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Environmental Technology Verification Program

Advanced Monitoring Systems Center

Test/QA Plan for Verification of Ambient Ammonia Monitors at Animal Feeding Operations

TEST/QA PLAN

FOR

VERIFICATION OF AMBIENT AMMONIA MONITORS AT ANIMAL FEEDING OPERATIONS

September 2, 2003

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ETV Advanced Monitoring Systems Center

Test/QA Plan for Verification of Ambient Ammonia Monitors at Animal Feeding Operations

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1.0 INTRODUCTION

1.1 Test Description

This test/quality assurance (QA) plan provides procedures for a verification test of monitors used to measure gaseous ammonia at animal feeding operations (AFO). The verification test will be conducted under the auspices of the U.S. Environmental Protection Agency's (EPA) Environmental Technology Verification (ETV) program. 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 purchase and application decisions.

The verification test will be performed by Battelle, of Columbus, OH, which is EPA's partner for the ETV Advanced Monitoring Systems (AMS) Center. The scope of the AMS Center covers verification of monitoring methods for contaminants and natural species in air, water, and soil. In performing the verification test, Battelle will follow procedures specified in this test/QA plan, and will comply with quality requirements in the "Quality Management Plan for the ETV Advanced Monitoring Systems Center" (QMP).[1]

1.2 Test Objective

Emissions of atmospheric ammonia have generated considerable interest over the past many years because of the role ammonia plays in nitrogen deposition, atmospheric acid-base chemistry, and aerosol formation. In particular, livestock agriculture is identified as the largest source of atmospheric ammonia in the U.S., and accounts for approximately 70% of emissions in the U.S.[2] As such, there is a need to accurately quantify these emissions. The objective of this verification test is to verify the performance of commercial ammonia monitors under normal operating conditions at two different AFO facilities. The test will also assess various

performance parameters by supplying the monitors being tested with compressed ammonia gas standards.

1.3 Technology Description

The monitors to be tested in this verification test include both open-path systems as well as point source monitors. Open-path systems typically have a light source and sensor, or a retro reflector, that are installed at a variable distance from one another (typically 100-200 meters). The light (infrared or ultraviolet) is emitted from the source and passes through the ambient air to the sensor where it is detected (or to the retro reflector where, it is reflected back to a detector positioned with the source). Ammonia that is in the optical path will absorb the light resulting in a decrease in the light intensity that reaches the detector.

Several technologies employ techniques that are classified as point source monitors. These technologies include photoacoustic monitors, ion selective electrodes, tunable diode lasers, chemiluminescent monitors, and others.

In general, each of the monitors to be tested in this verification test are continuous monitors and provide real-time or near real-time measurements of the ammonia concentration.

1.4 Organization and Responsibilities

The verification test will be performed by Battelle in cooperation with EPA, the vendors who will be having their monitors verified, and the U.S. Department of Agriculture (USDA). The organization chart in Figure 1-1 shows the individuals from Battelle, the vendor companies, EPA, and USDA who will have responsibilities in the verification test. The specific responsibilities of these individuals and organizations are detailed in the following paragraphs.

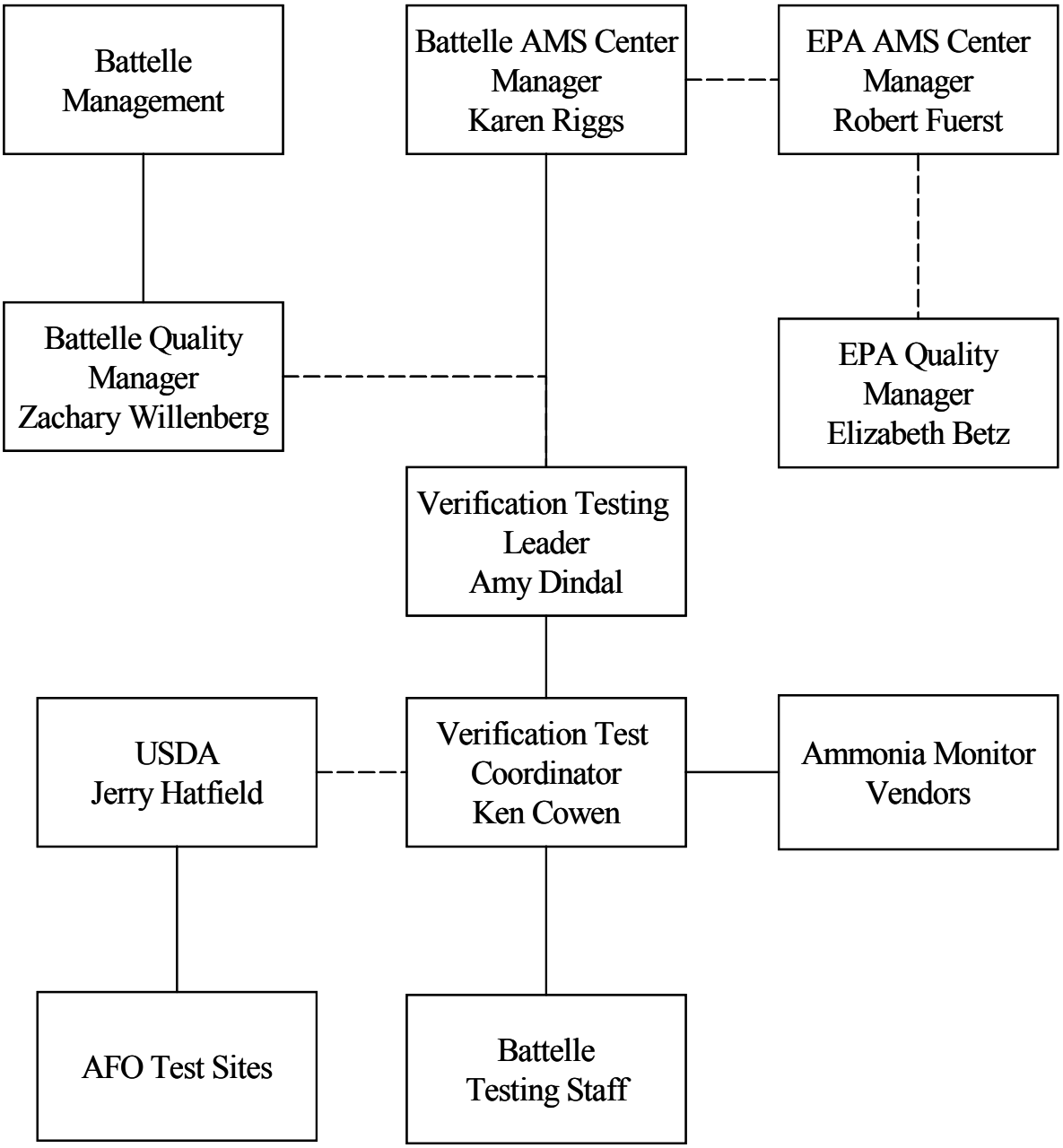

Figure 1-1. Organizational Chart for the Verification Test of Ambient Ammonia Monitors.

1.4.1 Battelle

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

- Coordinate Battelle, test site, USDA, and vendor staff to conduct the verification test
- Guide the Battelle/USDA/vendor team in performing the verification test in accordance with this test/QA plan
- Have overall responsibility for ensuring that this test/QA plan is followed
- Prepare the draft test/QA plan, verification reports, and verification statements
- Revise the draft test/QA plan, verification reports, and verification statements in response to reviewers' comments
- Respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary
- Serve as the primary point of contact for vendor representatives
- Coordinate distribution of final test/QA plan, verification reports, and statements
- Establish a budget for the verification test and monitor the effort to ensure that budget is not exceeded
- Ensure that confidentiality of vendor information is maintained.

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

- Support Dr. Cowen in preparing the test/QA plan and organizing the testing
- Review the draft test/QA plan

- Review the draft verification reports and statements.

Ms. Karen Riggs is Battelle's AMS Center manager. As such, Ms. Riggs will:

- Review the draft test/QA plan
- Review the draft verification reports and statements
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the verification test
- Ensure that vendor confidentiality is maintained
- Support Dr. Cowen in responding to any issues raised in assessment reports and audits
- Maintain communication with EPA's technical and quality managers
- Facilitate a stop work order if Battelle or EPA QA staff discovers adverse findings.

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

- Review the draft test/QA plan
- Conduct a technical systems audit once during the verification test
- Review results of performance evaluation audit(s) specified in this test/QA plan
- Audit at least 10% of the verification data
- Prepare and distribute an assessment report for each audit
- Verify implementation of any necessary corrective action
- Issue a stop work order if internal audits indicate that data quality is being compromised; notify Battelle's AMS Center Manager if such an order is issued
- Provide a summary of the QA/QC activities and results for the verification reports
- Review the draft verification reports and statements

- Ensure that all quality procedures specified in this test/QA plan and in the QMP[1] are followed.

Battelle testing staff will support Dr. Cowen in planning and conducting the verification test. These staff will:

- Assist in planning for the test, and making arrangements for the installation of the monitors
- Assist vendors and test site staff as needed during the monitor installation and verification testing
- Assure that test procedures and data acquisition are conducted according to this test/QA plan
- Contribute to the planning of statistical treatment of the ammonia monitor data as needed
- Perform statistical calculations specified in this test/QA plan on the ammonia monitor data as needed
- Provide results of statistical calculations and associated discussion for the verification reports as needed
- Support Dr. Cowen in responding to any issues raised in assessment reports and audits related to statistics and data reduction as needed.

1.4.2 Vendors

Vendor representatives will:

- Review the draft test/QA plan
- Approve the final test/QA plan

- Provide one ammonia monitor and the associated supplies for the duration of the verification test
- Commit or train a technical person to operate, maintain, and repair the ammonia monitor throughout the verification test
- Provide to Battelle staff the data from their monitor as requested by Battelle
- Review their respective draft verification report and verification statement.

1.4.3 EPA

EPA's responsibilities in the AMS Center are based on the requirements stated in the "Environmental Technology Verification Program Quality Management Plan" (ETV QMP).[3] The roles of specific EPA staff under the ETV QMP are as follows:

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

- Review the draft test/QA plan
- Perform, at her option, one external technical systems audit during the verification test
- Notify the EPA AMS Center Manager to facilitate a stop work order if an external audit indicates that data quality is being compromised
- Prepare and distribute an assessment report summarizing the results of the external audit, if one is performed
- Review the draft verification reports and statements.

Mr. Robert Fuerst is EPA's AMS Center Manager. As such, Mr. Fuerst will:

- Review the draft test/QA plan

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

1.4.4 USDA

This verification test will be conducted in collaboration with the USDA, who will provide co-funding for this test as well as in-kind support. The responsibilities of USDA are:

- Assist Battelle with the coordination of the AFO test sites for the purposes of ETV testing
- Assist Battelle with the coordination of the installation of vendors' equipment at the AFO test sites
- Contribute to the development of the draft test/QA plan
- Review the draft test/QA plan
- Provide on-site and laboratory staff to assist during testing
- Provide laboratory facilities for preparation of sampling media, and analysis of collected samples
- Conduct analysis of reference samples
- Provide all raw and final field and laboratory data to Battelle
- Assist in the operation of the vendor's monitors as needed
- Review draft verification reports and statements.

1.4.5 AFO Test Sites

This test will be conducted at two different AFO test sites. The responsibilities of these sites are:

- Provide access to the sites during testing for Battelle, USDA, EPA, and vendor representatives
- Provide adequate space for installation and testing activities.

2.0 VERIFICATION APPROACH

The overall objective of the verification test described in this plan is to provide quantitative verification of the performance of ambient ammonia monitors in realistic test conditions under continuous operation at two animal feeding operation facilities. The performance parameters that are addressed by this test/QA plan include:

- Accuracy
- Linearity
- Precision
- Comparability
- Interference effects
- Calibration drift
- Zero drift
- Response time
- Ease of use
- Data completeness.

Accuracy and linearity will be assessed for the monitors being verified by determining the degree of agreement with compressed gas standards. Precision will be assessed in terms of the repeatability of the ammonia measurements when supplied with ammonia gas standards. Comparability will be assessed by comparisons with a ammonia measurements using a reference method. Interference effects will be assessed by challenging the monitors being tested with compressed gas standards of various chemical species commonly emitted at AFOs. Calibration drift, and zero drift will be assessed by supplying the monitors being tested with commercial compressed gas standards of ammonia and zero gas, respectively. Response time will be

assessed by monitoring the time required to reach a stable reading when the monitors are supplied with compressed gas standards.

This verification test will be conducted concurrently for all of the monitors being verified, and will occur in two phases. The two phases will be conducted at separate animal feeding operations and will last approximately four weeks each. It is expected that the ammonia concentration will show considerable temporal variations during each phase of testing and that the average concentrations measured during the two phases will be substantially different. The performance of the monitors over those two periods will be assessed and reported. The amount of time each monitor is operational and any maintenance activities performed over the verification test periods will be recorded and reported, to help assess data completeness. Observations about the operation of the monitors will be made by the USDA and Battelle staff conducting the test, and will be used to assess the ease of use of each monitor being tested. Additionally, siting and installation requirements will be reported for each of the monitors.

3.0 TEST DESIGN

3.1 Site Description

[to be provided by USDA after clearance from AFOs]

3.2 Schedule

The verification test will be conducted in two phases. The first phase of testing will be conducted at a hog farm in Ames, Iowa, from approximately September 8 to October 3, 2003. The second phase of testing will be at a cattle farm in Bushland, Texas, from approximately October 13 to November 7, 2003. Prior to the beginning of each phase, there will be a shakedown period during which the vendors will install their monitors, ensure proper operation, and train Battelle/USDA staff on the routine operation of their monitors.

During each phase, two periods of intensive sampling will be conducted to collect reference samples using annular denuder technology as described in Section 3.4. The two intensive periods will be during the first and last weeks of each phase. The reference sampling will be conducted on a five sample per day schedule on each week day (i.e., Monday - Friday) during those weeks. Also during the first week of each phase, the monitors will be supplied with compressed gases to assess accuracy and linearity. The compressed gases will used to assess calibration drift and zero drift of the monitors. The calibration checks will be done on Monday, Wednesday, and Friday of the first and last weeks during the two test phases. During week two of the second phase of testing, the monitors will be supplied with compressed gas standards containing different interferent species. Figure 3-1 shows the proposed schedule for the testing activities for this verification test.

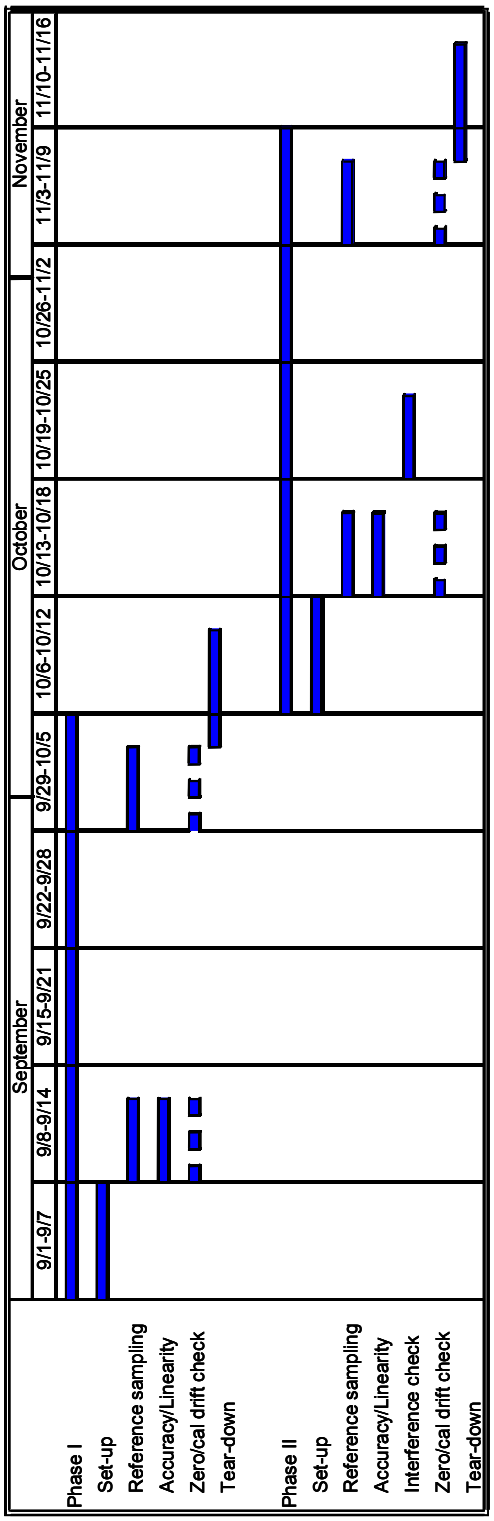

Figure 3-1. Proposed Schedule for Verification Test.

3.3 Installation

Each of the monitors being tested should be installed by the vendor with some means of supplying or sampling compressed gas standards (i.e., ammonia, zero gas) for the accuracy, linearity, and interference tests as well as the calibration and zero drift checks. During each phase the monitors being verified will be located within the fenceline of the AFO, and in the predominantly downwind direction from the primary ammonia source. Monitors employing open path techniques will be situated such that the optical path is perpendicular to the prevailing wind direction. These monitors will be installed such that all the optical paths are parallel to one another, and to the extent possible, with the same pathlength (or multiple thereof). Monitors employing point source measurements will be collocated with one another to the extent possible. These point source monitors will also be positioned such that they are along the pathlength of any open-path monitors being verified but offset from the optical path such that they do not interfere with the transmittance of reflectance of the light from the open-path monitors. Figure 3-2 illustrates an example of the potential arrangement of the monitors being tested and the reference method samplers.

In this figure, the x's represent potential locations where ammonia samples will be collected according to a reference method. As described below, simultaneous collection of several reference samples will be made at several points along the pathlength of the open-path technologies, with at least one sample collected at the location of the point source measurements. These reference samples will be used for establishing the accuracy of the ammonia monitors.

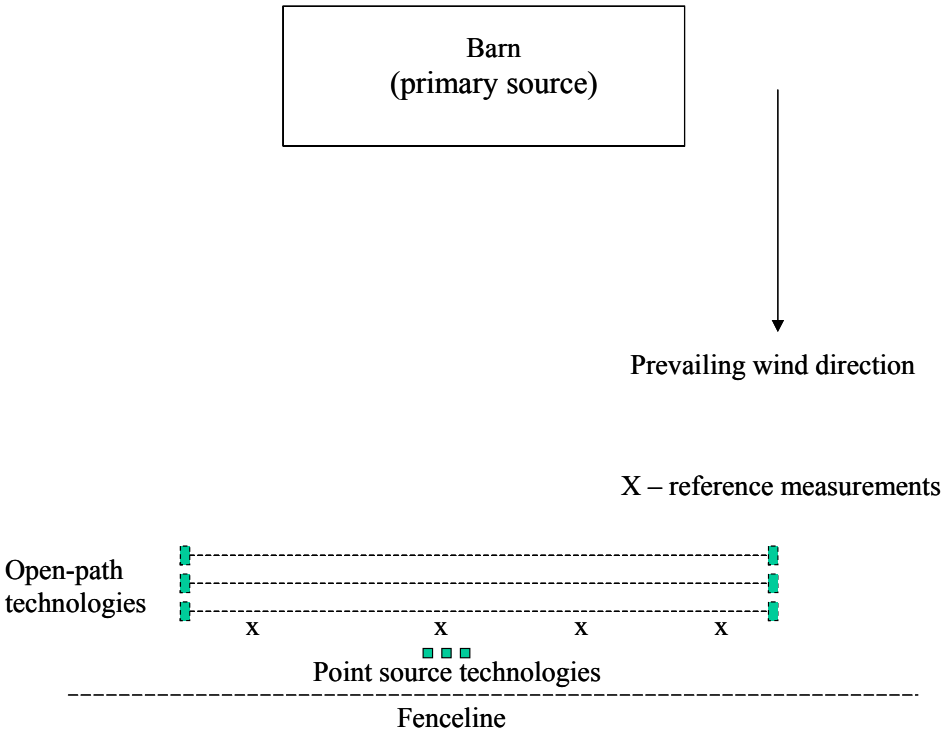

Figure 3-2. Illustration of potential installation during testing.

3.4 Reference Method

Reference samples will be collected using citric acid coated denuder sampling trains (see Figure 3-3). The method that will be used is based on the EPA Compendium Method IO-4.2 Determination of Reactive Acidic and Basic Gases and Acidity of Fine Particles (< 2.5 μm)[4], with some modifications to the method as described below. For this test, ambient air will first be drawn through an impactor at a nominal rate of 10 liters per minute (lpm) to remove particulate matter with aerodynamic diameters greater than 2.5 μm. The air will then pass through the citric acid coated denuder to remove gaseous ammonia. A single Teflon filter will be used to collect the particulate matter that passes through the denuder.

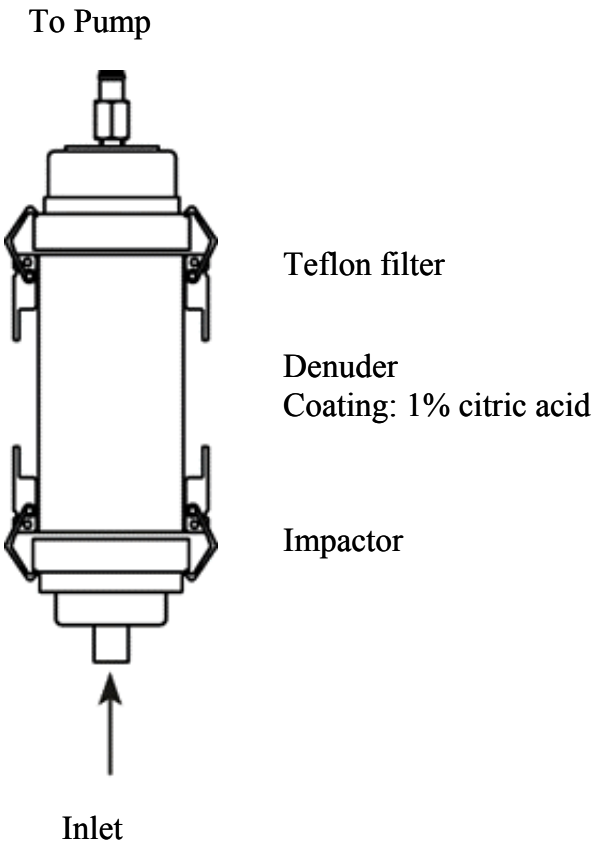

Figure 3-3. Illustration of Reference Method Sampling cartridge.

For this verification test, reference samples will be collected during the first and last week of testing during each phase. In order to capture diurnal variations in ammonia concentrations sampling is expected to be conducted on approximately the following schedule: 8:00 a.m. to 12:00 p.m., 12:00 p.m. to 2:00 p.m., 2:00 p.m. to 4:00 p.m., 4:00 p.m. to 8:00 p.m., and 8:00 p.m. to 8:00 a.m., such that 5 sets of samples are collected in each 24-hour period. The short term (2-hour and 4-hour) sampling is expected to capture the peaks in ammonia concentrations, whereas the 12-hour sampling will capture overnight concentrations. After each sampling period, the sampling media will be retrieved, sealed, and stored until being transported to the laboratory for analysis, and new sampling media will be installed.

3.5 Accuracy and Linearity Tests

On one day during the first week of testing during each phase, the monitors being tested will be supplied with a series of ammonia gas standards to assess accuracy and linearity. Prior to being supplied to the monitors, these standards will be diluted with zero air to achieve concenctrations between approximately 200 - 2000 ppb. Measurements will be recorded at each of five concentration levels. The accuracy and linearity tests will be performed independently for each monitor and need not be performed on the same day for all monitors.

3.6 Calibration/Zero Drift Checks

On Monday, Wednesday, and Friday, of the first and last weeks of testing during each phase, the monitors being tested will be supplied with an ammonia gas standard and zero air to check the calibration and zero drift of the monitors, respectively. It is expected that the order in which the monitors are to be checked will be randomly varied, as will the time of day.

3.7 Interference Testing

During the second phase of testing, the monitors being tested will each be supplied with several compressed gas standards containing different interferent compounds. The interference testing will be conducted performed independently for each of the monitors being tested and need not be performed on the same day for all monitors. Table 3-1 shows the interferent compounds to be supplied to the monitors and the approximate concentrations of these species.

Table 3-1. Interferent Gases and Approximate Concentrations

Interferent Gas	Approximate Concentration to be Supplied (ppb)
Nitrogen dioxide	100 ppb
Hydrogen sulfide	100 ppb
Diethylamine	100 ppb
1,3-butadiene	100 ppb

4.0 TEST PROCEDURES

4.1 Preparation of Sampling Media

The procedures to be used for the preparation of the sampling media are described below and are based on the procedures given in the EPA Compendium Method IO-4.2 Determination of Reactive Acidic and Basic Gases and Acidity of Fine Particles (< 2.5 μm)[4].

During each phase, preparation of the sampling media will be performed at a USDA National Soil Tilth Laboratory (NSTL) facility nearby to the respective test sites. To the extent possible the preparation of the denuders will be performed in an ammonia free glove box to prevent contamination. However, the NSTL has a semi-automated system for denuder preparation and extraction that will be used in the first phase of this test. This system in not located in an ammonia free environment but minimizes the exposure of the denuder channels to the atmosphere, thereby limiting potential contamination.

After the sampling media are prepared, they will be stored in the glove box until they are transported to the test site for installation in the sampling trains.

4.1.1 Denuder Cleaning

The procedure for cleaning the denuders is given below. This procedure will be used for all new and used denuders.

1. Wash the denuders thoroughly with distilled water for at least 2 minutes ensuring that all the channels in the denuder have been flushed.
2. After flushing, rinse the denuder with Milli-Q water at least three times, ensuring that each channel has been rinsed.

3. After rinsing, gently tap the denuder on a clean Kimwipe to shake out excess water.
4. Place all cleaned denuders on a clean Kimwipes on a clean surface and allow the denuders to air dry. **Note: if it is necessary to rapidly dry the denuders, they may be rinsed in methanol and allowed to air dry, or clean air may be blown through them.**
5. Once dry, cap both ends of the denuder with clean end caps and store them in an ammonia-free glove box until use.

4.1.2 Denuder Preparation

The procedure used to coat the denuders is given below. There are two types of caps that are needed for this process. One type of cap should have a solid face and the other should have a small hole in the center of the face to prevent pressure buildup during the preparation process. The cap with the hole in the face should not be used to store the denuder after preparation. Ensure that there are two solid caps available for the denuder storage.

1. Using clean gloves (disposable polyurethane, or comparable), cap one end of the denuder with a solid face cap and set upright on the capped end. Pour approximately 10 mL of 1% citric acid solution into the denuder.
2. Cap the open end of denuder using the cap with a hole. Cover the hole in the face of the cap and gently turn and rotate the denuder to ensure that the solution is distributed into all channels of the denuder.
3. Remove one cap and pour out excess coating solution. Remove the other cap and gently shake excess solution from denuder.
4. Attach denuder to drying assembly and flow clean, dry air through the denuder for approximately 10 minutes at approximately 10 liters per minute (lpm).

5. Remove denuder from drying assembly, and cap both ends with solid face caps. Store the capped denuder in the glove box until packed for transport to the site. Using the unique identification number inscribed on the denuder, record the date and time that the denuder was coated.

The denuders should be carefully packed to prevent breakage during transport to the test sites. The denuders should be used within 72 hours of being coated, and should be used within 24 hours of being transported to the field. A completed chain-of-custody form (Appendix B) must accompany the denuders during transport to and from the field.

4.1.3 Sampling Cartridge Assembly

The following is the procedure for assembling the sampling cartridges. Cartridge assembly should be performed in the laboratory on a clean bench-top, or in an ammonia-free glove box if available.

1. With clean (disposable polyethylene, or comparable) gloves, place a glass spacer on a clean Kimwipe.
2. Place the filter pact outlet port inside the spacer.
3. Place a Teflon™ filter inside the filter pack outlet port followed by three clean plastic filter screens.
4. Place the cartridge assembly housing inside the filter pack outlet port and fasten the housing to the top rim of the cartridge.
5. Hold the cartridge housing at a slight angle and insert the following components in order.

    one metal spring
    one HDPE ring spacer
    one glass spacer

one HDPE ring spacer
one citric acid coated denuder
one HDPE ring spacer
one glass spacer
a greased inlet impactor plate

(Note: For breakthrough checks substitute a second citric acid denuder for the first glass spacer.)

6. Slide the sample inlet into the cartridge housing and secure it with the side clips.
7. Place plastic caps on both ends of the cartridge to prevent contamination.
8. Attach label to cartridge indicating date packed,the denuder identification, and the filter identification.

Cartridges should be assembled in the laboratory at room temperature and transported to the test sites when possible. A completed chain-of-custody form must accompany the cartridges during transport to and from the field.

4.2 Sample Collection

Sampling should be performed as closely to the scheduled times as possible, with the start and stop times ideally within 15 minutes of the scheduled times. The procedure for sample collection is given below.

1. Allow the pump to warm up for approximately 5 minutes.
2. Assemble the sampling train as shown in Figure 3-2. Seal the open end of the inlet using a cap or stopper. Run the leak check for 5-10 seconds. The flow meter should indicate no flow if there are no leaks in the system. If there are no leaks indicated, turn off the pump and remove the cap or stopper from the inlet. If a leak is indicated, check the assembly for proper connections. Replace any

suspect gaskets or defective components. Record activities on field data sheet (Appendix A).

3. Turn the pump on and set the sample flow rate to 10 lpm.
4. Complete appropriate sections on field data sheet. Begin sampling.

4.3 Sample Retrieval

Sample retrieval should be performed as soon after the end of each sampling period as possible. Care must be taken to avoid sample contamination during retrieval.

1. After sampling period is over, complete the appropriate sections on the field data sheet.
2. Turn off pump.
3. With clean gloves (disposable polyethylene, or comparable), remove sampling cartridge and cap both ends with clean caps.
4. Install new sampling cartridge as needed.
5. Pack the capped sampling cartridge for transport to the laboratory. Complete the chain of custody form.

4.4 Sample Extraction

Sample extraction should be done as soon after sample collection as feasible (within 24-hours of sample collection) and should be done on the same day as the analysis. Extraction of the samples should be performed in an ammonia free glove box to prevent sample contamination. (Note: for this verification test, only the denuders will be extracted and analyzed. The filter may be archived for analysis at a later date as needed.)

1. Remove both caps from the denuder. Wipe the outer surface of the denuder with a Kimwipe moistened with Milli-Q water.
2. Place a clean solid faced cap on one end of the denuder, and pipet 10 mL of DDW into the denuder. Cap the open end of the denuder using a cap with a hole in the face.
3. Cover the hole in the face of the cap and gently turn and rotate the denuder to ensure that the solution is distributed into all channels of the denuder.
4. Holding the denuder so that the red cap with the hole in its is on top, shake the denuder to force the liquid to the bottom.
5. Remove the red cap from the top of the denuder. Hold the bottom red cap firmly and carefully remove the denuder from the cap. Most of the solution should remain in the cap. Gently shake the denuder again to get the remaining solution out.
6. Pour the extraction solution into a labelled sample vial and cap the vial tightly.
7. Store the vial in the glove box until analysis. (**Note: if analysis can not be performed within 24 hours of extraction, the samples should be frozen until analysis can be performed**).

4.5 Sample Analysis

Sample analysis will be performed by Flow injection analysis (FIA). FIA is a continuous flow method for rapidly processing samples. In this method a peristaltic pump draws sample from the sampler into the injection valve. Simultaneously, reagents are continuously pumped through the system. The sample is loaded into the sample loop of one or more injection valves. The injection valve is then switched to connect the sample loop in line with the carrier stream. This sweeps the sample out of the sample loop and onto the manifold. The sample and reagents

then merge in the manifold (reaction module) where the sample can be diluted, dialyzed, extracted, incubated and derivatized. Mixing occurs in the narrow bore tubing under laminar flow conditions. For each method, the operating parameters are optimized to address high sample throughput, high precision and high accuracy.

FIA of the collected samples will be conducted by USDA staff on the same day as the samples are extracted. The analysis will be done according to the QuikChem Method No. 10-107-06-2-A (Lachat Company, Loveland, CO). In this method, ammonia is heated with salicylate and hypochlorite in an alkaline phosphate buffer forming an emerald green color which is proportional to the ammonia concentration. The color is intensified by the addition of sodium nitroprusside and monitored photometrically. The FIA instrument will be calibrated on each day prior to analysis of the samples.

4.6 Accuracy/Linearity Checks

During the first week of each phase of testing the monitors being tested will independently be supplied with compressed ammonia gas standards to achieve measurements over a range of concentrations from approximately 0 - 2000 ppb (or the upper range of measurement of the monitor being tested, whichever is lower). The gases delivered to the monitors will be prepared by dilution of higher concentration ammonia standard gases (i.e., 100-500 ppm) in zero air using a calibrated dilution system, and the degree of dilution will be dependent on the nature of the technology being tested (i.e., point source or open path).

Accuracy and linearity will be assessed by establishing a multipoint calibration curve. To establish this curve, three non-consecutive measurements will be recorded at each of five different nominal concentration levels. The gas will be supplied to the monitor for at least two minutes or until a stable reading is achieved, (i.e., no apparent increase in signal is observed), whichever is later. Once a stable reading is achieved, the gas will be delivered for two more minutes, the concentration will be recorded, and the monitor will be supplied the next standard gas. Table 4-1 shows the nominal concentration values to be supplied to the monitors being

tested and the order in which the concentrations will be supplied. After the last measurement has been recorded, the monitor will be flushed with zero air for approximately 5 minutes.

Table 4-1. Nominal Ammonia Concentrations and Order for Accuracy/Linearity Checks

	Concentration				
	0 ppb	200 ppb	600 ppb	1200 ppb	2000 ppb
Measurement Number	1	2	3	4	5
	7	10	6	9	8
	11	12	13	14	15

4.7 Calibration/Zero Checks

On Monday, Wednesday, and Friday of the first and last weeks during the two phases of the verification test, a compressed ammonia gas standard will be supplied to each of the monitors being tested. The gas should be supplied to the monitor for at least two minutes or until a stable reading is achieved, (i.e., no apparent increase in signal is observed), whichever is later. Once a stable reading is achieved, the concentration should be recorded, and the monitor will be supplied with zero gas. Zero gas will be supplied to the monitor for at least two minutes or until a stable reading is achieved (i.e., no apparent decrease in signal is observed), whichever is later. Once a stable reading has been achieved, the concentration should be recorded and the monitor can resume sampling the ambient air. At least once during each phase, the time required to reach a stable reading for both the calibration check and the zero checks will be recorded for each monitor being tested. This time will be used to assess the response time of the monitors.

4.8 Interference Checks

During week two of testing in the second phase, the monitors being tested will independently be supplied with a series of interference gases (see Table 3-1). Prior to supplying the interferent gas, zero air will be supplied to the monitors being tested for at least two minutes or until a stable reading is achived (i.e., no apparent increase in signal is observed), whichever is later. Once a stable reading has been achieved the monitor response will be recorded and the interferent gas will be diluted with zero air and delivered to the monitors seperately for at least two minutes each or until a stable reading is achieved, (i.e., no apparent increase in signal is observed), whichever is later. Once a stable reading has been achieved, the concentration should be recorded, monitor will be flushed for at least two minutes with zero air and approximately 500 ppb of ammonia will be supplied to the monitor gas. The gases will be supplied for at least two minutes each or until a stable reading is achieved, (i.e., no apparent increase in signal is observed), whichever is later. Once a stable reading has been achieved, the interferent gas will be supplied to the monitor for at least two minutes, or until a stable reading is achieved, (i.e., no apparent increase in signal is observed), whichever is later. Once a stable reading has been achieved, the concentration should be recorded and zero air will be supplied to the monitor for approximately five minutes. This process should be repeated for each of the interferent gases.

5.0 STATISTICAL CALCULATIONS

The statistical calculations to be used to verify the ammonia monitor performance are described below. All calculations will be performed using Microsoft Excel™.

5.1 Relative Accuracy

The relative accuracy (RA) of the ammonia monitor with respect to the compressed gas standards will be assessed using Equation 1:

$$RA = \frac{1}{n}\left(\sum_{i=1}^{n}\frac{|d_i|}{x_i}\right) \times 100 \qquad (1)$$

where *d* refers to the difference between the standard gas concentration and the average of the ammonia monitor measurements recorded during sampling period, and *x* corresponds to the standard gas concentration. For open path monitors, relative accuracy will be based on the observed increase in signal over the background concentration measured. Relative accuracy will be calculated and reported independently for each of the two phases.

5.2 Linearity

Linearity will be assessed by a linear regression analysis using the compressed standard gas concentrations as the independent variable and results from the ammonia monitors being tested as the dependent variable. Linearity will be expressed in terms of slope, intercept, and coefficient of determination (r^2), and will be calculated independently for each phase of the verification test.

5.3 Precision

Precision will be calculated in terms of the percent relative standard deviation (RSD) of a series of ammonia monitor measurements made over the duration of each of the calibration and zero drift check periods. During each calibration and zero check all readings from monitors testing will be recorded, and the mean and standard deviation of those readings will be calculated. Precision (*P*) will then be determined as:

$$P = \frac{SD}{\overline{X}} \times 100 \quad \textbf{(2)}$$

where *SD* is the standard deviation of the monitor readings and $\overline{X}$ is the mean of the monitor readings. Precision will be calculated independently for each phase of testing.

5.4 Comparability

Comparability between the ammonia monitor results and the reference method results will be assessed by linear regression using the reference method ammonia concentrations as the independent variable and results from the ammonia monitors being tested as the dependent variable. For the open path monitors, the average of the reference method results along the optical path will be used as the independent variable. Linearity will be expressed in terms of slope, intercept, and coefficient of determination (r^2), and will be calculated independently for each phase of the verification test. It is expected that the measured concentration of ammonia will vary by at least a factor of five during each phase of testing. If this magnitude of variation is not achieved during either of the phases then comparability for that phase will be calculated using Equation 1 and reported as a percent difference rather than in terms of the linear regression results.

5.5 Calibration and Zero Drift

Calibration and zero drift will be reported in terms of the mean, relative standard deviation, and range (maximum and minimum) of the readings obtained from the ammonia monitor in the daily sampling of the same ammonia standard gas, and of zero gas. The calibration and zero drift will be calculated independently during each phase of testing such that up to 6 ammonia standard readings (Monday, Wednesday, and Friday for two weeks), and up to 6 zero readings, will be used for this calculation in each phase. This calculation, along with the range of the data, will indicate the day-to-day variation in zero and standard readings.

5.6 Interference Effects

The extent of interference will be calculated in terms of the ratio of the response of the monitor to the interfering species, relative to the actual concentration of the interfering species. For example, if 100 ppb of an interfering species results in a 1 ppb change in the response of the monitor, the interference effect will be reported as 1% (i.e., 1 ppb/100 ppb). The interference effects will be reported seperately for each interferent both in the absence and in the presence of ammonia.

5.7 Response Time

Response time will be assessed in terms of both the rise and fall times of each ammonia monitor when sampling the ammonia gas standard. Rise time (i.e., 0% - 95% response time) will be determined by recording all the monitor readings when the gas is supplied to the monitor. Once a stable response has been achieved with the gas standard, the fall time (i.e., the 100% to 5% response time) will be determined in a similar way, by recording all monitor readings as the gas supplied is switched from the ammonia standard back to zero gas. For monitors which

provide periodic rather than continuous readings, determination of rise and fall times may involve interpolation between readings.

Rise and fall times will each be determined once for each monitor during each phase of testing. Rise and fall times will be reported in units of seconds.

6.0 MATERIALS AND EQUIPMENT

6.1 Sampling Media and Equipment

The denuders, filters, and associated sampling equipment for collection of the reference method[4] sampling will be supplied by Battelle and/or USDA. The sampling cartridges used for this test will be ChemComb Model 3500 speciation sampling cartridges[5] (Rupprecht & Patashnick, Co., Inc., Albany, NY). Multiple sampling trains will be available such that five sets of four trains (i.e., five sampling runs with four trains each) may be sampled in a single day, in addition to at least two blank samples, two breakthrough checks, and two duplicate samples per day during the intensive sampling periods.

6.1.1. Denuders

The denuders used for the reference sample collection will be honeycomb type denuders used in the ChemComb sampling cartridges. The denuders will be coated with a 1% citric acid solution, analogous to the 1% citric acid solution used in the EPA standard method[3]. The solvents and chemicals used to prepare the denuders for sampling and to extract the denuders after sampling will be ACS reagent grade, and will be purchased from a reliable supplier. Reagents and solvents will be analyzed for ammonia prior to use to ensure no contamination is present.

6.1.2 Filters

The filters to be used for the reference sample collection will be 47 mm diameter Teflo™ filters (e.g., Gelmen Sciences, part number R2PJO47, or comparable).

6.1.3 Sampling Train Components

Each of the sampling trains will include a pump to draw the air through the sampling media, and a flow meter, or flow controller to maintain a nominally constant flow rate. The ChemComb sampling cartridges have an internal impactor to remove particulate matter with an aerodynamic diameter greater than 2.5 μm.

6.2 Analytical Equipment

Analysis of the collected samples will be conducted by flow injection analysis (FIA) using a Lachat QuikChem Model 8000 FIA+. The equipment and chemicals to be used for sample analysis will be provided by USDA and will be at the USDA facilities used during each phase of testing. All chemicals used for the sample extraction and analysis will be reagent grade.

6.3 Compressed Gases

6.3.1 High Purity Nitrogen/Air

The high purity gases used for zeroing of the monitors will be commercial ultra-high purity (UHP, i.e., minimum 99.999% purity) air or nitrogen.

6.3.2 Ammonia Standard Gases

Compressed gas standards containing ammonia will be obtained for use in the calibration drift checks of the monitors. These will consist of ammonia in a nitrogen matrix, at levels of approximately 100-500 ppm. These gases will be diluted as necessary to achieve ammonia concentrations in the range of approximately 200-2000 ppb.

6.4 Meteorological Equipment

Meteorological conditions during each phase of testing will be monitored continuously using a portable meteorological station (i.e., MetOne Instruments, or comparable). The station will include sensors for ambient temperature, relative humidity, barometric pressure, wind speed, and wind direction. Data from the station will be recorded continuously using a datalogger, and will be analyzed by the datalogger to provide hourly averages for the measured parameters.

7.0 QUALITY ASSURANCE/QUALITY CONTROL

7.1 Equipment Calibrations

7.1.1 Reference Method Sampling Equipment

Reference method sampling will be conducted according to the procedures described in the EPA standard method[4]. A single point calibration of the flow rate through each of the sampling systems (i.e., pump, flow controller, filter pack, denuder, cyclone) will be performed prior to the start of each phase using a dry gas meter with NIST-traceable calibration. The flow rate of each sampler will be checked at least once per day at the beginning and end of one sampling period using a dry gas meter or other flow meter. The flow rate will be recalibrated if the calibration check is not within ±5% of the nominal flow rate of 10 lpm (i.e., 9.5 lpm - 10.5 lpm). All calibration results must be documented for inclusion in the verification test data files and verification report.

7.1.2 Analytical Equipment

Analysis of the reference samples will be conducted in a laboratory using FIA. The FIA system for the reference sample analysis will be calibrated by USDA staff performing the analysis. The calibration will be conducted according to the manufacturer recommendations and will include concentrations of ammonia standard solutions that bracket the expected concentration of the sample solutions. Calibration standards will be run at the beginning and end of each analytical session, with one calibration solution run after each tenth reference sample. The calibration will be acceptable if the r^2 of the calibration curve is >0.98, and if the calibration checks agree within 10% of the standard solution concentration. All calibration results must be documented for inclusion in the verification test data files and verification report.

7.1.3. Meteorological Equipment

The sensors used for the meteorological monitoring will be calibrated by the manufacturer within one year of use in this verification test. All calibration results must be documented for inclusion in the verification test data files and verification report.

7.2 QA/QC Samples

7.2.1 Field Blanks

At least 10% of all samples collected will be field blanks. The field blanks will be collected by installing the sampling media (i.e., denuder and filters) in the sampling train but without drawing any air through the train. The media will then be recovered and handled like normal samples. If contamination levels are detectable and are greater than 5% of any of the measured sample concentrations for that day, no additional reference sampling will be conducted until the cause of the contamination is identified and rectified. Care will be taken to ensure that field blanks will be collected at each of the different sampling locations and during each of the different sampling periods (e.g., 8:00 a.m. to 12:00 p.m., etc.). Field blanks should be analyzed within 24 hours of collection to allow for prompt corrective action if needed.

7.2.2 Denuder Breakthrough Checks

Back-up denuders will be used with at least 10% of the samples collected to assess the degree of ammonia breakthrough. These breakthrough checks will be conducted at each of the four sampling locations and will include checks during each of the five sampling periods. At least one breakthrough check will be performed during each of the sampling periods during the first two days of sampling during each phase to allow for prompt corrective action if needed. If the measured ammonia concentration on the back-up denuders is greater than 10% of the

concentration on the front denuder, then back-up denuders will be used for all sample collection during those periods during which breakthrough might be anticipated to occur. Denuder breakthrough checks should be analyzed within 24 hours of collection to allow for prompt corrective action if needed.

7.2.3 Duplicate Samples

For at least 10% of samples, duplicate samples will be collected using a co-located sampling train. These duplicate samples will be collected at each of the sampling locations and during each of the sampling periods. It is expected that the difference in the measured ammonia concentrations between duplicate samples will be within 10% of the average concentration (i.e., difference divided by average < 10%). If relative differences greater than 10% are observed, reference sampling will continue and the cause of these differences will be investigated and rectified if possible.

7.2.2 Laboratory Blanks

Laboratory blank solutions will be prepared for the FIA analysis. These solutions will be analyzed prior to analysis of the reference samples. A laboratory blank solution will be analyzed after every 10^{th} reference sample to ensure no drift in the FIA instrumentation. If the blank levels are greater than 5% of any of the measured concentrations for that day, the cause of the contamination will be investigated and rectified (if possible). All analyses performed after the most recent acceptable blank will be invalidated and analysis of those samples will be repeated (if possible).

7.2.3 Calibration Checks

Calibration check solutions will be prepared using NIST-traceable ammonia solutions. The calibration check solutions will be analyzed after every 10th reference sample to ensure no drift in the FIA instrumentation. If the measured concentrations are not within ±10% of the standard solution concentration, the cause of the discrepancy will be investigated and rectified if possible. If such a discrepancy is observed, all analyses performed after the most recent acceptable spike will be invalidated and analysis of those samples will be repeated (if possible).

7.2.4 Dilution Checks

At each of the nominal ammonia levels to be used for the accuracy and linearity checks, at least one sample of the diluted will be collected using the reference method. These samples will be analyzed as regular samples and will used to check the accuracy of the dilution system. Agreement between the reference sample measurement and the calculated concentration from the dilution system should be within 10%. If the reference method result and the calculated concentration do not agree within 10%, no further accuracy or linearity checks will be completed until the source of the discrepancy is investigated and rectified.

7.3 Assessment and Audits

7.3.1 Technical Systems Audits

Battelle's ETV Quality Manager, will perform a technical systems audit (TSA) once during at least one phase of this verification test. The purpose of this TSA is to ensure that the verification test is being performed in accordance with this test/QA plan and that all QA/QC procedures are being implemented. In this audit, the Battelle ETV Quality Manager may review the reference sampling and analysis methods used, compare actual test procedures to those

specified in this plan, and review data acquisition and handling procedures. The Battelle ETV Quality Manager will prepare a TSA report, the findings of which must be addressed either by modifications of test procedures or by documentation in the test records and report.

At EPA's discretion, EPA QA staff may also conduct an independent on-site TSA during the verification test. The TSA findings will be communicated to testing staff at the time of the audit, and documented in a TSA report.

7.3.2 Performance Evaluation Audit

A performance evaluation (PE) audit will be conducted to assess the quality of the measurements made in this verification test. This audit addresses only those measurements that factor into the data used for verification, i.e., the sample flow rate, and the analytical laboratory measurements. This audit will be performed once during the verification test, and must be performed by analyzing a standard or comparing to a reference that is independent of standards used during the testing.

The flow rate of the reference method sampling assemblies will be audited once during each phase of testing using a dry gas meter (or other flow meter) that is independent of the meter used to calibrate the flow rate. Agreement between the audit flow rate and the nominal flow rate should be within ±5% (i.e., 9.5 lpm - 10.5 lpm). If agreement between the audit flow rate and the nominal flow rate is not within ±5%, then the flow rate of the sampler will be recalibrated.

The performance of the FIA used to analyze the reference samples will be audited by analyzing an ammonium standard that is independent of those used for the calibration. This sample will be provided as a blind audit sample and the operator of the FIA will not be aware of the concentration of the sample. If agreement between the measured concentration and the standard concentration is not within ±10%, the cause of the discrepancy will be investigated and rectified if possible. This audit will be performed at least once during each phase of the verification test.

7.3.3 Data Quality Audit

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

7.3.4 Assessment Reports

Each assessment and audit will be documented in accordance with Section 3.3.4 of the QMP for the AMS Center.[1] Assessment reports will include the following:

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

7.3.5 Corrective Action

The Battelle Quality Manager during the course of any assessment or audit will identify to the technical staff performing experimental activities any immediate corrective action that should be taken. If serious quality problems exist, the Battelle Quality Manager is authorized to stop work.

Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem, and will implement any necessary followup corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken.

8.0 DATA ANALYSIS AND REPORTING

8.1 Data Acquisition

Data acquisition in this verification test includes recording of the data from the monitors undergoing testing, documentation of sampling conditions and analytical results from the reference method, meteorological conditions, and recording of testing activities, such as the times of test activities, etc.

Data acquisition for the monitors undergoing verification will be performed by the vendors during the test. Each monitor must have some form of data acquisition device, such as a printout of analyzer response, or an electronic data recorder that stores individual analyzer readings. The vendor will be responsible for reporting the response of the monitor, for each of the phases of the verification test. The data from the monitors are to be retrieved by, or provided to Battelle regularly, and must include all individual readings of the monitor listed by time of day. Averaged results, e.g., ammonia data averaged over the period of a reference method sampling run, may also be provided, if available. If not provided, averaging will be performed by Battelle in data processing. Electronic data files are the preferred means of data transfer, with Excel® or comma separated variable file formats preferred. Electronic files requiring vendor's proprietary software will be supplied along with the software required to view the data.

Other data will be recorded in laboratory record books provided by Battelle and maintained by Battelle, vendor, and subcontractor staff involved in the testing. These records will be reviewed by Battelle to identify and resolve any inconsistencies. All written records must be in ink. Any corrections to notebook entries, or changes in recorded data, must be made with a single line through the original entry. The correction is then to be entered, initialed and dated by the person making the correction.

In all cases, strict confidentiality of data from each vendor's monitor, and strict separation of data from different monitors, will be maintained. Separate files (including manual

records, printouts, and/or electronic data files) will be kept for each monitor. At no time during verification testing will Battelle or USDA staff engage in any comparison in performance of the participating monitors.

Table 8-1 summarizes the types of data to be recorded; where, how often, and by whom the recording is made; and the disposition or subsequent processing of the data. The general approach is to record all test information immediately and in a consistent format throughout all tests. Data recorded by the vendors is to be retrieved by, or turned over to Battelle or USDA staff regularly during each phase of testing. Identical file formats will be used to make quantitative evaluations of the data from all of the monitors tested, to assure uniformity of data treatment. This process of data recording and compiling will be overseen by the Verification Test Coordinator.

8.2 Data Review

Records generated in the verification test will be reviewed by a Battelle staff member within two weeks after the completion of each phase of testing, before these records are used to calculate, evaluate, or report verification results. These records may include laboratory record books; data from the monitors; or reference method analytical results. This review will be performed by a Battelle technical staff member involved in the verification test, but not the staff member that originally generated the record. The test site, USDA staff, and/or vendor representatives will be consulted as needed to clarify any issues about the data records. The review will be documented by the person performing the review by adding his/her initials and date to a hard copy of the record being reviewed. Data relating to the reference samplinge from the first week of each phase will be reviewed by a Battelle staff member within 48 hours of generation to allow for any corrective actions to be implemented promptly.

Table 8-1. Summary of Data Recording Process

Data to be Recorded	Responsible Party	Where Recorded	How Often Recorded	Disposition of Data[(a)]
Dates, times of test events (site activities, etc.)	USDA/ Battelle staff	Laboratory record books/field data sheet	Start/end of test, and at each test activity.	Used to organize/check test results; manually incorporated in data spreadsheets as necessary.
Reference method sampling data	USDA/ Battelle staff	Laboratory record books, CoC forms, or file data sheets as appropriate	At least at start/end of reference sample, and at each change of a test parameter.	Used to organize/check test results; manually incorporated in data spreadsheets as necessary.
Meteorological conditions	Battelle	Meteorological station datalogger	Continuously	Used to assess meteorological conditions during testing as necessary.
Ammonia monitor readings	Vendor or designee	Data acquisition system (data logger, PC, laptop, etc.).	Continuously at specified acquisition rate throughout monitor operation.	Electronically transferred to spreadsheets
Reference sample analysis, and results	USDA/ Battelle staff	Laboratory record books, data sheets, or data acquisition system, as appropriate.	Throughout sample handling and analysis process	Transferred to spreadsheets

(a) All activities subsequent to data recording are carried out by Battelle.

8.3 Reporting

The statistical data comparisons described in Section 5.0 will be conducted separately for each commercial ammonia monitor tested. Separate verification reports will then be prepared, each addressing the monitor provided by one commercial vendor. The verification report will present the test data, as well as the results of the statistical evaluation of those data.

The verification report will briefly describe the ETV program and the AMS Center, and will describe the procedures used in verification testing. These sections will be common to each verification report resulting from this verification test. The results of the verification test will then be stated quantitatively, without comparison to any other monitor tested, or comment on the acceptability of the monitor's performance. The preparation of draft verification reports, the review of reports by vendors and others, the revision of the reports, final approval, and the distribution of the reports, will be conducted as stated in the Generic Verification Protocol for the Advanced Monitoring Systems Pilot.[6] Preparation, approval, and use of Verification Statements summarizing the results of this test will also be subject to the requirements of that same Protocol.

9.0 HEALTH AND SAFETY

The first phase of the verification test described in this test/QA plan will be performed in Ames, Iowa, and the second phase of the verification test will be performed in Bushland, Texas. All visiting staff at the AFO test site may be given a site-specific safety briefing by a representative of the test site .prior to the installation and operation of the monitors. All participants in this verification test (i.e., Battelle, USDA, EPA, and vendor staff) will adhere to the health and safety requirements of the test site. All equipment brought onto the test site will conform to the biosecurity protocol of the test site.

10.0 REFERENCES

1. Quality Management Plan (QMP) for the ETV Advanced Monitoring Systems Center, U.S. EPA Environmental Technology Verification Program, prepared by Battelle, Columbus, Ohio, Version 4.0 December 2002.

2. National Air Pollutant Trends, 1900-1998. EPA-454/R-00-02, U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards, Research Triangle Park, NC, 27711.

3. Environmental Technology Verification Program Quality Management Plan, EPA/600/R-03/021, December 2002.

4. Determination of the Strong Acidity of Atmospheric Fine Particles (<2.5 μm) Using Annular Denuder Technology, EPA/600/R-93/037, U.S. Environmental Protection Agency, Office of Research and Development, Research Triangle Park, North Carolina, 27711.

5. Operating Manual - ChemComb Model 3500 Speciation Sampling Cartridge, Revision A, Rupprecht & Patashnick, Co., Inc., Albany, NY, January 2000.

6. Generic Verification Protocol for the Advanced Monitoring Systems Pilot, Battelle, Columbus, Ohio, November 1998.

Appendix A
Example Field Data Sheet

ETV Verification of Ambient Ammonia Monitors
Reference Method Field Data Sheet

GENERAL

Site: _____ **Date:** _____
Sample ID: _____ **Operator:** _____

EQUIPMENT

MFC No.: _____ **Denuder ID:** _____
Flow Rate Set Point: _____ **Filter ID:** _____

SAMPLING DATA

Set Up Operator: _____ **Retrieval Operator:** _____
Start Time: _____ **Stop Time:** _____
Leak Check Before: _____ **Leak Check After:** _____

Time	Flow Rate (lpm)	Ambient Temp. (C)	Barometric Pressure (mm Hg)	Rel. Humidity (%)	Comments

Operator Comments:

_____
_____
_____
_____
_____

Appendix B
Example Chain of Custody Sheet