

SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

METHOD 207.1

**DETERMINATION OF AMMONIA EMISSIONS
FROM STATIONARY SOURCES**

SOURCE TEST ENGINEERING/ LABORATORY SERVICES

MONITORING AND ANALYSIS

April 2006

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TABLE OF CONTENTS

	PAGE
Table Of Contents	i
List Of Figures	iv
1.0 Overview	1
1.1 Applicability	1
1.2 Principle	1
1.3 Method Performance	2
1.3.1 Accuracy	2
1.3.2 Precision	2
1.3.3 Interferences	3
1.3.4 Range	3
1.3.5 Sample Storage Time	3
1.3.6 Duplicate Sampling	3
2.0 Sampling Preparation	4
2.1 Planning	4
2.1.1 Pre-Test Protocol (Optional)	4
2.1.2 Sampling Technique	4
2.1.3 Field Blank Trains	5
2.1.4 Fixed Gases Measurement	5
2.2. Equipment	5
2.2.1 Greenburg-Smith Impinger Train	5
2.2.1.1 Nozzle and Probe	5
2.2.1.2 Filter Holder	6
2.2.1.3 Impinger Train	6
2.2.1.4 Metering System	6
2.2.2 Mini-Impinger Train	7
2.2.2.1 Sampling Line	7
2.2.2.2 Impinger Train	7
2.2.2.3 Metering System	8
2.3 Sampling Reagents	8
2.3.1 Filters	8
2.3.2 Silica Gel	8
2.3.3 Water	8
2.3.4 0.1 N Sulfuric Acid Solution	8
2.3.5 Other Supplies	8

Table of Contents, Continued

	PAGE
2.4 Sample Train Assembly.....	9
2.4.1 Greenburg-Smith Train Assembly.....	9
2.4.1.1 Probe and Nozzle	9
2.4.1.2 Filter and Filter Holder	9
2.4.1.3 Impinger Train	9
2.4.2 Mini-Impingers Train Assembly.....	10
2.4.2.1 Sampling Line.....	10
2.4.2.2 Mini-Impinger Train	10
3.0 Field Procedures.....	11
3.1 Sampling With Greenburg-Smith Impinger Train.....	11
3.1.1 Calibrations	11
3.1.2 Pre-Test Determinations	11
3.1.3 Field Train Assembly.....	12
3.1.4 Pre-Test Leak Check.....	12
3.1.5 Sampling Operation	13
3.1.6 Post-Test Procedures.....	15
3.2 Mini-Impinger Train.....	16
3.2.1 Calibrations	16
3.2.2 Pre-Test Determinations	17
3.2.3 Field Train Assembly.....	17
3.2.4 Pre-Test Leak Check.....	17
3.2.5 Sampling Operation	17
3.2.6 Post-Test Procedures.....	18
4.0 Laboratory Procedures	19
4.1 Sample Recovery	19
4.1.1 Greenburg-Smith Impinger Train	19
4.1.2 Mini-Impinger Train	20
4.2 Analysis	21
4.2.1 Equipment	21
4.2.2 Reagents.....	21
4.2.3 Calibration.....	22
4.2.4 Analytical Procedure.....	23
4.2.4.1 Greenburg-Smith Impinger Train	24
4.2.4.2 Mini-Impinger Train	25
4.2.4.3 Sample Dilutions.....	26

Table of Contents, Continued

	PAGE
4.3 Calculations	26
4.3.1 Calibration Standards.....	26
4.3.2 Calibration Regression Line	27
4.3.3 Measured Concentration Of Ammonia.....	27
4.3.4 Average Measured Ammonia Concentration (Cavg)	27
4.3.5 Relative Percent Difference (RPD).....	27
4.3.6 Relative Percent Accuracy (RPA)	28
4.3.7 Matrix Spike Percent Recovery (%R).....	28
4.4 Quality Control	28
4.4.1 Initial Calibration.....	28
4.4.2 Calibration Verification	29
4.4.3 Measurement Stability	29
4.4.4 Matrix Spike Recovery	29
4.4.5 Temperature Probe.....	30
5.0 Emissions Reporting.....	31
5.1 Engineering Calculations.....	31
5.2 Report Information	32
5.3 Report Format.....	33

Appendix A

Appendix B

LIST OF FIGURES

	PAGE
Figure 207.1-1 Ammonia Train With Greenburg-Smith Impingers.....	36
Figure 207.1-2 Ammonia Train With Mini-Impingers.....	36
Figure 207.1-3 Source Test Data Sheet	37
Figure 207.1-4 Regression Calculation Sheet	38
Figure 207.1-5 Source Test Calculation Sheet	39

METHOD 207.1

DETERMINATION OF AMMONIA EMISSIONS FROM STATIONARY SOURCES

Section 1 of 5

1.0 Overview

1.1 Applicability

This method applies to the determination of gaseous ammonia emissions from stationary sources. Provisions for stack and non-stack (flux chamber¹) sampling are included. Data may be used for compliance determination, performance tests for permitting, risk assessments, or other purposes within the SCAQMD region.

1.2 Principle

For sources with an emission stack, free (uncombined) ammonia is withdrawn non-isokinetically using a sampling train consisting of a probe, glass impingers containing a sulfuric acid solution, a calibrated meter, and a pump. Non-stack applications, such as flux chamber sampling, require a sampling train consisting of a flexible connector, mini-impingers containing a sulfuric acid solution, a pump, and a calibrated meter to collect ammonia. In both applications, ammonia is captured and retained in the acid solution for later recovery and analysis.

Optionally, if particulate matter (such as ammonium salts) is a concern, a nozzle and an in-stack filter may be installed upstream of the probe.

The probe rinse and impinger solutions are recovered and analyzed for ammonia and ammonium compounds by ion selective electrode (ISE). In the ISE procedure, ammonium is converted to

¹ Kienbusch, M. *Measurement of Gaseous Emission Rates from Land Surfaces Using an Emission Isolation Flux Chamber—User's Guide*. EPA 600/8-86-008 (NTIS PB86-223161), February 1986.

aqueous ammonia at an elevated pH through addition of a strong base. The ammonia diffuses through the instrument's gas-permeable membrane, thereby altering the pH of an internal solution. The magnitude of the pH change is sensed from a potentiometric measurement, which is related to an ammonia concentration by a calibration curve.

Moisture content shall be determined by a Method 4.1 analysis of each sample.

This method presents the configuration used during validation testing. Mention of trade names in this test method does not constitute endorsement by SCAQMD; the model names and numbers are given as those used during the validation phase of the method. Other manufacturers of equipment or modifications to the analytical procedures may be used subject to demonstration of equivalency as approved by the SCAQMD.

1.3 Method Performance

1.3.1 Accuracy

Using Greenburg-Smith sampling trains, limited results from audit gas sampling indicates a 2 percent positive bias in the measured concentration at approximately 10 ppm, and a 4 percent positive bias in the measured concentration at approximately 100 ppm. EPA method validation calculations indicate that bias adjustment is not necessary.

1.3.2 Precision

The method precision (both field sampling and analytical precision), as quantified by the relative standard deviation, was determined to be 10% based on duplicate field samples.

1.3.3 Interferences

Sampling interferences include excess moisture which may dilute the absorbing solution, or strong bases which may neutralize the absorbing solution. For applications using the optional in-stack filter, water droplets (if present at stack conditions) combined with particulate matter may clog the sampling system and will cause a low bias.

Analytical factors which may influence the results include: the ambient air and solution temperatures, the stirring rate, and the immersion angle and depth of the ISE probe in the sample solution.

1.3.4 Range

The estimated analytical limit of detection (LOD) is 0.1 ppmw. The method has been field validated at ammonia concentrations from 0 to 445 ppmv. Analyses at higher concentrations will require a separate analysis of each impinger solution to address a possible bias due to lower ammonia capturing efficiencies at higher pH levels.

The analytical range is from 1 to 100 mg/L. However, if the sample concentration is beyond the limits of the calibration curve, the range may be extended through sample dilution.

1.3.5 Sample Storage Time

The maximum sample storage time (following recovery) is 4 weeks.

1.3.6 Duplicate Sampling

Two test runs shall be conducted, with one sample train collected during each test run. For the purposes of this method, these two trains shall be considered a duplicate sample. Samples collected concurrently do not qualify as duplicate samples, unless approved in writing by the Executive Officer. The tester must report the results from both test runs separately.

METHOD 207.1

DETERMINATION OF AMMONIA EMISSIONS FROM STATIONARY SOURCES

Section 2 of 5

2.0 Sampling Preparation

2.1 Planning

A pre-test plan is essential for conducting a successful test. Among the considerations during the planning phase are the evaluation of the process operation, the levels of emissions, and the purpose for testing. These and other issues are discussed below, as well as in Chapters I and II of the District Source Test Manual.

2.1.1 Pre-Test Protocol (Optional)

The protocol must demonstrate that the testing parameters calculated by the tester will meet the needs of the SCAQMD. It must include all of the elements and be written in a format to include all information specified in **Appendix A**. The tester should not proceed with the performance of the remainder of this method until the pre-test protocol is approved by the SCAQMD.

The source test firm should determine the source target concentration, reporting limit, planned sample volume and planned sampling time and include it in the test protocol. A discussion of these pre-test determinations may be found in **Appendix B**.

2.1.2 Sampling Technique

This method allows for either stack sampling using Greenburg-Smith impingers, or non-stack sampling (such as flux chamber procedures) using mini-impingers. The testing laboratory must clearly identify which sampling technique will be used.

2.1.3 Field Blank Trains

A minimum of one blank sampling train shall be analyzed per test set, but no less than one blank train per day. The blank trains must accompany the sampling trains to the test site, and shall be assembled and recovered in the exact manner as the sampling trains. A separate analysis of each train must be performed and reported; however, these results are presented for quality assurance purposes only, and shall not be used to adjust the data of the emission sample trains.

2.1.4 Fixed Gases Measurement

For stack samples, emission limits for ammonia are generally normalized to a percent level of oxygen. Additionally, measurements of oxygen, carbon dioxide, and carbon monoxide are required for molecular weight corrections in flow calculations. Sampling and analysis for these gases shall be in accordance with SCAQMD Methods 10.1 or 100.1.

2.2. Equipment

2.2.1 Greenburg-Smith Impinger Train

A schematic of the sampling train with the Greenburg-Smith impingers is shown in Figure 207.1-1. This schematic shows the impinger train affixed to the probe. If a flexible connection between the probe and impingers is desired, use an inert tubing that can withstand the heat at the probe connection. The sampling train consists of the components below.

2.2.1.1 Nozzle and Probe

Use a glass or type 316 stainless steel nozzle and probe. For stack temperatures less than the softening point, a Teflon probe is acceptable as long as the sampling point is no closer than 3.3 ft. from the stack wall for stack diameters greater than 7 ft., or at the center of the stack for diameters less than or equal to 7 ft. The nozzle shall be configured in a button-hook or elbow

configuration. Since isokinetic sampling is not required, it is not necessary to measure the nozzle diameter.

2.2.1.2 Filter Holder

Use materials such as stainless steel, Teflon, or Viton which do not react with the particulate matter or sampled gases. The filter holder design must provide a positive seal against leakage from the outside or around the filter. Attach the holder at the outlet of the nozzle.

2.2.1.3 Impinger Train

The train consists of four impingers connected in series with leak-free ground glass fittings, or any similar leak-free non-contaminating fittings. The first and second impingers must be of the Greenburg-Smith design with the standard tip. The third and fourth impingers must also be of the Greenburg-Smith design, but with a modified tip comprised of a 1.3 cm (1/2 in.) ID glass straight-end tube extending to about 1.3 cm (1/2 in.) from the bottom of the flask.

The first and second impingers contain 100 ml of 0.1N sulfuric acid (H_2SO_4), the third is empty, and the fourth contains a known weight of silica gel, or equivalent desiccant.

2.2.1.4 Metering System

The metering system includes a vacuum gauge, a leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), a dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 207.1-1. An alternative to the thermometers and dry gas meters is an equivalent temperature-compensated dry gas meter.

2.2.2 Mini-Impinger Train

A schematic of the sampling train with the mini-impingers is shown in Figure 207.1-2. The train is set-up downstream of the flux chamber, or other emission source. In contrast with the Greenburg-Smith train design, note that the mini-impinger sampling train does not include an optional upfront filter.

2.2.2.1 Sampling Line

The sampling line shall be made of material which is inert to the sample gases. Examples of acceptable sampling lines are polyethylene or Teflon tubing.

2.2.2.2 Impinger Train

The train consists of four glass mini-impingers, approximately 30 ml in capacity, connected in series with leak-free ground glass fittings, or any similar leak-free non-contaminating fittings. The first and second impinger tips are sized such that the bubbles will impact the bottom of the impingers. The third and fourth impinger tips shall extend at least three-fourths of the way into the impinger. The impinger tips shall be a straight-end design. Upon request, the testing laboratory shall provide evidence, such as controlled spiking test results, that its mini-impinger design captures the ammonia gas sample within 10 percent of the expected concentration.

The use of flexible connections between the impingers is an acceptable alternative to glass fittings.

The first and second impingers contain 15 ml of 0.1N sulfuric acid (H_2SO_4), the third is empty, and the fourth contains a known weight of silica gel, or equivalent desiccant.

2.2.2.3 Metering System

The metering system consists of a leak-free pump, a calibrated flow meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 207.1-2.

2.3 Sampling Reagents

2.3.1 Filters

The in-stack filter shall be constructed from sintered metal. Alternately, glass or quartz fiber filters without organic binder may be used.

2.3.2 Silica Gel

Indicating-type, 6 to 16 mesh. Use new silica gel as received. If previously used, dry at 175°C (350°F) for 2 hours. Other desiccants may be used, subject to the approval of the Executive Officer.

2.3.3 Water

Deionized or distilled to conform to ASTM specifications D-1193-99, Type 3. Water shall be blank-checked for ammonium ion. The resistivity should be 15 megaohms-cm or higher.

2.3.4 0.1 N Sulfuric Acid Solution

Dilute 2.8 ml of concentrated sulfuric acid (18M) to 1 liter with deionized water to form 0.1 N sulfuric acid. Alternately, obtain by purchase of reagent grade 0.1N sulfuric acid. Reserve 200 ml as a reagent blank if Greenburg-Smith impinger trains are analyzed (see Section 4.1.1), or 30 ml if mini-impinger trains are analyzed (see Section 4.1.2).

2.3.5 Other Supplies

Crushed ice, or dry ice pellets.

2.4 Sample Train Assembly

During the preparation and assembly, keep all openings covered when contamination can occur until just prior to assembly. All equipment, including balances, temperature gages, glassware, and safety equipment should be checked for readiness before proceeding.

2.4.1 Greenburg-Smith Train Assembly

2.4.1.1 Probe and Nozzle

Prepare probe and sampling nozzles for use. Thoroughly clean each component with soap and water followed by a minimum of three rinses with deionized water. Cap or seal open ends to prevent contamination prior to sampling.

2.4.1.2 Filter and Filter Holder

For glass or quartz filters, check filters visually against light for irregularities, flaws, or pinhole leaks. Using tweezers or clean disposable surgical gloves, place a filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

2.4.1.3 Impinger Train

Assemble the impingers in the tray. Load each of the first two impingers with absorbing solution. Greenburg-Smith impingers shall contain exactly 100 ml of absorbing solution. Leave the third impinger empty. Load the last impinger with approximately 200 to 300 grams of silica gel. If moisture content is to be performed gravimetrically, weigh each impinger plus contents to the nearest 0.1 g and record the weights.

2.4.2 Mini-Impingers Train Assembly

2.4.2.1 Sampling Line

Acquire a clean or unused sampling line. Connection of the sampling line to the mini-impinger train may be accomplished either with compression fittings, or by mating the ends with an inert plastic sleeve.

2.4.2.2 Mini-Impinger Train

Place mini-impingers in the sampling holder. Load each of the first two mini-impingers with exactly 15 ml absorbing solution. The third impinger is empty. Load the fourth impinger with approximately 30 to 45 grams of silica gel or equivalent desiccant. If moisture content is to be performed gravimetrically, weigh each impinger plus contents to the nearest 0.1 g and record the weights.

METHOD 207.1

DETERMINATION OF AMMONIA EMISSIONS FROM STATIONARY SOURCES

Section 3 of 5

3.0 Field Procedures

3.1 Sampling With Greenburg-Smith Impinger Train

Stack sampling using the Greenburg-Smith style impinger train shall apply the procedures below.

3.1.1 Calibrations

Follow the calibration procedures and maintenance schedule in Chapter III of the SCAQMD Source Test Manual. Calibration results shall be submitted with the final test report.

3.1.2 Pre-Test Determinations

If the sampling location is less than eight diameters downstream, and two diameters upstream from flow disturbances that could alter the ammonia concentration, then the presence of a stratified concentration distribution in the stack shall be assessed using a pollutant (not diluent) gas by applying the procedure described in Chapter X, Section 13 of the SCAQMD Source Test Manual. For stratified concentration distributions in the exhaust stack, traverse sampling is required per paragraph 2.6 of SCAQMD Method 100.1. Optionally, the tester may presume stratified conditions and conduct traverse sampling without performing the Chapter X, Section 13 procedure. If stratified conditions are absent, the sampling point should be either near the centroid of the stack's cross section, or at a point no closer than 1.00 m (3.3 ft) to the stack's inner wall.

Sampling should begin and end only when the process has been operating for a sufficient length of time and steady operations can be assured. A steady state is defined as operating at constant operating temperature, feed rate, fuel rate, product application, or throughput rate, etc., and that the production rate is steady throughout the process. For batch or cyclic processes, the sampling period must encompass at least one complete cycle or batch. The sampling period must also begin and end at the same point in the cycle so that portions of the cycle are not over or under-represented.

3.1.3 Field Train Assembly

Connect the nozzle, probe, and filter to the first impinger as shown in Figure 207.1-1. Orient the nozzle so that the opening will be pointing downstream (directed away from on-coming flow). If traverse sampling is necessary, mark the probe to indicate the sampling location within the stack. Ensure that the probe and filter system (if applicable) are properly installed and operational. Probe assemblies with in-stack filters may be connected to the first impinger of the ammonia train using polyethylene tubing. Connect the pump and meter to the outlet of the fourth impinger on the ammonia train. Visually inspect the impingers and other connecting glassware for damage which may have occurred during transport. Pour water in the impinger tray to a height above the impinger solution level. Place crushed ice or dry ice pellets around the impingers. If a determination for ammonia mass emissions is required, assemble the velocity measurement system per SCAQMD Methods 2.1 or 2.3, and prepare the gas sampling equipment per SCAQMD Methods 10.1 or 100.1.

3.1.4 Pre-Test Leak Check

Leak check the train at the sampling site by plugging the nozzle and drawing a 380 mm (15 in.) Hg vacuum. A lesser vacuum may be used if it is not exceeded during the test. The probe may

be leak checked separately at a pressure equal to the stack pressure minus 25 mm (1 in.) Hg. A leakage rate in excess of either 4 percent of the average sampling rate or 0.00057 m³/min (0.02 cfm), is unacceptable.

Start the pump with the bypass valve fully open and the coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause the impinger solution to back up into the probe. If the desired vacuum is exceeded, either leak check at this higher vacuum or end the leak check as shown below and start over. When the leak check is completed, slowly remove the plug from the inlet to the probe, and then turn off the vacuum pump. This prevents the impinger solution from being forced backward into the filter holder, and silica gel from being entrained into the third impinger.

Perform a leak check of the Pitot lines in accordance with District Method 2.1, Section 2.2.1, if exhaust flow velocity will be measured.

3.1.5 Sampling Operation

For each run, record the required data on the data sheet shown in Figure 207.1-3. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment (no less than 10 minute intervals), when changes in flow rates are made, before and after each leak check, and when sampling is halted. Record other data required by the sheet in Figure 207.1-3 at least once for each sample point during each time increment. Note that data entry under the Probe and Filter Temperature data columns in Figure 207.1-3 is not required, nor is a measurement of the nozzle diameter. The Stack Velocity Heads and Temperatures shall be recorded per Method 1.1 if a determination of ammonia mass

emissions is desired. The sampling rate shall be between 0.015 m³/min (0.50 cfm) and 0.030 m³/min (1.0 cfm) during the run.

Clean the portholes prior to the test run to minimize the chance of contamination. To begin sampling, remove the nozzle cap and verify that the Pitot tube and probe are properly positioned. When the stack is under significant negative pressure (height of absorbing solution in impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent the impinger solution from backing into the probe. If necessary, the pump may be turned on with the coarse adjust valve closed. When the probe is in position, block off the openings around the probe and porthole to prevent flow disturbance and dilution of the gas stream.

For tests which require a mass emission determination, prepare the fixed gas sampling system (SCAQMD Methods 10.1 or 100.1) and collect stack gas concurrently with the ammonia train. Utilize the Flue Factor Method in accordance with Section 2, Chapter X of the SCAQMD Source Test Manual, or optionally perform traverse sampling with simultaneous velocity measurements.

Traverse the stack cross section if stratification is present or suspected. Be careful to avoid bumping the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes. This minimizes the chance of extracting stack deposits.

During the test run, periodically add ice around the outside of the impingers. Also, periodically check the level and zero of the manometer. Note and investigate any changes in stack temperature or velocity pressure over those measured during previous tests or traverses. Changes can mean failure of sampling equipment or a change in process.

If the pressure drop of the filter becomes too high, making sampling difficult to maintain, the filter may be replaced during a sample run. Use another complete filter assembly rather than attempting to change the filter itself.

If a component change (e.g. filter assembly or impinger) becomes necessary during the sampling run, conduct a leak check immediately before the change is made. Use the pretest leak check procedure, but use a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is not greater than either 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate, the results are acceptable and no correction has to be applied to the total volume of dry gas metered. However, if the leakage rate exceeds either of these limits, the tester must either record the leakage rate and correct the sample volume as shown in Chapter X, Section 7, or void the sampling run immediately after component change.

At the end of the sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post test leak check, as outlined in 3.1.6. Also, leak check Pitot lines, if used, as described in Method 2.1. The lines must pass this leak check to validate the velocity head data.

3.1.6 Post-Test Procedures

A leak check is mandatory at the conclusion of each sampling run. Allow the probe to cool. When the probe can be safely handled, follow the procedures outlined in Section 3.1.4 at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is not greater than either 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate, the results are acceptable and no correction has to be applied to the total volume

of dry gas metered. However, if the leakage rate exceeds either of these limits, the tester must void the sampling run, or adjust the sample volume as discussed in Chapter X, Section 7.

Wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent contamination. Do not cap off the probe tip tightly while the sampling train is cooling down. This would create a vacuum in the probe, drawing absorbing solution from the impingers into the probe.

Before moving the sample train to the clean-up site, remove the probe from the sample train and cap the open outlets of the probe. Be careful not to lose any condensate that might be present. Cap the inlet to the impinger train. Remove the connecting tubing after the last impinger and cap the last impinger. If a flexible line is used between the probe and first impinger, disconnect the flexible line and gently allow any condensed water or liquid drain into the impingers. Cap off the open inlet and outlet of the flexible line and impinger train openings. Either ground glass stoppers, plastic caps, or Parafilm laboratory film (or equivalent) may be used to close these openings.

Transfer the probe and impinger assembly to the clean-up area. This area should be clean and protected from the wind to reduce chances of contaminating or losing the sample. It is strongly recommended that sample recovery be performed in a controlled laboratory environment.

3.2 Mini-Impinger Train

3.2.1 Calibrations

Follow the calibration procedures and maintenance schedule in Chapter III of the SCAQMD Source Test Manual. Calibration results shall be submitted with the final test report.

3.2.2 Pre-Test Determinations

For non-stack sampling or area source testing, issues such as stratification or steady state operations shall be addressed on a case-by-case basis. It is strongly recommended that a protocol be submitted for the District's written approval. For example, compost pile testing should address parameters such as the age of the pile tested, the sampling locations of the flux chamber with respect to the height of the pile, diurnal changes in the emission profile, emission calculation procedures, and other considerations which may affect the representation of the source.

3.2.3 Field Train Assembly

Attach tubing from the connection point of the flux chamber (or other sampling source) to the first impinger. Attach the pump and metering system as shown in Figure 207-2. If a rotameter is used, make sure that the rotameter is vertically upright. Visually inspect the impingers and other connecting glassware for damage which may have occurred during transport. Place crushed ice or dry ice pellets around the impingers. Pour water in the impinger tray to a height above the impinger solution level.

3.2.4 Pre-Test Leak Check

With the impinger train fully assembled, set the pump to the desired sampling rate. Plug the air flow to the impinger train and observe the meter reading. The leak check is considered successful if the meter indicates a flow rate less than 4 percent of the average sampling rate.

3.2.5 Sampling Operation

Set the pump to the desired sampling rate, and record the starting time. Periodically inspect and adjust the sampling rate to assure constant sampling. Also check the amount of ice to maintain

the impinger bath temperature. Should it become necessary to change components during sampling, leak check the train per paragraph 3.2.4 prior to, and after changing the components. Record the leak check results and the replaced component on field data sheets.

3.2.6 Post-Test Procedures

Following sampling, perform a post-test leak check per Paragraph 3.2.4, and record the results. Disconnect the train assembly, and cap or clamp the tubing at the impinger inlet and outlet to prevent contamination.

METHOD 207.1

DETERMINATION OF AMMONIA EMISSIONS FROM STATIONARY SOURCES

Section 4 of 5

4.0 Laboratory Procedures

4.1 Sample Recovery

The sample shall be recovered within 7 calendar days, and must be performed in an area protected from the wind, and free from dust and ammonia. The procedures for recovering the Greenburg-Smith and mini-impinger trains are described in separate sections below.

4.1.1 Greenburg-Smith Impinger Train

Inspect the train for general condition. Note if the silica gel is completely expended, and whether the train and its components are sealed. Observe if the impinger contents are clear and odorless, and record any other condition that may be reflected in the analysis. Carefully disconnect the probe, filter holder and the impingers. Remove the in-stack filter, and record its condition. Place and seal the filter in a Petri dish, and label the container to clearly identify it. Weigh each impinger and its contents to the nearest 0.1 gram, and record these weights. Check and record the pH of each impinger solution; if any of the impinger solutions have a pH greater than 2, then invalidate the test results.

Transfer the impinger catch to a 500 ml graduated cylinder (or a 1000 ml cylinder, if necessary). Rinse the inside of the probe, the impingers (except for the silica gel impinger), and all connecting glassware into the graduated cylinder with deionized (DI) water. Repeat the rinse at least two additional times, making sure to contact all interior surfaces with each rinse. Dilute the sample to 500 ml with DI water (or 1000 ml, if necessary). Fasten a stopper on the cylinder and

mix the contents well. Transfer the sample to a container and seal it securely. Label the container to clearly identify it.

Take 200 ml of the reagent blank (see Section 2.3.4), and dilute it to the sample volume with DI water. Mix the solution well, then transfer the blank to a container. Securely seal the container, and label it as a method blank.

Following the recovery, store the samples and method blank in a refrigerator for later analysis. The refrigerator temperature shall be maintained between 35°F and 45°F.

4.1.2 Mini-Impinger Train

Inspect the train for general condition. Note if the silica gel is completely expended, and if the train and its components are sealed. Observe if the impinger contents are clear and odorless, and record any other condition that may be reflected in the analysis. Carefully disconnect the probe and the impingers. Weigh each impinger plus contents to the nearest 0.1 gram and record these weights. Check and record the pH of each impinger solution; if any of the impinger solutions have a pH greater than 2, then invalidate the test results.

Transfer the impinger catch to a 100 ml graduated cylinder (or a 200 ml cylinder, if necessary). Rinse the inside of the probe, the impingers (except for the silica gel impinger), and all connecting glassware into the graduated cylinder with deionized water. Repeat the rinse at least two additional times, making sure to contact all interior surfaces with each rinse. Dilute the sample to 75 ml with DI water. Fasten a stopper on the cylinder and mix the contents well. Transfer the sample to a container and seal it securely. Label the container to clearly identify it.

Take 30 ml of the reagent blank (see Section 2.3.4), and dilute it to the sample volume with DI water. Mix the solution well, then transfer the blank to a container. Securely seal the container, and label it as a method blank.

Following recovery, store the samples and method blank in a refrigerator for later analysis. The refrigerator temperature shall be maintained between 35°F and 45°F.

4.2 Analysis

4.2.1 Equipment

- a. Electrometer (pH meter) with expanded millivolt scale capable of 0.1 mV resolution between -700 mV to +700 mV.
- b. Orion Ammonia Electrode (electrode), Model 95-12 or equivalent. Cat. No.951201, 9512NB or equivalent.
- c. Graduated cylinder, various sizes as needed.
- d. Beakers, 150 ml.
- e. Volumetric Pipettes, Class A borosilicate glass, various sizes as needed.
- f. Volumetric Flasks, Class A, various sizes as needed.
- g. Balance, analytical, accurate to 0.1 g.
- h. Magnetic stirrer, thermally insulated, and Teflon coated stirring bar.
- i. Micropipette, 0.1 ml to 1 ml.

4.2.2 Reagents

- a. Ionic Strength Adjustor (ISA), ammonia pH adjusting solution (Orion catalog # 951211, or equivalent).
- b. Ammonia standard solution, 0.1M NH₄Cl or 1400 mg/L as N (Orion catalog # 951006, or equivalent).

- c. Deionized (DI) or distilled water to conform to ASTM specifications D-1193-99, Type 3. Water shall be blank-checked for ammonium ion.
- d. Ammonia as Nitrogen standard, 1000 mg/L (Orion catalog #951007, or equivalent).
- e. Calibration Verification solution. Pipette 10 ml of 0.1M NH₄Cl standard solution (Orion catalog #951006, or equivalent) into a 500 ml volumetric flask and dilute to the mark with 0.04 N H₂SO₄. The Calibration Verification concentration is 0.002 M NH₄Cl, or 28 mg NH₃-N/L. For highly concentrated or dilute ammonia samples, the Calibration Verification solution may be adjusted accordingly to reflect the concentrations being analyzed. The Calibration Verification solution must be prepared prior to each analysis.

4.2.3 Calibration

A calibration must be performed on the same date as the analysis. Prepare initial calibration standards using the 1000 mg/L Ammonia as Nitrogen standard. Prepare at least four standards spanning the desired calibration range. For each calibration standard, transfer the stock solution using a pipette directly into a 1-liter volumetric flask, add 400 ml 0.1 N H₂SO₄, and bring to volume using deionized water. See Section 4.3.1 to calculate the concentration of each calibration standard. The calibration levels presented below was found to be suitable for most sources tested in the South Coast basin.

Calibration Level	Volume of stock solution in 1 liter (V1), ml
1	1
2	5
3	20
4	100

The calibration standards may be stored in a refrigerator for a maximum of 4 weeks, until required.

Place 100 ml of each standard solution into clean 150 ml beakers. Immerse the electrode into the standard of lowest concentration, and add 2 ml of ISA while mixing. The solution must turn and remain blue, indicating an alkaline mixture. Keep the electrode in the solution until a stable reading is attained. Record the solution color, solution temperature, and electrode reading. The procedures in Section 4.2.4 shall be applied to insure accurate electrode measurements.

Repeat the procedure with the remaining standards, progressing from lowest to highest concentration. Calculate the concentration of ammonia (mg NH₃-N/L) in the standards per Section 4.3.1. Plot the logarithm of the concentration of ammonia (mg NH₃-N/L) versus the electrode potential developed in the standard, and determine the calibration regression line per Section 4.3.2.

4.2.4 Analytical Procedure

The electrode is sensitive to measurement conditions, and should be carefully handled and maintained to prevent spurious readings. The electrode must be rinsed with DI water and blotted dry between measurements. To prevent air entrapment on the membrane surface, use an electrode holder that secures the electrode at a 20 degree angle from vertical. If bubbles appear on the sensing membrane, tap the electrode gently to remove them. Samples and standards should be stirred at a constant rate using a magnetic stirrer. For electrode storage, refer to manufacturer's instructions.

When taking measurements, use beakers that minimize the ratio of surface area to volume. Keep beakers containing standards and samples covered between readings.

4.2.4.1 Greenburg-Smith Impinger Train

Remove the samples and standards from the refrigerator, and allow them to equilibrate to room temperature. Record the temperature of the room. All analyses must be performed at equivalent ambient conditions.

Perform the analyses in the following order, which constitutes one batch:

- 1) Prepare calibration standards according to Section 4.2.3, and establish the calibration curve. Fit the calibration data to a linear regression per Section 4.3.2.
- 2) Prepare a matrix spike by transferring 100 ml of sample to a clean 150 ml beaker, and add 2 ml of the 1000 mg/L Ammonia as Nitrogen Standard. Prepare a second identical solution for a duplicate analysis.
- 3) Prepare each sample, DI water blank, and a Calibration Verification Standard by transferring 100 ml of each solution to separate, clean 150 ml beakers. Prepare a second identical set of solutions for duplicate analyses.
- 4) Perform analyses of the duplicate blanks, samples, Calibration Verification Standards, and matrix spike solutions beginning with the solution having the lowest expected concentration, followed by analyses of progressively more concentrated solutions. Immerse the electrode into each solution and add 2 ml ISA while mixing. The solution must turn blue and remain blue to indicate it is alkaline. Keep the electrode in the solution until a stable reading is obtained. Record the solution color, the stabilized potential, and the temperature of the sample solution.
- 5) An analysis of the duplicate Calibration Verification Standards are to follow every fifth duplicate sample to confirm the calibration curve is adequate.

- 6) Analyze no more than ten duplicate samples, one matrix spike (and one matrix spike duplicate) in a batch. Analyze the duplicate Calibration Verification Standards at the end of the batch. Refer to Section 4.4 for Quality Control requirements.
- 7) Convert all the potential readings to ammonia concentrations (as mg NH₃-N/L) using the calibration curve per Section 4.3.3.

4.2.4.2 Mini-Impinger Train

Remove the samples and standards from the refrigerator, and allow them to equilibrate to room temperature. Record the temperature of the room. All analyses must be performed at equivalent ambient conditions.

Perform the analyses in the following order, which constitutes one batch:

- 1) Prepare calibration standards according to Section 4.2.3, and establish the calibration curve. Fit the calibration data to a linear regression per Section 4.3.2.
- 2) Prepare each sample, DI water blank, and a Calibration Verification Standard by transferring 30 ml of each solution to separate, clean 50 ml beakers. Prepare a second identical set of solutions for duplicate analyses.
- 3) Perform analyses of the duplicate blanks, samples, and Calibration Verification Standard solutions beginning with the solution having the lowest expected concentration. Immerse the electrode into each solution and add 2 ml ISA while mixing. The solution must turn blue and remain blue to indicate it is alkaline. Keep the electrode in the solution until a stable reading is obtained. Record the solution color, the stabilized potential, and the temperature of the sample solution.

- 4) An analysis of the duplicate Calibration Verification Standards are to follow every fifth duplicate sample to confirm the calibration curve is adequate.
- 5) Analyze no more than ten duplicate samples in a batch. Analyze the duplicate Calibration Verification Standards at the end of the batch. Refer to Section 4.4 for Quality Control requirements.
- 6) Convert all the potential readings to ammonia concentrations (as mg NH₃-N/L) using the calibration curve per Section 4.3.3.

4.2.4.3 Sample Dilutions

If the sample is not within the calibration range, the sample must be diluted. Estimate the solution concentration by diluting a sample aliquot with 0.04 N H₂SO₄. Note that QC procedures are not necessary, since the estimation is qualitative. Based on the estimated concentration, determine the appropriate dilution that would yield a concentration in the middle of the calibration range. Prepare the sample with the appropriate dilution, and perform the analysis as specified in Sections 4.2.4.1 or 4.2.4.2, with all applicable QC.

4.3 Calculations

4.3.1 Calibration Standards

$$\text{mg NH}_3\text{-N/L} = (V1 / 1000) * S1$$

where:

S1: Concentration of the Ammonia as Nitrogen standard (refer to Section 4.2.3), 1000 mg/L Ammonia as Nitrogen.

V1: volume of the stock solution (ml) to make 1 liter calibration standard (refer to Section 4.2.3).

1000: 1-liter solution (1000 ml).

4.3.2 Calibration Regression Line

Determine a standard regression line of calibration standards versus electrode potential (P) in millivolts (determined in Section 4.2.3) using the relation below. Calculate the regression parameters using the calculation sheet shown on Figure 207.1-4.

$$P = M * \log (\text{mg of NH}_3\text{-N/L}) + B$$

where:

M = slope

B = intercept

4.3.3 Measured Concentration Of Ammonia

Calculate the measured concentration (C) of ammonia (mg of NH₃-N/L) from the electrode potentials recorded in Sections 4.2.4.1 or 4.2.4.2:

$$C = 10^{(P-B)/M}$$

4.3.4 Average Measured Ammonia Concentration (C_{avg})

$$C_{\text{avg}} = (C_1 + C_2) / 2$$

where:

C₁, C₂ = results from duplicate analyses (mg NH₃-N/L)

4.3.5 Relative Percent Difference (RPD)

The RPD is determined from duplicate analyses as follows:

$$\text{RPD} = (C_1 - C_2) / C_{\text{avg}} * 100$$

4.3.6 Relative Percent Accuracy (RPA)

Determine the RPA of the calculated concentration for Calibration Verification:

$$\text{RPA} = (\text{C}_{\text{avg-28}} / 28) * 100$$

where

28 = theoretical value of the check standard (mg NH₃-N/L) (see Section 4.2.2(e))

4.3.7 Matrix Spike Percent Recovery (%R)

%R is calculated as follows:

$$\%R = (\text{C}_{\text{spike}} * 0.104 - \text{C}_{\text{sample}} * 0.102) / 2.0 * 100$$

where:

C_{spike} = average result of matrix spike analysis (mg NH₃-N/L)

C_{sample} = average result from duplicate analyses of unspiked sample (mg NH₃-N/L)

4.4 Quality Control

4.4.1 Initial Calibration

Instrument performance parameters (slope and intercept) must be determined and demonstrated through regression analysis to be acceptable for this method. If any major changes are made to the system, recalibrate the instrument to verify these parameters and demonstrate the instrument's acceptability. Determine the slope (M) and intercept (B) if they have not been previously measured. Track instrument performance against previous test batches by control charting the slope values.

The slope of the calibration curve (M) shall be in the range of -54 to -60. In addition, the coefficient of determination (r²) of the calibration curve must be 0.997 or greater (see Figure 207.1-4 for the calculation of r²). If both these linearity criteria are not satisfied, the calibration

is unacceptable. To remedy this, the analyst may elect to check the electrode, prepare fresh standards, troubleshoot the instrument, or perform routine instrument maintenance prior to recalibrating the instrument.

4.4.2 Calibration Verification

The relative percent accuracy (RPA) of the Calibration Verification must be 10% or less. If the initial Calibration Verification check does not satisfy this criteria, prepare a new Calibration Verification standard and analyze it. If this standard also fails to meet the control limit, troubleshoot the instrument and perform another Calibration Verification check. If this third Calibration Verification fails, discard the initial calibration and determine new instrument performance parameters.

4.4.3 Measurement Stability

Due to the instrument's sensitivity to solution temperature, the calibration solutions, sample solutions, and Calibration Verification standard temperatures must all be within $\pm 2^{\circ}\text{C}$ of one another. All solution temperatures must be submitted with the laboratory report.

The relative percent difference (RPD) of duplicate measurements of the calibration output and sample results must be 5% or less. If the RPD exceeds this criteria, troubleshoot the instrument and prepare fresh standards.

4.4.4 Matrix Spike Recovery

An evaluation of the Matrix Spike Recovery (%R) is performed on a qualitative basis only, and is intended to provide a symptomatic overview of the analysis. Although there are no criteria for acceptability of %R, the data is useful for identifying possible errors in the analysis and must be submitted with the test report.

4.4.5 Temperature Probe

At three month intervals, the probe measuring the solution temperature shall be checked at two conditions: in chilled ice water (approximately 32°F), and in water at room temperature (approximately 75°F). Measurements shall be made simultaneously with a reference temperature sensing instrument. The reference instrument shall be calibrated per the Chapter III procedures of the SCAQMD Source Test Manual. The difference between the temperature probe and reference instrument shall agree within 1.5 percent of the absolute reference temperature (degrees Rankine).

METHOD 207.1

DETERMINATION OF AMMONIA EMISSIONS FROM STATIONARY SOURCES

Section 5 of 5

5.0 Emissions Reporting

5.1 Engineering Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation.

Calculate the mass of recovered ammonia in the sample trains as follows:

Greenburg-Smith Impinger Train Results

$$\text{Net Sample Collection (mg)} = C_{\text{avg}} * 17.03 / 14.01 * 0.500$$

where:

C_{avg} = average result of duplicate laboratory analyses (mg NH₃-N/L), per Section 4.3.4;

17.03 = MW of ammonia;

14.01 = MW of nitrogen; and,

0.500 = liters of recovered solution, per Section 4.1.1.

Mini-Impinger Train Results

$$\text{Net Sample Collection (mg)} = C_{\text{avg}} * 17.03 / 14.01 * 0.075$$

where:

C_{avg} = average result of duplicate laboratory analyses (mg NH₃-N/L), per Section 4.3.4;

17.03 = MW of ammonia;

14.01 = MW of nitrogen; and,

0.075 = liters of recovered solution, per Section 4.1.2.

To calculate the ammonia concentration and mass emissions at the stack, a calculation sheet similar to that shown in Figure 207.1-5 may be used. Other forms of the equations may be used as long as they give equivalent results.

Results of each duplicate train must be reported separately; the highest valid emission result shall be used for compliance purposes.

5.2 Report Information

The following information is to be included in the test report:

1. All printouts (as applicable), must be included in the final report and must be clearly identified as to:

-location/source	-range changes
-operator initials	-range of measurement
-date/running times	-calibrations
-actual test interval	-cal gas concentration/cyl. no.
-contaminant/diluent	-range of calibration
2. A summary of the Source Test results.
3. A brief process description. Indicate equipment and process operation during testing, as well as any other information which may influence the final report.
4. A simple schematic diagram of the process, showing the sampling location, with respect to the upstream and downstream flow disturbances.
5. The sampling and analytical procedures. Be specific about all aspects of sampling and analysis. Include diagrams of test equipment and methods.

6. Complete raw field data, including production and all operating data indicative of the testing interval, analyses, and the test results (show all calculations).
7. Complete laboratory and analytical data, including all calibration and QA/QC data. If spreadsheets are used, an annotated sample calculation shall be provided.
8. Calibration data regarding all sampling and measuring equipment utilized during testing (see SCAQMD Source Testing Manual, Chapter III).

5.3 Report Format

Each page in the test report must be identified by a sequential page number which can be uniquely referenced. The information in the final report shall be formatted as follows:

- I. Table of Contents
- II. Executive Summary. This section shall present tabulated results from all tested devices in the units of measurement referenced by the Permit or applicable Rule. Additionally, the compliance limits shall be tabulated in a column adjacent to the measured emission.
- III. Introduction. Provide a brief opening statement identifying the Facility I.D., the equipment A/N, P/O, or Device I.D. and the reason(s) for testing (applicable rules permit conditions, etc.). Also identify the test dates, the personnel on hand for the test, names, titles and phone numbers of responsible test firm and facility personnel.
- IV. Equipment Description Including Fuel Meter(s), if applicable. This section shall include a statement that verifies acceptability of the method test location and the

operating condition during the test. Provide a simple schematic diagram of the process, showing the sampling location, with respect to the upstream and downstream flow disturbances, as well as the control equipment. The diagram must show all major pieces of equipment, and whether flow is combined from other sources, or diverted to other locations. A summary of the process and operating parameters (such as the ammonia injection rate) during testing shall also be included.

V. Test Critique. Submit a “self-critique” of anything that transpired during the test which is useful in the interpretation of the test results.

VI. List of Sampling and Analytical Methods Used. This section shall include a list of the test methods used. Do not include copies or descriptions of the source test methods if the methods were adhered to as written. If exceptions were made to the methods, submit only an explanation of the exceptions.

VII. Appendices

A. SCAQMD Method Results

B. Schematics of Stack Sampling Locations

C. Field Data Sheets - testing method and process data sheets shall be compiled in separate sections

D. QA/QC - include all calibration data and QC/QC results required by the field sampling procedures. Data shall be submitted demonstrating that field equipment and instruments used during the test meet the Chapter III requirements of the SCAQMD Source Testing Manual.

- E. Laboratory Analytical Results, including all raw data including (but not limited to) solution temperatures, annotated calculations, and analytical QA/QC data.
- F. Calibration Data and Calculations
- G. Calibration Gas Certificates
- H. Chain of Custody Information (as necessary)
- I. Process Operating Data Including Fuel Usage During Test
- J. Calculations Using Fuel F-Factor (if necessary)
- K. Copy of SCAQMD Permit/ Application
- L. Signed “Statement of Non-Conflict as an Independent Laboratory” per District Rule 304(k)

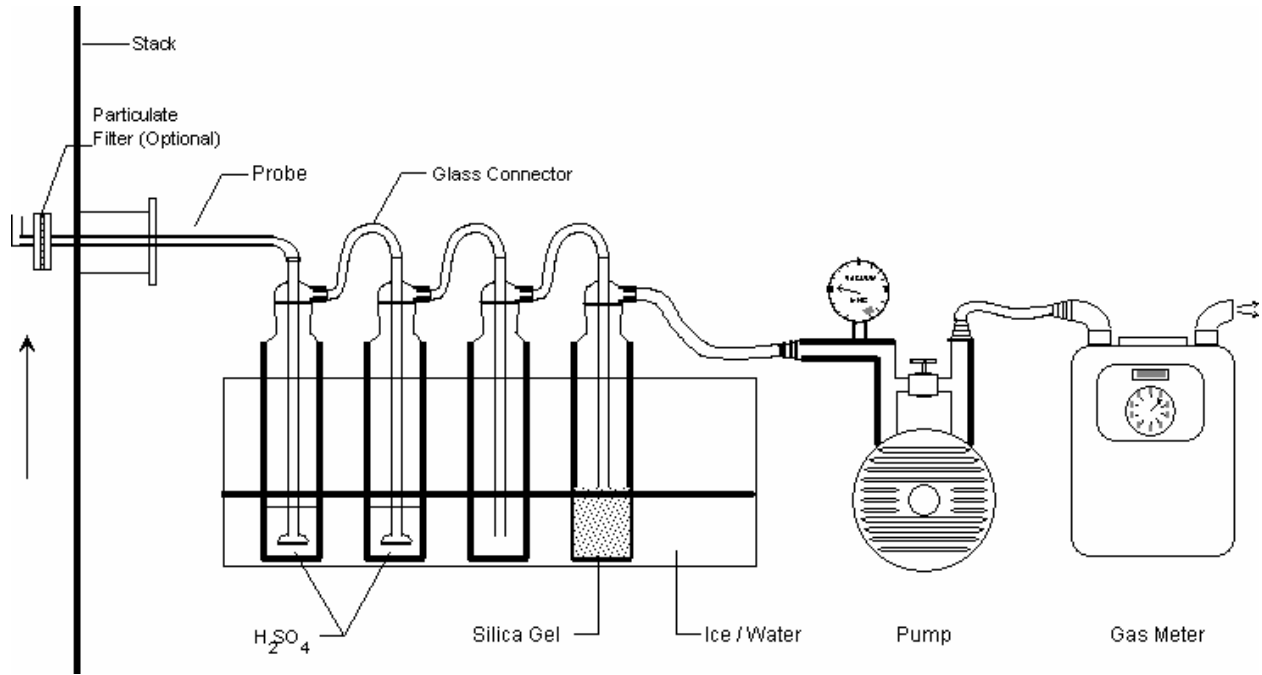


Figure 207.1-1
Ammonia Train With Greenburg-Smith Impingers

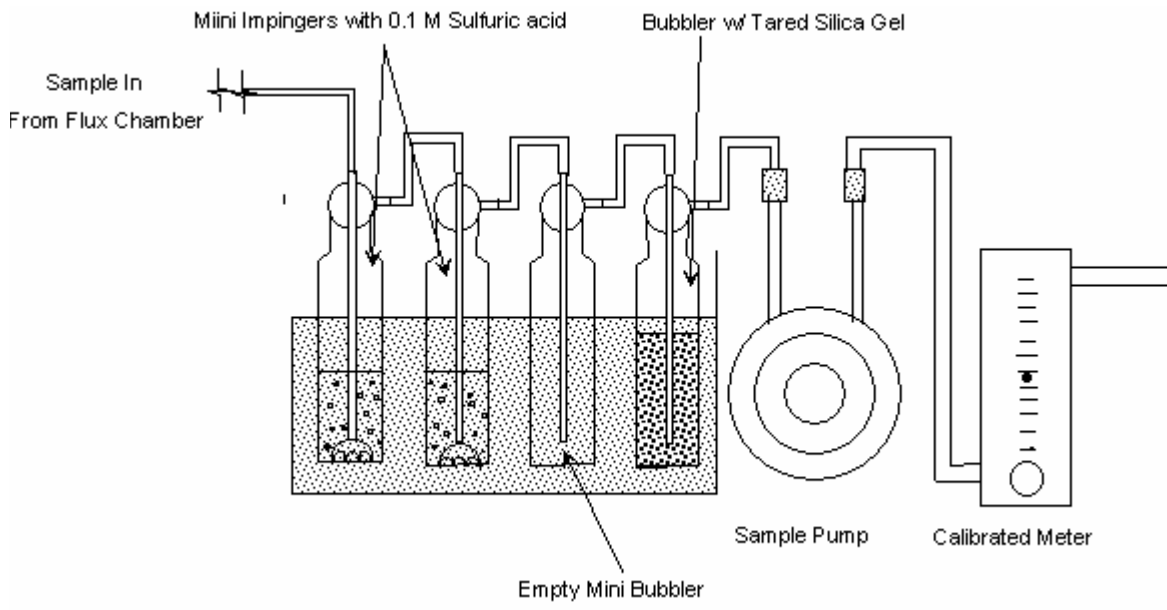


Figure 207.1-2
Ammonia Train With Mini-Impingers

SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT
REGRESSION CALCULATIONS

Laboratory Ref. No.: _____

Analysis Date: _____

Facility Tested: _____

Analyst: _____

Calib. Level	Calib. Std. mg NH ₃ -N/L	(X) log ₁₀ (Calib. Std.)	(Y) Elect. Pot. (mv)	(X ²)	(Y ²)	(XY)
1	1	_____	_____	_____	_____	_____
2	5	_____	_____	_____	_____	_____
3	20	_____	_____	_____	_____	_____
4	100	_____	_____	_____	_____	_____
Sum		_____	_____	_____	_____	_____
		(S _x)	(S _y)	(S _{x2})	(S _{y2})	(S _{xy})
Average		_____	_____	_____	_____	_____
		(A _x = S _x / 4)	(A _y = S _y / 4)	(A _{x2} = S _{x2} / 4)	(A _{y2} = S _{y2} / 4)	

$$SD_x = \sqrt{[Ax2 - (Ax)^2] \frac{4}{3}} = \underline{\hspace{2cm}}$$

$$SD_y = \sqrt{[Ay2 - (Ay)^2] \frac{4}{3}} = \underline{\hspace{2cm}}$$

$$\text{Slope} = M = \frac{Sxy - 4(Ax)(Ay)}{Sx2 - 4(Ax)^2} = \underline{\hspace{2cm}}$$

$$\text{Intercept} = B = Ay - M(Ax) = \underline{\hspace{2cm}}$$

$$\text{Coeff. of Determination} = r^2 = \left(M \frac{SDx}{SDy} \right)^2 = \underline{\hspace{2cm}}$$

Figure 207.1-4
Regression Calculation Sheet

Appendix A

Protocol Preparations

COMPANY INFORMATION

LEGAL NAME AND FULL ADDRESS of the test facility.

Name: _____

Address: _____

City: _____ State: _____ Zip: _____

Phone No. _____ Fax No. _____

LEGAL NAME AND FULL ADDRESS of the subcontracted laboratory.

Laboratory Name: _____

Address: _____

City: _____ State: _____ Zip: _____

Phone No. _____ Fax No. _____

Personnel performing the test

TABLE 1

Individual's Name and Degree	Position or Title	Duties

Protocol preparation checklist

Process Information

- Detailed process description
- Process flow diagram
- Normal operating conditions, and normal operating parameters
- Process variability (cyclic, batch, steady state, intermittent)
- Nature of emissions
- Approximate temperature, pressure, velocity and profile, moisture content, stratification and other relevant characteristics of the emission source
- Effects of operating conditions on emissions
- Description of process monitoring equipment if any

Purpose of the test

- Applicable Federal, State, and/or District rule(s), quoted
- Applicable permit condition(s), annual test requirement(s), ERCs, BACT, variance, and other applicable requirements, quoted
- Description of engineering study
- Description of other purpose
- Estimate of the threshold values of pollutants of interest

Representativeness of data and process

- Proposed operating conditions, including conditions under which testing will be temporarily halted or aborted
- Dimensioned diagram of duct/stack and all vanes, dampers, connecting ducts etc.
- Location, description and accessibility of ports
- Proposed sampling points
- Check for cyclonic flow and/ or stratification
- Description of methods by reference to applicable source test methods
- Sampling equipment diagrams
- Detailed description of **any method modifications** required, and purpose
- Proposed sampling times and rates and their relationship to process variability (batch, cyclic, intermittent or steady state)
- Number of sampling runs
- Compliance with Rule 217
- Available power supply

Quality Assurance and Data Integrity

- Validation of equipment calibrations
- Proposed blanks, spikes, in-field calibrations

Calculations and reporting

- Proposed calculations and data handling
- Units of emissions and name of reported compound(s) (example NO_x as NO₂)
- Other proposed data that is to be reported, such as process parameters
- Raw laboratory analytical data, including calibration curve, blanks, and standards
- Laboratory (analytical) calculations, with annotations to facilitate review

Format

- Format recommended by SCAQMD

Appendix B

Pre-test calculations

Source Target Concentrations (STC)

The tester should not proceed with the test unless a target concentration has been chosen. A basis shall be selected for determining each target concentration from: (a) regulatory limits, (b) environmental risk assessments, and (c) the interests of SCAQMD, the tester, and the stationary source.

(1) Regulatory Limits

The regulatory limit shall be the basis for determining a target concentration for stationary source emission in those cases where the purpose of the emissions test is to demonstrate compliance with the established regulatory limit.

(2) Environmental Risk Assessments

In some cases testing is conducted for an environmental risk assessment. A pre-test estimate of the permissible risk shall then be used to determine the target concentration for stationary source emissions.

(3) Interests of the SCAQMD, the Tester, and the Stationary Source

In cases where the emissions test is not being performed to demonstrate compliance with a regulation, the emission results from previous tests at the facility or from similar sources may be used to estimate the source target concentrations.

Determining Reporting Limit (RL)

To obtain the RL, assume that:

- (1) all the ammonia in the gas sampled is recovered from the liquid of the probe rinse and the first impinger; and,
- (2) the volume of the liquid of the probe rinse and the first three impingers is 500 ml for Greenburg-Smith impingers, or 75 ml for mini-impingers.

Equation 207-1 shall be used to calculate the RL:

$$RL = C_1 \times V_R \times (17.03 / 14.01) \quad 207-1$$

Where:

RL = analytical mass reporting limit, mg

C_1 = lowest concentration on a calibration curve. This calibration curve shall meet the QC requirements of Section 4.4.1, and must have been generated within six months prior to the anticipated test date, mg NH₃-N/L

V_R = assumed liquid volume of probe rinse and first impinger, ml
= 0.500 L for Greenburg-Smith impingers, or 0.075 L for mini-impingers.

17.03 = molecular weight of ammonia

14.01 = molecular weight of nitrogen

Planned Sampling Volume (PSV)

The planned sample volume is the volume of emissions that must be sampled to collect for analysis a quantifiable mass of ammonia. The PSV is based on the reporting limit and the source target concentration, and is further adjusted by a safety factor to assure quantification. Higher values of the safety factor provides greater assurance for quantification, and may depend on considerations such as process variability, historical data, and analytical capabilities. Equation 207-2 shall be used to calculate the PSV:

$$PSV = RL / STC \times F \qquad 207-2$$

Where:

PSV = planned sample volume, dscm

RL = analytical mass reporting limit, mg

STC = source target concentration, mg/dscm

F = safety factor, $F \geq 1$

Planned Sampling Time (PST)

The planned sampling time is the time required to collect the minimum sample volume at the proposed volumetric sampling rate (VSR). The tester should use an average volumetric sampling rate appropriate for the source to be tested; for Greenburg-Smith impingers, the sampling rate is generally between 0.5 and 0.75 cfm. For mini-impingers, the rate is generally between 0.75 lpm and 1.0 lpm. If the VSR cannot be achieved in the field, the sampling time shall be revised using the following equation to achieve the target PSV. The sampling time must be such that the emissions test is conducted during representative operation conditions of the source. Note for permit or rule requirements limiting emissions to a mass per hour basis, a one hour minimum sample shall be collected. Equation 207-3 shall be used to calculate the PST:

$$PST = PSV / VSR \qquad 207-3$$

Where:

PST = planned sampling time, hours

PSV = planned sample volume, dscm

VSR = achievable volumetric sampling rate, dscm/hour