of fuel handling and fuel waste produced during the test series.
- b. Fill height will be set at 25% for all runs during this test.
- c. TBD1 and TBD2 are the two unknown fuel leak rates that are to be determined to produce the desired net volume change relative to the water ingress rates.

Data collected or calculated with each run will include, but are not limited to:

1. Did the ATG detect a fuel leak or how much time elapsed before the test was aborted?
  - a. How much fuel height change was measured independently?
  - b. How much fuel height change was measured by the ATG or what fuel leak was reported by the ATG?

2. Did the technology detect water ingress?
  - a. What was the height of the water phase at the bottom of the tank after how much time?
  - b. How much water was measured by the technology?
  - c. How much water was added to the tank before the system responded? (Sum of water added before the run to achieve 75% of vendor-specified height and the water added during the run.)
3. Did the technology register increases in water after initially responding?
  - a. What was the volume of water added between the initial response and the first registered increase? For later increases, what was the volume of water added between the most recent increase and the previous increase?
  - b. What was the height of the increase as measured by the technology? For later increases, what was the height measurement between the most recent and the previous increase?

#### **4. Fuel LD**

The fuel LD test is similar to the original protocol<sup>3</sup> in that it establishes run conditions from all combinations of variables. The variables include three levels of ethanol content, two levels of fuel height in the tank, and two leak rates including a zero leak rate. The two levels of fuel height in the tank differ from the water ingress tests. They were selected to encompass the portion of the tank that produces the smallest float displacement (i.e., 50% full) as well as a level below this height (25% full). The two levels of fuel leak rate were selected to establish the conditions that will produce data to meet the 95% probability of detecting a leak at 0.2 gal/hr with a 5% false alarm rate (0 gal/hr).. Should a vendor request that their technology be verified to the 0.1 gal/hr level, the leak rates will be adjusted to 0 and 0.1 gal/hr. Table 6 presents the run conditions to be established to be performed during the fuel leak test when a fuel leak is induced.

The number of runs necessary is based on the confidence bounds for the estimated proportion of leak detections and false alarms. If 59 runs when the fuel leak rate is equal to a set rate produce 59 leak detections then the estimated detection rate is 100% with a lower exact 95% confidence bound of 95.05%. Therefore, it is reasonable to conclude that the system detects fuel leaks of that tested rate with at least 95% probability. Conversely, if 59 runs when the fuel leak rate is 0 gal/hr yield zero leak detections, then the estimated false alarm rate is 0% and the exact

95% upper confidence bound for the rate is 4.95%, so it is reasonable to conclude that the false alarm rate is below 5%. Table 7 presents the run conditions to be established to be performed during the fuel leak test when a fuel leak is zero.

**Table 6. Summary for Fuel Leak Detection Runs with a Leak(a)**

Number of Runs	Fuel Type	Fuel Leak Rate (gal/hr)	Fuel Height (% of full height)
10	E0	0.2 <sup>b</sup>	50%
10	E0		25%
10	E15		50%
10	E15		25%
10	E85		50%
10	E85		25%

- a. Table order does not reflect the order in which runs are performed. Run order will be established during testing so as to minimize the amount of fuel handling and fuel waste produced during the test series.
- b. Alternate fuel leak rates may be 0 and 0.1 for technologies being verified for 0.1 gal/hr rate.

**Table 7. Summary for Fuel Leak Detection Runs Without a Leak(a)**

Number of Runs	Fuel Type	Fuel Leak Rate (gal/hr)	Fuel Height (% of full height)
10	E0	0	50%
10	E0		25%
10	E15		50%
10	E15		25%
10	E85		50%
10	E85		25%

- a. Table order does not reflect the order in which runs are performed. Run order will be established during testing so as to minimize the amount of fuel handling and fuel waste produced during the test series.

Data collected or calculated with each run will include, but are not limited to:

1. Did the ATG system detect a fuel leak?
  - a. If so, after what length of wait time did the ATG detect the leak?
  - b. What leak rate was reported by the ATG?

Table 8 presents a summary of the designs for the water ingress and fuel leak tests. The associated performance parameters for each test are also provided as well as the variables and number of runs.

**Table 8. Summary of Four Main Tests**

Test	Test Description	Performance Parameter	Independent Variables	# of Runs
1a: Continuous Water Ingress Test- Minimum detection height	Water ingress detection of continuous water ingress with a splash or without a splash to determine the minimum water level that the ATG can detect	<ul style="list-style-type: none"> <li>▪ Accuracy</li> <li>▪ Sensitivity</li> <li>▪ Precision</li> <li>▪ Operational factors</li> </ul>	<ul style="list-style-type: none"> <li>▪ Water ingress method/rate</li> <li>▪ Fuel height in tank</li> <li>▪ Fuel type</li> </ul>	12 Runs + 4 Duplicates
1b: Continuous Water Ingress Test- Smallest detection increment	Water ingress detection of continuous water ingress with a splash or without a splash to determine the smallest change in water level that the ATG can detect	<ul style="list-style-type: none"> <li>▪ Sensitivity</li> </ul>	<ul style="list-style-type: none"> <li>▪ Water ingress method/rate</li> <li>▪ Fuel height in tank</li> <li>▪ Fuel type</li> </ul>	Continuation of runs in Test 1a while collecting 10 incremented measurements
2: Quick Dump	Water ingress detection of a quick water dump, then a fuel dump to induce and observe phase separation	<ul style="list-style-type: none"> <li>▪ Phase separation</li> <li>▪ Operational factors</li> </ul>	<ul style="list-style-type: none"> <li>▪ Water dump</li> <li>▪ Fuel dump</li> <li>▪ Fuel type</li> </ul>	3 Runs
3: Water Ingress + LD Test	Water ingress and fuel LD during water ingress and fuel leak	<ul style="list-style-type: none"> <li>▪ Accuracy</li> <li>▪ Sensitivity</li> <li>▪ Precision</li> <li>▪ Operational factors</li> </ul>	<ul style="list-style-type: none"> <li>▪ Fuel leak rate</li> <li>▪ Water ingress method/rate</li> <li>▪ Fuel type</li> </ul>	18 Runs + 4 Duplicates
4: LD	Fuel LD	Binomial results <ul style="list-style-type: none"> <li>▪ Accuracy</li> <li>▪ False alarm rate</li> <li>▪ Operational factors</li> </ul>	<ul style="list-style-type: none"> <li>▪ Fuel leak rate</li> <li>▪ Fuel height in tank</li> <li>▪ Fuel type</li> </ul>	59 runs at the desired detectable leak rate and 59 runs at zero leak rate
		Measured rate comparisons <ul style="list-style-type: none"> <li>▪ Accuracy</li> <li>▪ Precision</li> <li>▪ Operational factors</li> </ul>		6 runs with 10 duplicates at all combinations of variables

**Other Monitoring Data**--Other variables may influence the operability of ATG systems being evaluated, and information on these other variables will be collected during the tests but not controlled. Table 9 presents a list of these other variables, their measurement methods, and monitoring frequency.

**Table 9. Other Independent Variables to be Monitored During Testing**

Variable	Measurement Method	Monitoring Frequency
Ambient temperature	Thermometer*	Continuously/record every ½ hour
Barometric pressure	Barometer*	Continuously/record every ½ hour
Fuel density/specific gravity/other properties	Density meter*	Semi-continuously
Tank vibration (for laboratory fuel leak tests only)	Vibration meter*	Continuously
Ground water level (for field tests only)	Monitoring well or Geoprobe	Once prior to each test
Tank size, geometry, and material of construction	Construction specifications	Once prior to tank use

\* Calibration will be performed initially and as stated in the instrument manual.

## **B1.1 Test Procedures**

### ***B1.1.1 Pre-test Checks and Dry Runs***

Table 10 presents a list of items to check prior to beginning the verification test. This checklist will be completed before proceeding and lists the tasks necessary to establish a safe laboratory for this verification test. In addition to the checklist, dry runs will be conducted to establish the laboratory procedures for testing, ensure the variables are achievable, and verify that these preselected variables will elicit a significant change in the condition in order to evaluate the performance of ATGs. These will include, for example, establishing a procedure for water introduction techniques and/or mixing methods, maintaining and adjusting temperature, establishing and verifying the fuel height, establishing a fuel blending and transferring procedure, and discerning the best vantage point to video record the tests. Other considerations of QAPP implementation that will be determined during the pre-test checks and dry runs are described in sections B1.1.2 to B1.1.4.

### ***B1.1.2 Order of Tests***

Run conditions will be established in such a manner so as to minimize the amount of fuel handling and fuel waste produced during the test series. The order will be established prior to each test type (water ingress or fuel leak); however, it may be varied during the test series if the situation indicates that more efficient fuel handling could be achieved or less fuel waste could be

produced. The actual order of runs and tests performed will be documented in the project records and presented in the verification report(s).

**Table 10. Pre-test Checklist**

Pre-test Item	Laboratory Tests	Field Tests
QAPP, including JHAs, reviewed by test staff and vendors		
Instrument and equipment calibrations current		
Fire extinguishers charged and nearby		
Waste containers are available		
Spill kits are available		
Technology installed per vendor instruction		
Technology preliminary checks performed		
Synchronize time of data recording devices (ATG consoles and DVR)		
Threshold level for water detection technology is known/proven		
Equipment is functional with adequate power		
Test vessel inspected for leaks		
Groundwater depth is determined		
Facility manager has been notified		
“Do Not Enter--Test in Progress” signs posted		
Adequate fuel supply on hand		
Adequate ethanol supply on hand		
Adequate water supply on hand		
Consoles and data recorders has adequate paper supply, if needed		
Adequate storage media for DVR		
Adequate power supply for DVR		
Test conditions identified and order established		

***B1.1.3 Sequence of Procedures***

The sequence will be important when conducting an efficient test series. The following discussion is suggested as a means to maximize the accuracy of test conditions.

Establishing variables--The first two variables for which conditions must be established are the fuel-related variables. For tests with E0 and E85, fuel will be added to the laboratory test vessel until the desired height is achieved within 1%. For E15, the test vessel may be filled to

desired calculated height with E0, an appropriate volume of ethanol or E85 will be added to the test vessel, and the vessel contents will be re-circulated until the ethanol concentration is consistent throughout and within 10% of the target ethanol content. The height of fuel will be measured after the liquid becomes quiescent, and the height will be verified or adjusted to be within 1% of the target for the run condition. Alternatively, larger batches of E15 may be mixed in a storage tank to within 10% of E15 and dispensed into the test vessel to the appropriate fill height for the target run conditions.

When water ingress tests are to be conducted, the next condition that will be established is the initial water height in the test vessel. An initial volume of water will be introduced to the bottom of the test vessel in such a manner so as to avoid agitation of the fuel present in the tank. The water will be introduced through one or multiple connector(s) at the bottom of the tank, thus minimizing, to the extent possible, mixing with the fuel. The volume of water to be added in this manner will be the volume needed to produce a water layer approximately 75% of the vendor-stated threshold height needed for the water detection technology to react. If multiple technologies are being evaluated at the same time, this volume will be such that none of the threshold heights are exceeded for any technology.

After all of the above variable conditions are achieved, the test will begin by using a peristaltic pump to add water to and/or extract fuel from the test vessel. Proper tubing will be selected to achieve the desired flow rates during testing.

Duration of tests--The E0 tests will be performed first to establish a minimum wait time for the ATGs to detect the water ingress and/or fuel leak. The wait times for the two other fuels will be estimated by adding the E0 baseline time and a calculated estimate based on the pre-test experiments.

#### ***BI.1.4 Special Considerations***

Any water present in the test vessel after a test will be drained from the bottom of the tank, characterized with respect to its Resource Conservation and Recovery Act (RCRA) properties, and disposed of properly. The remaining contaminated fuel will be removed from the test vessel and properly characterized for disposal. Due to the costs associated with the waste generated during this verification test, the dense phase may be dealt with separately or together with the wasted fuel.



## **B1.2 Statistical Analysis**

### Water Detection

- The minimum height of water that the technology reliably detects will be assessed using the methodology from the original protocol, with some updates to account for different variables and subsequently the different number of test runs. The bias and variance and standard deviation (the square root of the estimated variance) of the results will be reported along with a tolerance limit of water that is 95% likely to cause the technology to detect water.
- The minimum increase in water that can be detected will be assessed using the methodology from the original protocol where the minimum water level change (MLC) will be reported as with the increment of water that is 95% likely to cause the technology to report a water depth estimate.

### Leak Detection

- Data from the independent runs will be combined to estimate the proportion of runs during which leaks are detected. When the induced leak rate is 0 gal/hr, then the proportion of detections is the estimated false alarm rate. When the induced leak rate is positive (0.2 gal/hr) then the proportion of detections is the estimated probability of detection. Exact binomial 95% confidence bounds will be calculated in the manner described by Clopper and Pearson.<sup>7</sup> The bounds will be calculated in Microsoft Excel™ following widely accepted data processing within the program.
  - For runs where the true leak rate was non-zero (set to the rate at which the vendor desires to be certified), the analysis will report the number of runs, the number of runs with detections, the estimated proportion of runs with detections, and an exact 95% lower confidence bound on the probability of detection.
  - For those runs where the true leak rate was zero, the analysis will report the number of runs, the number of runs with detections (all of which are false alarms), the estimated proportion of runs with false alarms, and an exact 95% upper confidence bound on the probability of a false alarm.
- The run results may also be interpreted using the measured leak rate output from the ATG technology compared to the induced leak rates (measured rate comparison).

The bias and variance and standard deviation (the square root of the estimated variance) of the results will be reported.

Given these calculations, the following performance parameters will be evaluated.

- Accuracy (water ingress test), expressed in terms of whether the depth at which water is detected is less than or equal to the height stated by the vendor. (This analysis assumes that the depth stated by the vendor is claimed to be a height at which their technology would detect water at least 95% of the time.) Also whether the estimated minimum increase that can be detected is less than or equal to the detectable increase stated by the vendor or to the nearest 1/8<sup>th</sup> of an inch (whichever is smaller).
- Accuracy (fuel leak test), expressed in terms of whether there is evidence that the probability of detection is  $\geq 95\%$  and the probability of false alarm is  $\leq 5\%$ .
- Accuracy (fuel leak test), expressed in terms of the average difference between the measured and induced leak rates over the number of runs and the variation with respect to that average difference.
- Sensitivity (water ingress test), expressed as the minimum value for water height at which the probability is at least 95% that the water detection technology detects the presence of water in the bottom of the tank.
- Precision (water ingress and leak rate tests), calculated as the ratio of the mean technology-measured water height or leak rate at the specified end point of a test to the standard deviation of that same quantity.
- False Alarm Rate (fuel leak test), expressed as the proportion of test trials when the true leak rate was zero but the system detected a leak. Reported both as the proportion and as an upper 95% exact confidence bound.

### ***B1.2.1 Accuracy***

#### Water Detection

If the estimated minimum amount of additional water that is detected in an increase is less than or equal to the amount specified by the vendor, then the vendor-stated smallest change in the water level that the technology can detect will be reported. The bias will be calculated as below in Equation 1 as an estimate of accuracy.

$$\text{Bias} = \sum_{i=1}^{n_i} \frac{L_i - S_i}{n} \quad \text{Equation 1.}$$

Where  $n$  is the number of runs,  $L$  is the technology measured increase in water height, and  $S$  is the independently measured increase in water height.

### Leak Detection

For binomial analysis, if the lower confidence bound on the probability of leak detection is  $\geq 95\%$  then the ATG leak detection will be reported at the induced leak rate value. For the measured rate comparison, the bias will be reported and calculated as above in Equation 1. For the leak rate bias,  $L$  is the technology reported leak rate, and  $S$  is the induced leak rate.

### ***B1.2.2 Sensitivity***

Sensitivity is a measure of the extent to which the methods and instrumentation associated with a given technology are actually able to detect the event of interest when in fact the event has occurred. A technology is determined to have higher sensitivity as the event becomes more difficult to detect with a certain degree of confidence. As a result, the event that is most difficult to detect with specified confidence is determined. Sensitivity differs according to the nature of the test and type of event.

- For the water ingress test, sensitivity is quantified by the minimum value for water depth at which the probability is at least 0.95 (95%) that the water detection technology will detect the presence of water in the bottom of the tank given that the true water depth (tolerance limit). In addition sensitivity is quantified by the smallest detectable change in the water height once water is detected with at least a 95% probability of detecting the change (minimum water level change).
- For the fuel leak test, sensitivity is not estimated. Conceptually, it might correspond to the minimum leak rate that would detect a leak 95% or more of the time. Because this test protocol only uses one non-zero leak rate, it is impossible to estimate a meaningful sensitivity figure.

If the estimated minimum height that would be detected at least 95% of the time is less than or equal to the height specified by the vendor, then the vendor-stated height will be reported as the minimum height for the technology to detect water ingress. The tolerance limit (TL) will be used for this comparison. To calculate the TL follow the below calculations.

1. Calculate the mean ( $\bar{x}$ ) of the observations as in Equation 2.

$$\bar{x} = \sum_{i=1}^n \frac{x_i}{n} \quad \text{Equation 2.}$$

Where n is the number of observations and x is the recorded observation from the technology.

2. Calculate the standard deviation (SD) of the observations as in Equation 3.

$$SD = \left[ \frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1} \right]^{1/2} \quad \text{Equation 3.}$$

Where n is the number of observations, x is the recorded observation from the technology, and  $\mu$  is the mean of the observations.

3. Find k from a table of tolerance coefficient for one-sided normal tolerance interval with a 95% probability level and a 95% coverage for the number of observations.<sup>8</sup>
4. Calculate the TL as in Equation 4.

$$TL = \bar{x} + k SD \quad \text{Equation 4.}$$

Where  $\mu$  is the mean of the observations, k is the tolerance coefficient, and SD is the standard deviation of the observations.

The estimated minimum height that would be detected at least 95% of the minimum detectable change of the water height, the minimum water level change (MLC) will be calculated by following the below steps.

1. Calculate the difference (d) between the technology observation and the independently-measured water increment heights for all observations as in Equation 5, noting the group of observations from each run during Test 1.

$$d_{ir} = w_{tr} - w_{mr} \quad \text{Equation 5.}$$

Where  $w_{tr}$  is the technology measured increment of the  $r^{\text{th}}$  run and  $w_{mr}$  is the independently measured water increment of the  $r^{\text{th}}$  run.

2. Calculate the average of the differences (D) for each group of observations from the Test 1 runs as in Equation 6.

$$D_r = \sum_{i=1}^n \frac{D_{ir}}{n_r} \quad \text{Equation 6.}$$

3. Calculate the variance ( $Var_r$ ) of the differences separately for each group of observations from the Test 1 runs as in Equation 7.

$$Var_r = \sum_{i=1}^n \frac{(d_i - D_r)^2}{n_r - 1} \quad \text{Equation 7.}$$

4. Calculate the pooled variance ( $Var_p$ ) of the groups as in Equation 8.

$$Var_p = \frac{(n_{r1} - 1)Var_{r1} + \dots + (n_{r\#} - 1)Var_{r\#}}{\sum_{r=1}^{\#} (n_r - 1)}$$

5. Calculate the pooled standard deviation ( $SD_p$ ) as in Equation 8.

$$SD_p = \sqrt{Var_p} \quad \text{Equation 8.}$$

6. Find the tolerance coefficient (k), for two-sided tolerance intervals with 95% probability and 95% coverage from a tolerance factor table<sup>9</sup>.
7. Calculate the MLC that the technology can detect using Equation 9.

$$MLC = k SD_p \quad \text{Equation 9.}$$

### ***B1.2.3 Precision***

Precision is a measure of the extent to which the methods and instrumentation associated with a given technology yield results that are reproducible. For a given set of test conditions, precision is characterized by the ratio of the mean of a technology-measured value to its standard deviation. Precision is determined as follows:

- For the water ingress test, precision corresponds to the ratio of the  $\bar{x}$  associated with the technology-measured water height at the specified end point of a test to the SD of water heights measured at that same point in the test.
- For the fuel leak test, precision will be reported for the measured rate comparison similarly to the water ingress test comparison in terms of the  $\bar{x}$  over the SD as calculated in Equations 2 and 3, respectively using the measured leak rate by the technology and the induced leak rate.

### ***B1.2.4 False Alarm Rate***

The false alarm rate is relevant only to the fuel leak test with a zero-induced fuel leak rate. It is represented by the probability that the methods and instrumentation determine that a fuel leak is present when in fact there is no true fuel leak. The estimated false alarm rate will be the proportion of trials that detect a leak when none is present. That proportion will be reported for every set of test conditions along with its corresponding exact 95% upper confidence bound. A summary estimate that is aggregated across all 0 induced leak rate run conditions will also be reported.

#### ***B1.2.5 Phase Separation***

Phase separation during water ingress tests, specifically for Test 2, will be determined when a change in appearance of the tank contents occurs. This change is a differentiation of a separate liquid layer that forms below the fuel at the bottom of a storage tank due to water ingress. This situation, should it occur, will be observed visually and by using a digital video recorder (DVR) during testing. Test conditions leading to phase separation will be documented to define the testing environment in which phase separation occurred (i.e. the phase separation layer height, fuel temperature and density, etc.). The water will be dyed to aid in the visualization of the phase separation.

#### ***B1.2.6 Operational Factors***

Operational factors such as maintenance needs, calibration frequency, data output, consumables used, ease of use, and repair requirements will be evaluated based on technical staff and VTC observations for all tests performed.

### **B1.3 Reporting**

The data obtained in the verification test will be reported and the statistical analyses described above will be conducted separately for each technology being tested. Information on the performance parameters will be compiled, and separate verification test reports will be prepared for each vendor. Each report will show separate verification results from the specific technologies undergoing testing for each vendor. Each verification test report will present the test procedures and test data, as well as the results of the statistical evaluation of those data. If a test is inconclusive, the result will be reported; however, depending on the testing situation, the

VTC, or designee, with input from a Battelle statistician will determine if the run will be repeated and whether it will be included in the statistical analysis.

All actions taken on the technology (such as maintenance, cleaning, and calibration) will be documented at the time of the test and reported. In addition, descriptions of the data-recording procedures, use of vendor-supplied software, and fuel supplies or other consumables used will be presented in the report. The verification test 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 test report. The results of the verification test will then be stated quantitatively without comparison to any other technology tested or comment on the acceptability of the technology performance.

## **B2 SAMPLING METHODS REQUIREMENTS**

### **B2.1 Sample Collection, Storage and Shipment**

Fuel ethanol content determination will be performed before testing to verify that the ethanol concentration is within  $\pm 10\%$  of the target level. The E0 and E85 will be received, sampled, and verified for each batch of fuel delivered. Use of a Certificate of Analysis will suffice for this purpose if provided with the fuel delivery. For analyses conducted after fuel receipt, if the E85 is not within the  $\pm 10\%$  target range, ethanol or E0 will be mixed to blend the fuel to be within the target range. Since the E15 will be mixed on site, either in the test vessel or in a storage container, the fuel will be sampled by batch before testing with E15 begins. The sampling volume will be between 10 and 250 milliliters, which will be placed into a glass sampling jar with a Teflon-lined cap and sent to an analytical laboratory for analysis ethanol content. Per the analysis methods (ASTM D4815 and D5501), samples may be refrigerated prior to analysis and brought up to room temperature for analysis. Therefore, samples will be shipped in coolers with blue ice to keep the sample temperatures between 0 and 5°C (32 to 40°F)<sup>5,6</sup>.

For water ingress testing using ASTM E203 or 1064, the phase separated layer will be sampled when a phase change is observed and after each run is completed. At the beginning and end of the run, the water content of the fuel will be tested. These analyses of the dense phase and fuel are to characterize the water ingress testing condition. The 1.5 to 2 milliliter glass sampling vials and Teflon-lined caps for this analysis method will be solvent washed and dried overnight in a 100°C oven<sup>10</sup> and allowed to cool in a desiccator before filling and sealing. Sampling ports

will be used to draw out samples using syringes from various places in the test vessel. Ports will be located to cause the least amount of agitation to the liquid in the vessel while sampling. Samples collected will be stored in desiccators before analysis. The samples will be hand delivered to the analysis laboratory at the Battelle main campus located in Columbus.

The analysis methods for the fuel ethanol content and water content determinations are described in Section B4. Duplicate samples for both analytical determinations will be collected at a frequency of 10% of the samples from the same sampling port into a separate sampling jar for analysis. This will evaluate the reproducibility of the sampling method. Duplicate sample analysis from the same sampling jar at a frequency of 10% analyzed will evaluate the reproducibility of both ASTM D4815 and D5501<sup>5, 6</sup> or the equivalent method. Duplicate sample analysis of every sample is specified for water determination by the Karl-Fischer titration methods, and the sample results are acceptable when they are less than 10% different<sup>10, 11</sup>.

## **B2.2 Digital Video Recording**

The water ingress tests will be performed in a transparent vessel so that the physical impact of adding water to the vessel can be seen and video recorded. To facilitate visualization of the physical changes occurring within the test vessel, colored food dye will be mixed into all water introduced to the vessel in a sufficient amount so as to clearly show the water phase of the system. In addition to dye, visualization will be enhanced by placing a grid with incremental pattern spacing within the view area of the vessel to clearly display the height(s) and width(s) of various liquid phases in the tank. Digital video recordings will be collected for each water ingress test and possibly each fuel leak test.

## **B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS**

Each sample will be labeled with a unique sample identifier along with the date/time collected and the name of the technical staff. Sample custody will be documented throughout collection and analysis of the test samples following the Battelle Standard Operating Procedure (SOP) for Chain of Custody<sup>12</sup>. A chain-of-custody (COC) form will include details about the sample such as the time, date, location, and person collecting the sample. The COC form will track sample release from the sampling location to the analysis laboratory. Each COC form will be signed by the person relinquishing samples once that person has verified that the COC form is



accurate. Upon arrival at the analysis laboratory, COC forms will be signed by the person receiving the samples (if different from the sample collector) once that person has verified that all samples identified on the COC forms are present. Copies of all COC forms will be delivered to the VTC and maintained with the test records.

#### **B4 ANALYTICAL METHODS REQUIREMENTS**

At the beginning of the test, fuel samples will be collected from the ethanol-blended fuel to confirm ethanol and water content. In addition, samples will be taken from the phase separated layer on the bottom of the test vessel for water content determination and from the fuel after water ingress testing for ethanol content determination. As presented in Table 12, analytical technicians will conduct these analyses according to the QC requirements stated in the specific analytical methods.

Ethanol content will be determined by ASTM D4815<sup>5</sup> and D5501<sup>6</sup> using gas chromatography or an equivalent method(s). Water content will be determined using an automated Karl-Fischer titration water analysis instrument following either ASTM E203<sup>10</sup> for lower water content or ASTM E1064<sup>11</sup> for higher water content. The fuel water content determined at the beginning of the tests will use ASTM E203<sup>10</sup>; phase separated samples at the conclusion of the tests may have a high water content and will therefore be analyzed using ASTM E203<sup>10</sup>.

#### **B5 QUALITY CONTROL**

Steps will be taken to maintain the quality of data collected during this verification test. QC samples are incorporated into the sampling and analysis design to assess the quality of the method of assessment. QC procedures and acceptance criteria are presented in Table 11, and sample analysis quality control assessments are presented in Table 12. In addition, instruments and equipment used for this verification will operate at the expected ranges and calibration records will be verified and kept for all monitoring instrumentation (i.e., thermocouple, density measurement, etc.) and equipment used for establishing the variables. All data collected will be within the accepted QC criteria (or corrective action will be taken) and the true measured value will be reported.

**Table 11. Data Collection Quality Control Assessments for the ATG Verification Tests**

Measured Parameters	Method of Assessment	Frequency	Acceptance Criteria	Corrective Action
Tank tightness (for field tanks only)	Site specific tightness test procedure	Once before testing begins	An acceptable tank tightness test immediately prior to testing	Repeat test, repair tank, or select alternate test tank
Induced fuel leak rate (peristaltic pump)	Compare with metered rates	Twice before testing begins, intermittently during leak simulation testing	$\pm 10\%$ of target leak rate and $<10\%$ difference of duplicate measurements	Review data to troubleshoot results and adjust leak-inducing pump as necessary
Induced water ingress rate (peristaltic pump)	Compare with measured and calculated metered rates in triplicate	Twice before testing begins, intermittently during ingress simulation testing	$\pm 10\%$ of target ingress rate and $<10\%$ difference of duplicate measurements	Review data to troubleshoot results and adjust ingress-inducing pump as necessary
Ethanol content	ASTM D4815 or D5501 or equivalent method	Once for each batch delivered or prepared	$\pm 10\%$ of target ethanol content	Review data to troubleshoot results and adjust as necessary
Water content	ASTM E203 or E1064: Karl-Fischer Titration	Once before and after each water ingress test run	As determined by assessment method	Review data to troubleshoot results and adjust as necessary
Fuel height	Dip stick or independent ATG to at least the nearest $1/16^{\text{th}}$ of an inch	Once before each test run begins	$\pm 1\%$ of target height	Review data to troubleshoot results and adjust as necessary
Water height	Standard ruler to at least the nearest $1/32^{\text{nd}}$ of an inch	At the intervals specific to the test being performed	as measured and observed in video recording	Review data and adjust as necessary

**Table 12. Analytical Method Quality Control Assessments for the ATG Verification Tests**

Method	Type of Analysis	QC Check	Frequency	Acceptance Criteria	Corrective Action
ASTM D4815 and D5501 or equivalent method	Ethanol	Analytical Blank	Once per ten samples	Less than the method limit of quantification	Source of error should be investigated, corrected, and the sample(s) re-analyzed
		Sample Duplicate Analytical Duplicate	Once per ten samples	Within 10% difference	Source of error should be investigated, corrected, and the sample(s) re-analyzed
		Calibration Curve	Once per 30 samples	At least a five point calibration curve with $r^2 > 0.99$	Review data to troubleshoot results perform instrument maintenance as necessary, and re-analyzed sample(s)
		Continuing Calibration Verification (CCV)	Once per ten samples	Within 20% of the target concentration	Review data to troubleshoot results perform instrument maintenance as necessary, and re-analyzed sample(s)
ASTM E203 <sup>10</sup> and E1064 <sup>11</sup>	Water	Control Standard	Once with every sample	Within 5% of target concentration	Review data to troubleshoot results perform instrument maintenance as necessary, and re-analyzed sample(s)
		Sample Duplicate	Every sample	Within 10% difference	Review data to troubleshoot results perform instrument maintenance as necessary, and re-analyzed sample(s)

**B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE**

The instruments used during the verification test, including those defined in Tables 9 and 11, will be inspected and maintained according to the instrument manuals or the laboratory standard operating procedures of the analysis laboratory. Operation of the technologies during the verification test will be performed by Battelle technical staff as directed by the vendor. The test vessel will be visually inspected every testing day to ensure it does not leak.

## **B7 CALIBRATION/VERIFICATION OF TEST PROCEDURES**

The calibration and verification of the test procedures lie in the QC assessments and acceptance criteria presented in Tables 11 and 12. The instruments used to determine the test conditions during the tests will be calibrated per the instrument manual, the methods being used to make each measurement, or the standard operating procedures of the analysis laboratory. For each measurement type, the equipment calibration will be verified initially and thereafter as specified in Tables 9 or 11, or as determined in the pre-testing checks. Calibration procedures, checks, and results will be documented in the project files. Testing will not occur until instrument calibration results meet the acceptance criteria.

All calibrations performed will be documented by the verification staff in the project LRB or data collection forms. The ATG technology vendor will provide the Battelle verification staff with the necessary training/information to properly calibrate and maintain each ATG technology. Calibration of the ATGs will be done as often as suggested by the vendors. Vendors will be required to describe the necessary calibration procedures and devices specific to the ATG technologies being verified.

## **B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES**

All materials, supplies, and consumables used to establish the test conditions will be ordered by the VTC 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 OUST staff to guide selection of manufacturers and materials. E10 is currently the only ethanol-blended fuel with a standard reference material (SRM 2297). The performance of ASTM D4815 will be verified with this National Institute of Standards and Technology (NIST) provided SRM for E10 fuel, if available. The performance of ASTM D5501 will be verified with the NIST provided SRM for ethanol (SRM 2900). The ethanol content for each fuel will be verified before the beginning of testing with that fuel.

Fuel supplies will either be used as purchased from a fuel blending facility or blended on site using E0 or E10 blended with E85 or ethanol. All fuel and ethanol supplies, as well as generated liquid wastes, will be stored in tanks or containers approved for the material being stored. Fuel, ethanol, and liquid wastes storage areas will be on impermeable surfaces with

adequate secondary containment. Arrangements will be made with trained waste handling technicians for removal and disposal of wastes generated during testing.

Supplies must meet the following criteria:

- Solvent and reagent grades are based on the intended use. All materials must meet the purity requirements of the method.
- Equipment used to generate data must provide appropriate sensitivity.
- A certificate of analysis must be provided and retained for reagents and standards.
- The quality and purity of expendable materials must be documented and adequate to meet the data quality objectives of the client.

## **B9 NON-DIRECT MEASUREMENTS**

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

## **B10 DATA MANAGEMENT**

Various types of data will be acquired and recorded electronically or manually by verification staff during this verification test. All data and observations for the operation of the technologies will be documented by the vendors or verification staff on data sheets, in LRBs, or captured electronically. Table 3, presented previously, summarizes the types of records to be collected and maintained during the study. Results from the laboratory analytical instruments will be compiled by laboratory staff in electronic format and submitted to the VTC or other verification staff upon obtaining results before the beginning of each test run.

Records received by or generated by any of the verification staff during the verification test will be reviewed by the VTC or designee within 2 weeks of receipt or generation, respectively, before the records are used to calculate, evaluate, or report verification results. The review will be documented as the dated initials of the reviewer. Table 13 summarizes the checks to be performed. 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 that originally received or 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 verification 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 QAPP. The data obtained from this verification test will be compiled and reported for each technology. Results for technologies from different vendors will not be compared with each other.

All electronic testing records and documents will be stored on a test-specific networked ETV SharePoint site. Testing data will be uploaded to the SharePoint Site on a weekly basis. This site is within the protected Battelle network and is backed up regularly. The goal of this data delivery schedule is prompt identification and resolution of any data collection or recording issues.

In addition, once testing is complete, all testing records and documents are sent to Battelle’s RMO for archival within 2 months of project close-out.

**Table 13. Data Verification Checks**

<b>Data Verification Activity</b>
QC samples and calibration standards will be analyzed according to this document, and the acceptance criteria will be met. Corrective action for exceedances will be taken.
100% hand-entered and/or manually calculated data will be checked for accuracy.
Calculations performed by software will be verified at a frequency sufficient to ensure that the formulas are correct, appropriate, and consistent.
For each cut and paste function, the first and last data values will be verified against the original source data.
Data will be reported in the units specified in the QAPP.
Results of QC will be reported.

## **SECTION C**

### **ASSESSMENT AND OVERSIGHT**

#### **C1 ASSESSMENTS AND RESPONSE ACTIONS**

One of the major objectives of the QAPP is to establish mechanisms necessary to anticipate and resolve potential problems before the quality of performance is compromised. Internal QC measures described in this QAPP, which is peer reviewed by a panel of outside experts, implemented by the technical staff, and monitored by the VTC, will yield day-to-day information on data quality. The responsibility for interpreting the results of these checks and resolving any potential problems resides with the VTC. 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 VTC, who will work with the Battelle QM to resolve any issues. Action will be taken to identify and appropriately address the issue and minimize losses and correct data, where possible. VTC will also relay testing progress and data to the EPA PO on a weekly basis to ensure that EPA has real-time access to the data as generated and testing continues to fulfill the DQOs. Battelle will be responsible for ensuring that the audits described in the following subsections are conducted as part of this verification test. See Table 2 for the proposed verification test schedule of audits.

Any changes to the approved QAPP must be reported within 24 hours and documented in a formal deviation submitted to the Battelle AMS Center Manager, EPA PO and EPA QM. If approval by EPA or his designee is not received within 24 hours of notification testing will be halted until a suitable resolution has been achieved.

##### **C1.1 Performance Evaluation Audits**

A PEA will be conducted to assess the quality of the variable measurements made in this verification test. The PEA will verify that the measured parameters for leak rate and water ingress rate in Table 11 are achievable within the stated acceptance criteria.

In addition, the ethanol and water content determination methods will be initially tested against certified standards. The accuracy of the analytical methods will be evaluated in the PEA by analyzing a NIST traceable certified standard, when available. For the low level ethanol

content determination method D4815, SRM 2297- Reformulated Gasoline (10% Ethanol) will be used. For the high level ethanol content determination method D5501, SRM 2900-Ethanol-Water Solution, (nominal 95.6%) will be used. The results of this E10 standard are acceptable when within 10% of the target ethanol content. The water standard concentration and source will be determined during the pre-checks and dry runs and will also be NIST traceable if available. The results of the water standard are acceptable when within 5% of the target control standard concentration. If the results do not meet the requirements in Table 11, they will be repeated. If the outlying results persist, the VTC, or designee, and the laboratory representative will discuss corrective actions, and the PEA will be repeated. The results from the PEA will be sent to the EPA PO and EPA QM within 10 days of receipt of the results. The PEA report will include the raw data, certificate, calculations of the comparison to the expected concentration, and a discussion of corrective action, if applicable.

## **C1.2 Technical Systems Audits**

Battelle QM will perform a TSA at least once during this verification test. The purpose of this audit is to ensure that the tests are being performed in accordance with the AMS Center QMP<sup>1</sup> and this QAPP, including a review of the fuel leak rates and water ingress rates. During this audit, the Battelle QM, or designee, will compare actual test procedures to those specified or referenced in this plan and review data acquisition and handling procedures. The auditor will include a review of the testing facility, equipment (calibration, maintenance, and operation) and observation of testing and records (including custody forms). He/she also will check data acquisition procedures and may confer with the vendor and technical staff. The TSA will be guided by a project-specific checklist based on this QAPP.

A TSA report will be prepared as a memo to the VTC within 10 business days after completion of the audit; the completed checklist will be attached. The Battelle AMS Center Manager, EPA PO and EPA QM will be copied on the memo. The VTC will respond to the audit within 10 business days. The Battelle Quality Manager or designate will verify that all audit Findings and Observations have been addressed and that corrective actions are appropriately implemented. A copy of the complete TSA report with corrective actions will be provided to the EPA PO and EPA QM within 10 business days after receipt of the audit response. 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 QM, or designee, will audit at least 25% of the sample results acquired in the verification test and 100% of the calibration and QC data per the QAPP requirements. A checklist based on the QAPP will guide the audit. An initial ADQ will be conducted on the first batch of test data within 10 business days of when data were posted on the project SharePoint site to identify errors early in the data reduction process. The first batch is defined as the testing and variable data generated over the first 2 weeks of testing by the VTC. The remaining data will be audited after all data for a technology or method has been posted on the project SharePoint site and once all statistical analyses are complete. The primary focus of this second audit will be the variable data. Finally, a third ADQ, performed by the Battelle QM or designee, will trace the data from initial acquisition, through reduction and statistical comparisons, to final presentation in the reports and verification statements. It will also confirm reconciliation of the two ADQs.

All formulae applied to the data will be verified, and 25% of the calculations will be checked. Data for the technologies will be reviewed for calculation and transcription errors and data traceability. An audit report will be prepared as a memo to the VTC within 10 business days after completion of each data audit; the completed checklist will be attached. The Battelle AMS Center Manager, EPA PO and EPA QM will be copied on the memo. The VTC will respond to the audit within 10 business days. The Battelle QM or designate will verify that all audit Findings and Observations have been addressed and that corrective actions are appropriately implemented. A copy of the complete ADQ report with corrective actions will be provided to the EPA PO and EPA QM within 10 business days after receipt of the audit response. EPA QA staff will also conduct an independent ADQ.

### **C1.4 QA/QC Reporting**

Each assessment and audit will be documented in accordance with Section 3.3.4 of the AMS Center QMP<sup>1</sup>. The results of the PEA, including raw data and calculations, will be

reported as stated in Section C1.1. The results of the TSA and ADQ will be submitted to EPA.

Assessment reports will include the following:

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

## **C2 REPORTS TO MANAGEMENT**

The Battelle QM, 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 QM is authorized to notify the Battelle AMS Center Manager who will issue an order to stop work. Once the TSA or ADQ report has been prepared, the VTC will respond to each Finding or Observation following the timeline defined in section C1 and will implement any necessary corrective action. The Battelle QM will verify that corrective action has been implemented effectively.

In addition to this QAPP, a final report for each participating vendor and a verification statement for each technology verified will be prepared and reviewed. The final report is a comprehensive document describing the verification test and will include all technologies from a particular vendor. The verification statement is a three-to-five page summary of each technology, the test procedures, and the test results. Each draft report and verification statement will be submitted to the respective vendor for review as well as the expert peer reviewers. They are then reviewed by EPA PO and EPA QM. Upon approval by EPA, each verification statement will be signed by a senior manager of Battelle and by an EPA laboratory director. Original signed verification statements will also be provided to the respective vendors. Upon final review and approval, the documents will then be posted on the ETV website ([www.epa.gov/etv](http://www.epa.gov/etv)). A summary of the required assessments and audits, including a listing of responsibilities and reporting timeframes, is included in Table 14.

**Table 14. Summary of Assessment Reports<sup>a</sup>**

<b>Assessment</b>	<b>Prepared By</b>	<b>Report Submission Timeframe</b>	<b>Submitted To</b>
TSA	Battelle	TSA response is due to QM within 10 business days  TSA responses will be verified by the QM and provided to EPA within 20 business days	EPA ETV AMS Center
ADQ 1 (first batch)	Battelle	ADQ will be completed within 10 business days after receipt of first data set	EPA ETV AMS Center
ADQ 2 (raw data)	Battelle	ADQ will be completed once all data are received and analyzed	EPA ETV AMS Center
ADQ 3 (synthesized data and verification report)	Battelle	ADQ will be completed within 10 business days after completion of the verification report review	EPA ETV AMS Center

a. Any QA checklists prepared to guide audits will be provided with the audit report.

## **SECTION D**

### **DATA VALIDATION AND USABILITY**

#### **D1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS**

The key data review requirements for the verification test are the collection of QC samples as outlined in the QAPP, a comparison of raw data sheets and comments against final data to flag any suspect data, and a review of final data to resolve any questions about apparent outliers. The QA audits, as described within this document are designed to assure the quality of these data. The key data verification requirements for this test are stated in Section B10 of this QAPP. The data generated during this test will be reviewed by a Battelle technical staff member within 2 weeks of receipt or generation of the data. The reviewer will be familiar with the technical aspects of the verification test but will not be the person who generated the data. This process will serve both as the data review and the data verification and will ensure that the data have been recorded, transmitted, and processed properly. Furthermore, this process will ensure that the monitoring systems data were collected under appropriate testing conditions.

#### **D2 VALIDATION AND VERIFICATION METHODS**

Data verification is conducted as part of the data review as described in Section B10 of this QAPP. A visual inspection of handwritten data will be conducted to ensure that all entries were properly recorded or transcribed and any erroneous entries were properly noted. Electronic data from the ATG technologies will be inspected to ensure proper transfer from the data logging system. All calculations used to transform the data will be reviewed to ensure the accuracy and the appropriateness of the calculations. Calculations performed manually will be reviewed and repeated using a handheld calculator or commercial software (e.g., Excel). Calculations performed using standard commercial office software (e.g., Excel) will be reviewed by inspection of the equations used for the calculations and verification of selected calculations by handheld calculator. Calculations performed using specialized commercial software (i.e., for analytical instrumentation) will be reviewed by inspection and, when feasible, verified by handheld calculator, or standard commercial office software.

To ensure that the data generated from this test meet the goals of the test, a number of

data validation procedures will be performed. Sections B and C of this QAPP provide a description of the validation safeguards employed for this verification test. Data validation efforts include the completion of QC activities and the performance of a TSA audit as described in Section C. The data from this test will be evaluated relative to the measurements to ensure that the DQOs are met. Data failing to meet these criteria will be flagged in the data set and not used for evaluation of the technologies, unless these deviations are accompanied by descriptions of their potential impacts on the data quality.

An ADQ will be conducted by the Battelle QM 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 QAPP and the resulting ETV verification report(s) will be subjected to review by the ATG vendors, EPA, and expert peer reviewers. These reviews will assure that this QAPP and the resulting verification report(s) meet the needs of potential users of the ATG systems. Performance data for the ATG technologies, collected under conditions where the QC requirements for the duplicate and PEA samples were met, will be presented in the final verification report without any further comment. Performance data and variable 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 (DOC) and Adobe portable document format (PDF) and subsequently posted on the ETV website (

## SECTION E

### REFERENCES

#### E1 REFERENCES

1. *Quality Management Plan for the ETV Advanced Monitoring Systems Center, Version 8.* U.S. Environmental Technology Verification Program, Battelle, April 2011.
2. *Environmental Technology Verification Program Quality Management Plan.* January, 2008 (EPA/600/R-08/009).
3. Ken Wilcox Associates, I., *Standard Test Procedures for Evaluating Leak Detection Methods: Automatic Tank Gauging Systems (ATGS).* 1990, USEPA Solid Waste and Emergency Response/Research and Development.
4. RFA, *Building Bridges to a More Sustainable Future: 2011 Ethanol Industry Outlook.* 2011.
5. ASTM, *D4815-09: Standard Test Method for Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C<sub>1</sub> to C<sub>4</sub> Alcohols in Gasoline by Gas Chromatography.* November 2009.
6. ASTM, *D5501-09: Standard Test Method for Determination of Ethanol Content of Denatured Fule Ethanol by Gas Chromatography.* May 2009.
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8. Lieberman, G., *Tables for One-Sided Statistical Tolerance Limits*, ed. I.Q. Control. Vol. Vol. XIV, No 10. 1958.
9. *CRC Handbook of Tables and Probability and Statistics*, ed. W.H.B. (ed.). 1966: The Chemical Rubber Company.
10. ASTM, *E 203-08: Standard Test Method for Water Using Volumetric Karl Fischer Titration.* November 2008.
11. ASTM, *E 1064-08: Standard Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration.* May 2008.
12. *SOP ENV-ADM-009, Standard Operating Procedure for Sample Chain-of-Custody* Battelle, September 2007.

**Appendix A**  
**Automatic Tank Gauging (ATG) Systems**  
**Environmental Technology Evaluation (ETV)**  
**Verification Test Stakeholder Panel Lists**  
**ATG ETV Technical Panel**

<b>Name</b>	<b>Affiliation</b>
Andrea Barbery	U.S. EPA Office of Underground Storage Tanks (OUST)
Greg Baretta	Engineering Consultant Bureau of Storage Tank Regulation (Wisconsin)
Jim Barnette	SIGMA
Mark Barolo	U.S. EPA OUST
Michael Doucette	Northeast Tank Services
Mike Eck	Army Environmental Command
Laura Fisher	UST Leak Prevention Unit (California)
Jerry Flora	JDF Consulting
Sam Gordji	SSG Associates, University of Mississippi
Jeffrey Guthrie	Environment Canada
Kevin Henderson	Mississippi Department of Environmental Quality
Brad Hoffman	Tanknology
Steve Howell	National Biodiesel Board (NBB), MARC-IV Consulting Inc.
Curt Johnson	Alabama Department of Environmental Management
Mike Juranty	New Hampshire Department of Environmental Services Waste Management Division
Kevin Keegan	Tanknology
Brian Knapp	American Petroleum Institute (API)
Fran Kremer	U.S. EPA Office of Research and Development (ORD)
Ed Kubinsky	Crompco, LLC
Bill Moore	Utah Department of Environmental Quality, Division of Environmental Response and Remediation
Kristy Moore	Renewable Fuels Association (RFA)
Mark Morgan	Petroleum Marketers Association of America (PMAA)
Marcel Moureau	Marcel Moreau Associates
Mohamed Mughal	Army Environmental Command (AEC)
Shaheer Muhanna	Georgia Department of Natural Resources
John Neate	Strategies for Change
Marcia Poxson	Michigan Dept of Environmental Quality
Stephen Purpora	Protanic
Bob Renkes	Petroleum Equipment Institute (PEI)
Peter Rollo	Delaware Natural Resources and Environmental Conservation
Erik Sirs	U.S. EPA Region 10
Tim Smith	U.S. EPA OUST

<b>Name</b>	<b>Affiliation</b>
Willo Smith	7-11
Jim Weaver	U.S. EPA National Exposure Research Laboratory/ORD
Ken Wilcox	Ken Wilcox Associates, Inc. (KWA Associates)
John Wilson	U.S. EPA Robert S. Kerr Environmental Research Center
Andrea Zajac	Michigan Department of Environmental Quality

### ATG ETV Vendor Panel

<b>Name</b>	<b>Affiliation</b>
Randy Barnes	Alert Technologies
Randy Boucher	Franklin Fueling Systems
Ken Cornett	Veeder-Root
Tom D'Alessandro	OMNTEC Mfg., Inc.
Howard Dockery	Simmons
John Levy	Pneumercator Company, Inc.
Douglas Mann	VISTA Leak Detection, Inc.
Dan Marston	Franklin Fueling Systems
Bob Moss	Veeder-Root
Bill Nelson	Franklin Fueling Systems
Kent Ried	Veeder-Root
Lorraine Sabo	Franklin Fueling Systems
George Thuemling	Varec, Inc.
Larry Tripp	AMETEK APT
Jim Walton	OPW Fuel Management Systems
Greg Young	Vaporless Mfg., Inc.





