New Jersey Department of Environmental Protection Air Quality Permitting Program Bureau of Technical Services

Technical Manual 1004

Guidelines for Compliance Stack Emission Test Programs

For technical questions on this manual, call: **Bureau of Technical Services at (609) 530-4041**

> E-mail: Where

<u>first.last@dep.state.nj.us</u> first - first name last - last name

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I. INTRODUCTION

The purpose of this manual is to provide general guidance on conducting stack tests at NJ facilities and the necessary interactions between the regulated community and/or their consultants with the Bureau of Technical Services. It is not intended to provide step-by-step instructions on preparing a stack testing protocol, observing or conducting emission tests, or preparing or reviewing emission test reports. It does contain references for specific test methods and related material.

Each protocol is source specific and will be evaluated on individual merit. Final approval will be based on the ability of the written protocol to satisfy all required testing parameters by means approved by the Bureau of Technical Services and having sound scientific basis.

Appendix II of this document contains Protocol Templates for some of the more commonly used test methods. These templates include check-off boxes and fill in the blank sections that allow one to tailor the Template to the source specific situation. You are encouraged to utilize these templates in preparing your protocol. Use of the Templates does not relieve the tester from knowing and understanding the complete method. Should a test method be revised where the current version of the method is in conflict with a Template in this manual, the revisions should be addressed in the "Proposed Deviations from this BTS Template or the Method " section of the applicable Template.

All testing shall be performed in accordance with the approved protocol. Unapproved deviation from the protocol is not acceptable and will be justification to require repetition of the test project. A NJ Certified laboratory must complete all analytical work for affected methods. For additional details, please refer to N.J.A.C. 7:18, "Regulations Governing the Certification of Laboratories and Environmental Measurements" which can be found at www.nj.gov/dep/oqa/labcert.html.

Laboratory audits for applicable methods may be provided, or may be required to be purchased by the facility. Some audits require field sampling prior to analysis. All required audits will be analyzed with the field samples and the results included in the test report.

Final test reports must contain sufficient raw data to accurately reproduce the test results. Failure to provide this data will be grounds for rejection.

It is the goal of the Bureau of Technical Services to move to a program of electronic submittals, in a consistent format, for protocols and test reports. One option currently available would be to use EPA's Electronic Reporting Tool (ERT). At some point, electronic submittals may be mandated.

II. APPLICABILITY

This guideline is intended for source operations required to perform compliance stack emission tests as a condition of Preconstruction Permit or Operating Permit approval, Permit renewal, Federal regulation, etc., including but not limited to:

- N.J.A.C. 7:27 and N.J.A.C. 7:26
- Title 40, CFR, Part 51
- Title 40, CFR, Part 60

- Title 40, CFR, Part 61
- Title 40, CFR, Part 62
- Title 40, CFR, Part 63
- Title 40, CFR, Part 264
- Title 40, CFR, Part 266

III. DEFINITIONS

Definitions of most terms are located in the appropriate test method, N.J.A.C. 7:27 Subchapter, NSPS Subpart, NESHAPS Subpart or referenced material.

BIF - Title 40, Code of Federal Regulations, Part 266; Standards for the Management of Specific Hazardous Wastes and Specific Types of Hazardous Waste Management Facilities.

BOP Permit – a Title V Operating Permit issued by the Air Quality Permitting Program.

BTS - NJDEP, Division of Air Quality, Air Quality Permitting Program, Bureau of Technical Services.

CEMS – Continuous Emission Monitoring System.

CFR – Code of Federal Regulations.

COMS - Continuous Opacity Monitoring System.

DAS – Data Acquisition System.

EPA (or USEPA) – United States Environmental Protection Agency.

MACT – Title 40, Code of Federal Regulations, Part 63: NESHAP for Source Categories, a.k.a. Maximum Achievable Control Technology.

N.J.A.C. 7:27 - Chapter 27, Bureau of Air Pollution Control adopted by the Department of Environmental Protection pursuant to authority delegated at N.J.S.A. 26:2C-1 et seq. and N.J.S.A. 26:2D-1 et seq. and was filed and became effective prior to September 1, 1969.

NESHAPS - Title 40, Code of Federal Regulations, Part 61: National Emission Standards for Hazardous Air Pollutants.

NSPS - Title 40, Code of Federal Regulations, Part 60; Standards of Performance for New Stationary Sources.

PCP – Preconstruction Permit issued by the Air Quality Permitting Program.

PPE – Personal Protective Equipment.

Stack Emission Test - A series of three valid sampling runs (per contaminant sample train), each of

which shall be a minimum sampling time of one hour, unless otherwise specified or approved by the Department.

IV. PROTOCOL DEVELOPMENT & APPROVAL

Each protocol shall be developed as a site-specific document. As a rule, generic protocols are not acceptable, although the use of Protocol Templates (Appendix II) is encouraged when applicable. The protocol shall include information about the source(s), site(s), test methods, sample procedures, sample recovery and analysis, production data, report preparation, quality control and quality assurance.

A. <u>SOURCE INFORMATION</u>

A stack emission test protocol shall contain the following source identification information:

- 1. Facility name as it appears on the Permit;
- 2. Facility address;
- 3. 5-digit Program Interest or APC Plant ID Number (if available);
- 4. Source designation;
- 5. PCP or BOP Permit Number (if applicable);
- 6. Facility test project coordinator's name and telephone number;
- 7. Brief description of the source operation;
- 8. Special safety requirements and/or PPE (special respirators, etc.) including any safety limitations on Department observers (i.e. no facial hair, etc.);
- 9. A list of air contaminants to be sampled, the emission allowables, and the test method(s) to be utilized; and
- 10. The name of the test consultant firm that will be completing the test, along with their address and phone number, and the test firm contact person (if available).
- 11. The name of the laboratory or laboratories that will be completing the analytical work (if available), including their NJ certification information for the applicable methods.

B. <u>SITE INFORMATION</u>

The protocol shall contain specific physical information concerning the sampling location(s). It is highly recommended that the test contractor perform a pre-sampling

investigation prior to protocol submittal. The minimum information required for each sampling location is:

- 1. Internal stack diameter If actual measurements are not available, estimates, based on blueprints, design specifications or other means will be acceptable; however, prior to sampling, measurements must be taken and provided to the BTS on-site representative(s).
- 2. The number and location of sampling ports a schematic indicating the number and location is required.
- 3. The upstream and downstream distances from the sampling ports to the nearest flow disturbances shall be included on the schematic.
- 4. The number and location of sampling points required.
- 5. The stack flow parameters, including but not limited to approximate stack temperature, moisture and air flow rates.

C. <u>METHODS INFORMATION</u>

The protocol shall include detailed information about the sampling/analytical methodology to be utilized for each air contaminant in the test plan. Justification for choosing a specific method should be included. (*Note:* Merely listing or including a photocopy of a written method is not an acceptable alternative to a detailed protocol). The test method must be tailored to the source specific application.

If an air contaminant is affected by more than one regulation (i.e. New Jersey Air Permit and a Federal Regulation such as NSPS or MACT), the methodology must be applicable to each regulation. It may be necessary to use more than one method. The method description should include:

- 1. The name and source of the proposed test method;
- 2. The approximate in-stack air contaminant concentration that represents the emission limit vs. the method detection limit;
- 3. A complete description of the sampling train, including materials of construction, impinger solutions and reagents;
- 4. Analyzer spans and approximate calibration gas values;
- 5. Calibration procedures for all pertinent sampling and on-site analytical equipment (unless otherwise specified by the method, the sampling and analysis systems shall be designed to determine the concentration of the parameter at the in-stack limits and be calibrated such that the in-stack Permit limits are bracketed by two calibration points);

- 6. Sampling procedures, including sample run times;
- 7. Sample recovery procedures, including holding time limits and sample preservation requirements prior to sample analysis;
- 8. Quality assurance/quality control measures; and
- 9. Any proposed deviation from the written method, including a complete justification on the necessity for the alteration. (*Note:* BTS cannot approve alterations to Federal Reference Methods required to demonstrate compliance with Federal regulations. If an individual proposes to determine compliance with a Federal regulation with a method other than specified by the EPA regulation, such request will be forwarded to EPA Region II for review).

D. PROCESS AND CONTROL PARAMETERS

The protocol shall indicate the type of information that will be monitored during stack emission testing. This information should reflect the conditions of the PCP or BOP Permit or applicable regulation. For example:

- Raw material weights or volumes;
- Any analysis of raw materials (sulfur in fuel, metals in sludge, etc.),
- Air pollution control equipment parameters (pressure drops, liquid flow rates, temperatures, etc.);
- Fuel usage rates;
- Production output (quantity, linear feet, weight, megawatts, etc.); and
- Any other pertinent production related information.

Testing must be conducted at worst-case permitted operating conditions with regard to meeting the applicable emission standards, unless otherwise specified in the PCP or BOP Permit or applicable regulation, but without creating an unsafe condition. Failure to do so could result in limits on future operation (a Permit Modification may be required by the facility) or a requirement to repeat the test program. The protocol **must** state the expected production level.

(*Note:* Any specific process/production or air pollution control equipment parameter specified in the PCP or BOP Permit conditions shall be monitored during the stack emission test program. This data shall be included in the final test report).

Production/process and air pollution control equipment data is to be reported following the format included in Appendix I. This format may be altered to reflect specific production/process and air pollution control equipment items for each source operation. Facility DAS or computer printouts may also serve as a substitute reporting format. **Data must be summarized for each test run or test period, as applicable.**

E. PROTOCOL REVIEW AND APPROVAL

BTS shall approve all air pollution control compliance protocols prior to scheduling stack emission tests.

- 1. General guidelines for protocol approval are:
 - a) A minimum of three valid runs;
 - b) A minimum sampling time of 60 minutes per run or a batch cycle, whichever is longer;
 - c) If an existing stack test method is available (NJDEP or USEPA), it shall take precedence over non-validated proposed methods.

Prior to allowing non-validated proposed methods, BTS may request that the individual perform a validation study in accordance with USEPA Reference Method 301.

- 2. The sequence in a BTS protocol review shall be as follows:
 - a) BTS reviews the Facility Specific PCP or BOP Permit requirements.
 - b) Review the conditions required for stack emission testing and continuous emission monitoring. If CEMS/COMS are required, separate protocols are required and the CEMS/COMS shall be installed and certified consistent with the Facility Specific requirements.
 - c) Determine any special conditions associated with an applicable Subchapter, Subpart or regulation.
 - d) Compare the application of the proposed method(s) in the protocol with the conditions of the Facility Specific requirements.
 - i) The method(s) proposed must be appropriate for the air contaminant and source, and comply with all regulations governing the test.
 - ii) The detection limit of the proposed method shall be capable of demonstrating compliance with the air emission limits indicated in the PCP or BOP Permit, or applicable air regulation.
 - iii) Any alteration to an existing stack emission test method shall not be considered by BTS unless the reason for the alteration is to provide a means of collecting valid data that could not otherwise be obtained with the method as written; or, the method as written cannot be performed for reasons other than convenience or expense.

- e) If the protocol is lacking information necessary to complete a review by BTS or if the method proposed is not appropriate, BTS will address these issues to the facility and its representatives in a written Notice of Deficiency (NOD). The NOD will indicate a time requirement for response. The time limit will be thirty days; however, BTS may extend or reduce the response time limit if the circumstances justify such action.
- f) When an individual's protocol contains sufficient information, an approval letter will be issued authorizing the scheduling of a mutually acceptable stack emission test date. When considering proposed testing dates, the individual should allow a minimum of 30 calendar days for scheduling.
- g) A testing date(s) may be reserved by contacting the Section Chief, Emission Measurement Section, or his/her designee. Having verbally established a testing date(s), the individual or agents thereof shall confirm their intent to test in writing, within two (2) working days of the verbal approval. Telefacsimile transmission or e-mail are acceptable written verification procedures. Failure to confirm may result in forfeiture of the schedule testing date(s).
- 3. Service fees will be invoiced to the facility consistent with N.J.A.C. 7:27-8.6 or N.J.A.C. 7:27-22.31.

V. CONDUCTING & OBSERVING COMPLIANCE STACK EMISSION TESTS

The stack test is not the time to troubleshoot the equipment. Once the process is stabilized and operating at the production level necessary for compliance testing and the testing team has completed any preliminary measurements and set-up (Items 1, 2 and 3 that follow), sampling shall begin. Adjusting or tuning the process based on real time emissions data not normally available to the equipment operators is prohibited.

The test project should begin within a reasonable time (within 2 hours) from the established scheduled start time. Failure to do so may result in a cancellation of official testing activities for the day at the discretion of the BTS observer and will require rescheduling of a test date(s). Observation service fees will still be assessed (see Item 6 to follow).

All test runs that are started should be completed unless there is a valid technical difficulty with the testing equipment (ie: failed leak check during a port change, when done) or for safety reasons. Only the Department may void a sample run for reasons other than technical difficulty with the test equipment or for safety reasons. If the facility representative feels the results from the particular run were affected by a process upset, an additional run may be conducted and an explanation must be included in the test report. Data from <u>all</u> completed test runs must be submitted, regardless of whether three runs are completed or additional runs are performed.

When conducting a stack emission test, an individual shall not deviate from their approved test protocol, unless such deviations are approved by BTS. The stack emission testing individual shall:

- 1. Check all source data including:
 - stack diameter;
 - upstream/downstream diameters;
 - stack temperature, pressure, moisture and air flow.

Prior to testing, it may be necessary to alter the number of traverse points, nozzle diameter, etc. based on these actual measurements. All changes must be given to the on-site BTS observer.

- 2. Provide the following information to the on-site BTS observer on the day of conducting the compliance stack emission tests:
 - a) The data from the pre-test cyclonic flow traverse.
 - b) Pre-test calibration data for all pertinent sampling equipment.
- 3. Verify with the responsible individual that the source is operating at a production rate consistent with Section IV.D. of this manual (or the rate agreed to in the protocol) prior to the start of sampling. (Note: At the discretion of the Regional Enforcement Office, any individual conducting compliance tests while operating at a production rate other than the level indicated above may be required to file for a Permit Modification to update the operating conditions to reflect the maximum process rate which was achieved during testing. If the Permit is conditioned for lower process rates, operating at a higher level must have prior approval, in writing, either through a Permit Modification or from the Regional Enforcement Office, or a violation of the Permit would occur, resulting in possible enforcement action against the facility. As part of the approval to return to a higher production rate, additional stack testing requirements may be imposed).
- 4. During sampling, a representative responsible for the source operation or a representative of the consultant testing firm is required to gather process, production and air pollution control equipment information. An example of the format requested for the stack test program is shown in Appendix I. This format may be altered to conform to the needs of the specific source (See Section IV, Item D).
- 5. Set up all equipment, conduct leak checks, sample and recover samples as specified in the approved protocol.
- 6. Service fees will be invoiced to the facility consistent with N.J.A.C. 7:27-8.6 or N.J.A.C. 7:27-22.31. Please note that observation fees will still be assessed for test days where the tests are cancelled or postponed while the BTS observer is or was on-site, **including instances where the BTS observer cancels tests for any reason**.

VI. SAFETY DURING TESTING

Stack sampling and source evaluation exposes DEP officials and consultants to potential safety hazards in the field. Ensuring the safety of all field personnel at facilities is an issue that the BTS takes very seriously.

To insure the safety of all field personnel, stack sampling platforms, both permanent and temporary, and access ways leading to and from the platforms or testing locations, must be designed and erected in such a manner as to conform to published safety laws and regulations. If the BTS observer identifies an unsafe condition that poses an undue risk to BTS, test consultant or facility field staff, the test will be postponed at his/her discretion.

A short list of safety concerns that could result in a test cancellation is as follows. This list is by no means all-inclusive.

- Respiratory hazards not addressed by PPE.
- Improper scaffold assembly and/or tie off.
- Corroded and/or structurally compromised surfaces.
- Unsafe, inappropriate or nonexistent (but necessary) climbing equipment, or other PPE.
- Unsecured electrical hazards.
- Dangerously icy, wet, or hot surfaces.
- High winds.

VII. REPORT PREPARATION & REVIEW

1. After completion of the test project, a report will be submitted to BTS within the time frame specified by the PCP or BOP Permit conditions and/or enforcement document. If no time frame has been specified, then the test report shall be submitted to BTS within thirty (30) calendar days, consistent with N.J.A.C. 7:27-8.4(f)5 for a PCP, or forty-five (45) calendar days, consistent with N.J.A.C. 7:27-22.18(e)3 for a BOP . If additional time is required, the individual responsible for the report preparation shall contact the appropriate Regional Enforcement Office.

The report shall include sufficient raw data to permit BTS staff to reproduce all results. The report shall contain the following:

- Prior facility information (Items 1 through 7 of Section IV.A. of this document);
- Source description and actual site information (diameters, dimensions, etc.);
- Summary of results (including audit results), including a comparison to the permitted emission allowables, and including a narrative describing qualified results and explaining any non-conforming quality control items;
- Production data including process and control device parameters;

- Copies of all raw field and laboratory data (including calibrations, standards, strip charts, etc.) and laboratory NJ certification information;
- Sample calculations; and
- Pre and post test equipment calibration records;

(*Note:* Failure to provide adequate information may result in rejection of the report; and thereby require a repetition of the stack emission tests).

- 2. All stack emission test reports must include the certification language specified in N.J.A.C. 7:27-1.39 as required by N.J.A.C. 7:27-8.15(c) or N.J.A.C. 7:27-22.18(h). The report must also be certified by a Professional Engineer or Certified Industrial Hygienist as required by N.J.A.C. 7:27-8.4(c) or N.J.A.C. 7:27-22.18(h). At the discretion of the Department, failure to provide these certifications will be justification to void the test results and require additional tests.
- 3. A copy of the summary of emission results with a comparison to the permitted emission allowables must be submitted to the Regional Enforcement Office having jurisdiction over the facility concurrent with the submittal of the complete test report to BTS. The summary report must be certified in the same manner as the complete test report.
- 4. The compliance basis for each parameter will be as stated in the applicable PCP or BOP Permit conditions. If the compliance basis is not stated in the Permit conditions, each test run will be evaluated individually in determining compliance, unless the basis is specified elsewhere by rule, regulation or DEP guidance for that specific parameter.
- 5. Upon review of the stack test report, BTS may request additional data to verify the test results or source operating parameters. At the discretion of the Department, failure to provide this additional data will be justification to void the test results and require additional tests.
- 6. Upon completion of the review of the test report by BTS, a letter will be sent to the facility indicating the findings of the review. The results of our review will also be forwarded to the appropriate Regional Enforcement Office for final disposition. Any technical questions on the findings shall be in writing and directed to the Section Chief, Emission Measurement Section, BTS. Questions related to the compliance status should be directed to the Regional Enforcement Office.
- 7. Service fees will be invoiced to the facility consistent with N.J.A.C. 7:27-8.6 or N.J.A.C. 7:27-22.31.

APPENDIX I

EMISSION TEST PRODUCTION REPORT FORM

NEW JERSEY DEPARTMENT OF ENVIRONMENTAL PROTECTION

EMISSION TEST PRODUCTION REPORT FORM

Introduction

The following form has been established in order to determine operating conditions for equipment associated with stack emission tests. This form is to be completed by plant personnel during the stack sampling procedure and should accurately reflect all operations of equipment during each test run. All information supplied will be deemed as being authentic and accurate to operational conditions and will be used for determining compliance status.

The form should be submitted to the appropriate Regional Enforcement Office having jurisdiction, with a copy included in the final test report submitted to the Bureau of Technical Services.

Instructions

The information being requested on the form should be filled out completely. Failure to complete may result in invalidation of the stack test. If a particular section of the form is not applicable, write "NA" in that section. If accessory information is available or required to completely provide the mandated information, this should be attached to the form as an addendum, which should note the section on the form it pertains to. If multiple stacks are being tested, one report form should accompany each.

SECTION I -	This information should be supplied as reported in the Preconstruction Permit
	or Operating Permit, for the equipment being tested.

- SECTION II Self Explanatory
- SECTION III List all conditions that pertain to operation parameters of equipment or control apparatus. State if the required condition is being achieved, if not explain why and when it will be met. If the condition directly relates to operations during the stack test they should be recorded and listed, with time of reading, during each test run (i.e. minimum temperature requirements, CEM readout) in Section III B.
- SECTION IV Supply the information requested for the subsection(s) which apply to the source operation. If conditions vary during each test run and/or from source to source they must be listed separately. Check the appropriate space that relates to the operating production rate during the test.

NOTE: Subsection IV A should contain a breakdown of raw materials by listing each component individually by chemical name. Include Chemical Abstract System (CAS) # if available. List percent used corresponding to each test run. If Material Safety Data Sheets (MSDS), production strip charts or logs or other information are available which accurately give raw materials, they may be attached as supplemental addendums.

- SECTION V Information in this section should be verified and recorded to establish the operation of the control devices from available direct readouts. These readouts should be recorded every 15 minutes. Copies of dated and signed strip charts may be used as replacement provided they clearly define the require information.
- SECTION VI Samples may be required to be taken and analyzed by the company or representatives of the company. This analysis is required to be conducted by a state certified lab. A duplicate sample may be required by the DEP person observing the test. This sample will be analyzed by the Department for verification of analytical results.

AIR COMPLIANCE AND ENFORCEMENT REGIONAL OFFICES

CENTRAL REGIONAL OFFICE

PO Box 407

Trenton, NJ 08625-0407

(609) 292-3187

Fax No. (609) 292-6450

COUNTIES: Burlington, Mercer, Middlesex, Monmouth and Ocean

NORTHERN REGIONAL OFFICE

7 Ridgedale Avenue

Cedar Knolls, NJ 07927

(973) 656-4444

Fax No. (973) 656-4080

COUNTIES: Bergen, Essex, Hudson, Hunterdon, Morris, Passaic, Somerset, Sussex, Union and Warren

SOUTHERN REGIONAL OFFICE

One Port Center

2 Riverside Drive

Camden, NJ 08162

(856) 614-3601

Fax No. (856) 614-3613

COUNTIES: Atlantic, Camden, Cape May, Cumberland, Gloucester and Salem

EMISSION TEST PRODUCTION REPORT FORM

I.	Company Name		APC Plant II	D#
	Plant Location			
	Permit Number (include PCP or B	OP designation)		
	Designation of Equipment		-	
II.	Emission Test Date(s)			
	Tests Conducted By:			
	Name of Firm			
	Business Address			
	Phone Number			
	Test Team Representatives _			
	-			
	Test Time (Start/Finish)	<u>Run #1</u> /	<u>Run #2</u> /	<u>Run #3</u> /
III.	Permit Operating Conditions A. List Conditions	Achieved (Y	es or No)	
		-		
		-		
		-		
		-		
		-		

B. Log of Permit conditions during Stack Test (Record at least every 15 minutes)

CONDITION	RUN #	READOUT	TIME OF RECORDING

IV. Equipment Operation/Process Parameters Number of Sources Connected ______ Number of Sources Operating ______

Production Rate:

Normal _____ Maximum _____

A. Raw Materials:

	RUN #1	RUN #2	RUN #3
USAGE RATE			
lbs/hr			
BREAKDOWN			
(% by weight)			

B. Surface Coating:

Material Being Coated _____

		Type of Coating		
		Coating Rate (gals/hr)		
		Is Coating Altered (Yes	s or No)	
		With		
		Distance From Coating Head	d to Exhaust Duct	
	C.	Fuel Burning / Incineration:		
		Type of Fuel		
		Fuel Burning Rate	(lbs/hr), (gals/hr), (ft ³ /hr)	
		Fuel Additives	%	
		Meter Reading (if available)	Time	
		Type of Waste Constituents		
		Auxiliary Fuel		
		Burning Rate		
	D.	Other:		
		Description of Operation and Proc	cess Rate	
V.	Cor	ntrol Equipment Parameters		
	CE	MS Required (Yes/No)		
		Contaminant(s)?		

STACK TEST CEMS READING

Parameter Cont/Read	Parameter Cont/Read	Parameter Cont/Read	Time	Test Run #

A. Control Equipment Performance Parameter

Parameter	Reading	Time	Run #

B. Additional Observations

Fugitive Emissions (Y	(es/No)
-----------------------	---------

Equipment Location

Visible Emission From Stack (Yes/No)

Odors Noticeable (Yes/No)

Vicinity of Equipment (Yes/No)

Near Exhaust Stack (Yes/No)

Off Property (Yes/No)

VI. Samples

Type of Sample
Time of Sampling
Sample By
Sample Taken From
To Be Analyzed For
Analyzed By
Form Information Supplied By: Name/Title (Please Print)
I contifu up der nonalter of low that I halieve the information movided in this de our

I certify under penalty of law that I believe the information provided in this document is true, accurate and complete. I am aware that there are significant civil and criminal penalties, including the possibility of fine or imprisonment or both, for submitting false, inaccurate or incomplete information.

Signature(s)/Date

DEP Usage Only

Rec'd By

Sample Rec'd Date/Time Rev'd By

APPENDIX II

PROTOCOL TEMPLATES FOR SELECTED METHODS

METHOD 1 - SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

Applicability and Principle

The correct selection of sampling ports and traverse points from which sampling for air pollutants will be performed will be selected pursuant to EPA Method 1. This method and BTS template are applicable only to stacks ≥ 12 inches internal diameter.

Stack Dimensions and Stack Gas Parameter

Internal Stack Diameter* = _____ in.

For rectangular ducts, the equivalent diameter listed above, calculated by Eqn. 1-1 of the method.

The stack dimensions are _____in. x _____in.

Stack Temp. (F) _____ ACFM _____ SCFM _____

Moisture (%) _____ DSCFM_____

Stack Diameters from the nearest flow disturbance (the minimum distances allowed to the nearest disturbance are two diameters downstream and one-half diameter upstream) are:

Downstream_____ Upstream_____

Note: If the minimum distances cannot be met, either a new sample location must be selected or the alternative measurement procedure in Section 11.5 of the method will be used. Specify equipment and procedures below:

Stack Diagram Detailing Sample Port Locations and Downstream/Upstream Distances:

The number of traverse/sample points will be selected as follows (choose one):

_____ Figure 1-1 of the method for isokinetic sampling. _____ Figure 1-2 of the method for non-isokinetic sampling.

The number of traverse points will be _____.

The location of the traverse/sample points will be selected as follows (choose one):

Table 1-2 of the method for round stacks. Table 1-1 of the method for rectangular stacks. The matrix layout will be _____x ____.

Sample ports will be located on a plane perpendicular to the direction of flow. For round stacks, two ports will be placed at 90 degrees to each other. For rectangular stacks, the number and location of the ports will be dictated by the chosen sample matrix (Table 1-1) and the division of the stack cross section into equal areas.

The traverse points will be located as follows. Any "adjusted" traverse points pursuant to Section 11.3.2 or 11.3.3 of the method will be noted:

Verification of the **absence of cyclonic flow** will be performed as per Section 11.4 of the method prior to the start of sampling. Documentation will be supplied to the on-site BTS observer and included in the final report.

Proposed deviations from this BTS Template or the Method

(Insert any proposed deviations here)

METHOD 2 - DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE (TYPE S PITOT TUBE)

Applicability

This method is applicable for the determination of the average velocity and the volumetric flow rate of a gas stream using an S type pitot tube. This method is not applicable at measurement sites that fail to meet the criteria of Method 1, Section 11.1. Also, the method cannot be used for direct measurement in cyclonic or swirling gas streams.

Apparatus (*Check applicable*):

Type S Pitot tube made of metal tubing with an external diameter between 0.48 and 0.95 centimeters (3/16 and 3/8 inch). There shall be an equal distance from the base of each leg of the pitot to its face-opening plane. The face openings shall be aligned as shown in Figure 2-2 of the Method, however slight misalignments are permissible. The pitot shall have a known coefficient, determined as outlined in Section 10 of the Method. If a baseline coefficient value of 0.84 is assigned to the pitot tube and upon inspection does not meet the criteria set forth in Section 10 of the Method, that pitot tube will not be used.

_____Standard Pitot tube. If a standard pitot tube will be used, it must meet the specifications of Section 6.7 and 10.2 of the method. It will be checked to insure the impact holes are free from obstruction. If there are particulates in the gas stream then the steps in Section 6.1.2 of the method will be taken to insure the holes are not plugged during the test (ie. back purging of the pitot and comparing the pre and post readings).

Differential Pressure Gauge (choose one)

_____An inclined manometer will be used.

_____The equivalent of an inclined manometer will be used as follows:

(If another type of differential pressure gauge is used, ie. Magnahelic, etc., their

calibration must be checked after each test series.)

Note: most gauges have a 10 in. (H₂O column) inclined vertical scale, having 0.01 in. H₂O divisions on the 0 to 1 in. scale, and 0.1 in. H₂O divisions on the 1 to 10 in. vertical scale. This type of manometer is acceptable for delta p values as low as 1.3 mm (0.05 in.) H₂O. **However, a gauge of greater sensitivity will be used if any of the following is found to be true:** (1) the arithmetic average of all delta p readings at the traverse points in the stack is less than 1.3 mm (0.05 in.) H₂O; (2) for traverses of 12 or more points, more than 10 percent of the individual delta p readings are below 1.3 mm (0.05 in.) H₂O, or (3) for fewer than 12 points, more than one delta p reading is below 1.3 mm (0.05 in.) H₂O.

As an alternative for the preceding three criteria, Equation 2-1 in Section 12.2 of the method can be used to determine the necessity of using a more sensitive differential pressure gauge.

Expected delta p range = _____

Inclined manometer scale = _____

An alternative flow measuring device will be used as follows:

Procedure

A pretest leak check of the pitot tube and manometer will be conducted as per Section 8.1 of the method. The manometer will be leveled and zeroed prior to use as well as periodically checked during the test.

The velocity head and temperature will be measured at each traverse point specified by EPA Method 1. The static pressure in the stack will be measured during the test as well as the atmospheric pressure. The stack gas dry molecular weight will be determined in accordance with EPA Method 3 or 3A.

Calculations

All calculations will be performed in accordance with Section 12 of the method.

Proposed deviations from this BTS Template or the Method

(Insert any proposed deviations here)

METHOD 3 - GAS ANALYSIS FOR THE DETERMINATION OF DRY MOLECULAR WEIGHT

Applicability and Principle

This method is applicable for the determination of CO_2 and O_2 concentrations and dry molecular weight of a sample from an effluent gas stream of a fossil-fuel combustion process or other process. The sample is taken in a flexible bag and analyzed with an orsat analyzer or alternatively, with analyzers using the procedures from Method 3A.

A gas sample is extracted from a stack by either single point integrated or multi-point integrated sampling. The gas sample is analyzed for percent CO_2 , O_2 , and if necessary CO.

Apparatus

Pump. A leak free diaphragm-type pump, or it's equivalent, to transport the sample to the flexible bag.

Rate Meter. A rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate will be used. A flow rate of 500 to 1000 cc/min. will be used unless another rate is required and described in the "Proposed Deviations" section of this template.

Flexible Bag. Any leak free plastic (Tedlar, Teflon, Mylar, etc.) bag or equivalent having a capacity consistent with the selected flow rate and sample time **leak checked** in accordance with Section 6.2.6 of the method. Type of Bag _____

Pressure Gauge. A water filled U-tube manometer, or equivalent, of about 30 cm (12 in.) for the bag leak check.

Vacuum Gauge. A mercury manometer, or equivalent, of at least 760 mm (30 in.) Hg, for sampling train leak check.

Procedure (*select one*)

_____ Single Point Integrated. The sampling point will be at the centroid of the cross section of the duct. The bag will be leak checked as in Section 6.2.6 of the Method. The train will be leak checked as in Section 8.2.2 of the Method. The sampling system will be purged prior to connecting the sample bag. The sample will be taken at a constant rate simultaneous with, and for the same total length of time as, each pollutant emission rate determination. At least 30 liters of sample will be taken.

<u>Multi-Point Integrated Sampling</u>. The minimum number and location of the traverse points will be chosen in accordance with EPA Method 1. The procedures outlined in Sections 8.2.2 through 8.2.4 of Method 3 will be followed, except that the sample will be taken while traversing the stack from each sample point for an equal length of time.

Emission Measurement Test Procedure

Within 8 hours of collection, the sample will be analyzed for percent $CO_{2 \text{ and}}$ percent O_{2} using an orsat analyzer. The orsat analyzer will be leak checked in accordance with Section 6 of the Method prior to any sample analysis. The analysis and calculations for each sample will be repeated until any three analysis differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). The three molecular weights will be averaged and reported to the nearest 0.1 lb/lb-mole.

Several compounds may interfere with the orsat analysis (ie. acid gases, unsaturated hydrocarbons, nitrous oxide and ammonia). Source specific compounds that may interfere with the orsat analysis are: ______.

Action to be taken:

Date the orsat analyzer was checked against known O₂ and CO₂ standards _____

Alternatively, if direct analysis of the bag samples will be performed by EPA Method 3A procedures, it will be indicated here and the procedures will be included in the protocol.

Calculations

The calculations will be performed in accordance with Section 12 of the method.

Proposed Deviations from this BTS Template or the Method

(Insert any proposed deviations here)

METHOD 3A DETERMINATION OF CARBON DIOXIDE AND OXYGEN FROM STATIONARY SOURCES

Applicability and Principle

A sample is continuously extracted from the effluent stream portion of the sample stream is conveyed to an instrumental analyzer(s) for the determination of CO_2 and O_2 concentrations. Performance specifications and test procedures are provided to ensure reliable data.

<u>Apparatus</u>

A **measurement system** for Carbon Dioxide and Oxygen that meets the specifications in Section 13.0 of this method will be used.

The **Sample Probe** will be of sufficient length to traverse the sample points. The sampling probe shall be heated to prevent condensation if also being used to measure SO_2 and/or NOx.

The **Sample Line** will transport the sample gas to the moisture removal system and be heated (sufficient to prevent condensation) if also being used to measure SO_2 and/or NOx.

The **Moisture Removal System** will be a refrigerator-type condenser or similar device to continuously remove condensate from the sample gas while maintaining minimal contact between the condensate and the sample gas.

The **Sample Transport Lines** will transport the sample from the moisture removal system to the sample pump, sample flow rate control, and sample gas manifold.

A **Calibration Valve Assembly** with a three-way valve assembly, or equivalent, for introducing calibration gases either directly to the analyzer in direct calibration mode, or into the measurement system at the probe in system calibration mode, will be utilized. When in the system calibration mode, the system must be able to flood the sampling probe and vent excess gas.

A **Particulate Filter**, either an in-stack or heated (sufficient to prevent water condensation) out-of-stack filter will be used. If an out-of-stack filter is used, it will be included in the system bias test.

A **Leak-free Pump**, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system will be used. The pump will be constructed of any material that is nonreactive to the gas being sampled.

A **Recorder**, a strip-chart recorder, analog computer, or digital recorder for recording measurement data. The minimum data recording requirement is one measurement value per minute. A strip-chart recorder can only be used as a back-up system.

Analytical Span and Calibration Gases

The span of the monitoring system is equivalent to the high-level calibration gas value (see below) and will be selected such that to the extent practicable, the measured emissions will be between 20 - 100% of the selected calibration span. If at any time during the run the measured gas concentration exceeds the readable range of the analyzer, the run may be considered invalid. If the average of any run exceeds the calibration span value, the run is invalid.

The in stack concentration is expected to be _____ for O_2 . The in stack concentration is expected to be _____ for CO_2 .

The **high-level calibration gas sets the calibration span** and results in measurements being (to the extent practicable) equivalent to 20 to 100 percent of the span. Based on the expected concentrations above, the high-level gas (span) concentration to be used during the source test will be ______ for O_2 and ______ for CO_2 .

The **mid-level calibration gas** will be equivalent to 40 to 60 percent of the span. The mid-level calibration gas concentration to be used during the source test will be ______ for O_2 and ______ for CO_2 .

The **low-level calibration gas** will be less than 20 percent of the calibration span. A zero gas meeting the definition of "zero air material" in 40CFR72.2 may be used. The low-level gas concentration to be used during the source test will be ______ for O_2 and ______ for O_2 .

The calibration gases for CO_2 analyzers will be CO_2 in N_2 , CO_2 in air or a calibration gas mixture, as indicated in Section 7.1 of the method. The calibration gases will be (check one):

- Certified within an uncertainty of 2.0 percent in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards" (EPA Protocol gases). If a zero gas is used for the low-level gas, it will meet the definition of "zero air material" in 40CFR72.2, as opposed to being an EPA Protocol gas.
- _____Blended gas mixtures meeting the protocol above, provided that the additional gas components are shown not to interfere with the analysis.
- Calibration gases will be prepared from EPA Protocol gases using Method 205. Separate procedures for Method 205 will be included in the protocol. Note: Part 75 applications require EPA approval for the use of Method 205.

Note: Pre-cleaned or scrubbed air may be used for the O_2 high-level calibration gas, provided it does not contain other gases that interfere with the O_2 measurement.

Analyzer Calibration

The **analyzer calibration error check** (or system calibration test for dilution systems) will be conducted by introducing the low-level, mid-level, and high-level gases to the analyzer (or through the entire measurement system, introduced at the probe, for a dilution system). During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow rate at the analyzer. The analyzer responses to each calibration gas will be recorded. The analyzer calibration error check will be considered invalid if the gas concentration displayed by the analyzer exceeds ± 2 percent of the span for any of the calibration gases, or > 0.5 percent O_2 or 0.5 percent CO_2 absolute difference (as applicable).

The **sampling system bias check** will be performed by introducing first an upscale gas (midrange or high-level, whichever more closely approximates the stack concentration) at the calibration valve assembly installed at the outlet of the sampling probe, and then the low-level gas. During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow at the analyzer. This check will be considered invalid if the difference between the calibration bias check and the calibration error check for the same calibration gas exceeds \pm 5% of the span, or > 0.5 percent O₂ or 0.5 percent CO₂ absolute difference (as applicable).

During the initial sampling system bias check, the **measurement system response time** is determined, as detailed in Sections 8.2.5 and 8.2.6 of Method 7E.

Documentation that an interference check has been conducted in accordance with Section 8.2.7 of Method 7E will be available on-site and will be included in the final test report. Any specific technology, equipment or procedures intended to remove interference effects will be operating properly during the stack test.

Stratification Determination

Prior to sampling, or as part of the first test run, a stratification check must be performed in accordance with Section 8.1.2 of Method 7E. If more than one instrumental method that requires a stratification check is being performed, the stratification check need only be done on one of them. A stratification check is not required for stacks < 4 inches in diameter. Additionally, when Method 3A is being used <u>only</u> to determine the stack gas molecular weight, single point sampling at a point selected in accordance with Section 8.2.1 of Method 3 can be used without performing a stratification check.

A stratification check _____ is _____ is not required (check one) for this test program. If required and being done using a Method 3A analyzer, the procedures to follow will be employed.

The stratification check will be conducted at either (1) twelve traverse points selected in a accordance with EPA Method 1, or (2) three traverse points spaced on a line passing through the centroidal area at 16.7, 50.0 and 83.3 percent of the measurement line. Each point will be sampled for a minimum of twice the response time.

The minimum number of traverse points required for sampling will be determined as follows. If each traverse point differs from the mean by no more than the least restrictive of:

Difference from mean	Stratification Class	Number of required sample
		points
\pm 5%, or \pm 0.3% O ₂ or CO ₂ (as	Unstratified	A single point that most closely
applicable)		matches the mean.
Between \pm 5% and \pm 10%, or \pm 0.5%	Minimally stratified	Three (3) sample points spaced
O_2 or CO_2 (as applicable)		at 16.7, 50.0 and 83.3 percent
		of the measurement line. *
Greater than $\pm 10\%$, and greater than	Stratified	Twelve (12) sample points
$\pm 0.5\%$ O ₂ or CO ₂ (as applicable)		located consistent with EPA
		Method 1 criteria.

* For a minimally stratified stack with an internal diameter greater than 2.4 meters (7.8 feet), the three sampling points may be located at 0.4 meters (1.3 feet), 1.0 meters (3.28 feet) and 2.0 meters (6.56 feet) along the measurement line showing the highest average concentration. This option will only be available if the stratification check consisted of twelve points.

Emission Measurement Test Procedure

The sampling probe will be placed at the first sample point and sampling will begin at the same rate used during the bias check. A constant rate \pm 10 percent will be maintained during the entire sample run. Sampling will commence only after twice the response time has elapsed. Sampling will be conducted for an equal length of time at each traverse point.

Immediately following the completion of the test period and hourly during the test period, the low-level calibration gas and an upscale calibration gas (the mid-level or high-level as appropriate) will be re-introduced one at a time to the measurement system at the calibration valve assembly. No adjustments to the measurement system will be made until both the low-level and upscale bias and drift checks are made. The analyzer response will be recorded. If the bias values exceed the specified limits, the test results preceding the check will be invalidated and the test will be repeated following corrections to the measurement system and full recalibration. If the drift values exceed the specified limits, the run may be accepted but the test measurement system will be fully recalibrated and the results reported using Eqn. 7E-5b (Eqn. 7E-5a if a non-zero gas is used for the low-level calibration gas) of Method 7E.

Measurement System Performance Specifications

Drift, less than or equal to ± 3 percent of the span value for the low-level or upscale gas. **Sampling System Bias**, less than or equal to ± 5 percent of span value for the low-level or upscale gas. **Calibration Error**, less than or equal to ± 2 percent of span for each calibration gas.

An alternative acceptance criteria for each of these measurements is less than or equal to 0.5 percent O_2 or 0.5 percent CO_2 absolute difference (as applicable).

Emission Calculation

The average gas effluent concentration will be determined from the average gas concentration displayed by the gas analyzer and is adjusted for the zero and upscale sampling system bias checks, as determined in accordance with the procedures specified above. The average gas concentration displayed by the analyzer may be determined by averaging all of the effluent measurements indicated by the data acquisition system for the test run. The minimum frequency for data recording will be one-minute averages during the run. A chart recorder will only be used as a backup to the data acquisition system. The effluent gas concentration will be calculated using Eqn. 7E-5b (Eqn. 7E-5a if a non-zero gas is used for the low-level calibration gas) of Method 7E.

$$Cgas = (Cavg - Co) \frac{CMA}{CM - Co}$$
Eqn. 7E-5b

$$Cgas = (Cavg - CM) - CMA - COA + CMA - CMA - COA + CMA - C$$

Proposed Deviations from this BTS Template or the Method

(Insert any proposed deviations here)

METHOD 4 - DETERMINATION OF MOISTURE CONTENT IN STACK GASES

Applicability and Principle

This method is applicable for the determination of the moisture content of stack gas.

A gas sample is extracted either at a constant rate or utilizing an isokinetic sampling train; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

Apparatus

Probe. Stainless steel or glass tubing, sufficiently heated to prevent water condensation, and is equipped with either an in-stack filter (ie. glass wool plug) or heated out of stack filter as described in EPA Method 5.

Condenser. The condenser will consist of four impingers connected in series with ground glass, leak-free fittings or any similar non-contaminating fittings. The first, third and fourth impingers will be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm. (1/2 in.) ID glass tube extending to about 1.3 cm. from the bottom of the flask. The second impinger will be of the Greenburg-Smith design with the standard tip. The first two impingers will contain known volumes of water, the third will be empty, and the fourth will contain a known weight of 6 to 16 mesh indicating type silica gel, or equivalent desiccant.

Cooling System. An ice bath container and crushed ice, or equivalent, are used to aid in condensing moisture.

Metering System. This system will include a vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3 deg. C (5.4 deg. F) and a dry gas meter capable of measuring volume to within 2 percent.

Barometer. Mercury, aneroid or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in.).

Graduated Cylinder and/or **Balance**. These items are used to measure the condensed water in the impingers and silica gel to within 1 ml or 0.5g. Graduated cylinders will have subdivisions no greater than 2 ml. The balance will be capable of weighing to the nearest 0.5 g or less.

Procedure

A minimum total gas volume of 0.60 scm (21 scf) will be collected, at a rate no greater than $0.021 \text{ m}^3/\text{min}$ (0.75 cfm). The moisture determination will be conducted simultaneous with, and for the same total length of time as, the pollutant emission rate run. The train will be set up as shown in Figure 4-1 of the method. The probe and filter (if applicable) will be heated to about

120 deg. C (248 deg. F), to prevent water condensation ahead of the condenser. After the train is heated and the impingers iced down, a leak check will be performed with an acceptable rate of 4 percent the average sampling rate or 0.02 cfm, whichever is less. During the run the sampling rate will be maintained within 10 percent of constant rate. The dry gas meter volume will be recorded at the beginning and end of each sampling time increment and whenever sampling is halted. More ice will be added, if necessary, to maintain a temperature of less than 20 deg. C (68 deg. F) at the silica gel outlet. When the run is completed, a post leak check is performed, with the same acceptance criteria as for the pre-test leak check. The volume and weight of condensed moisture is measured to the nearest ml and 0.5 g, respectively.

Contaminant(s) tested and sampling time _____

Moisture train sampling time _____

In gas streams that contain water droplets, this method may produce a positive bias. If this is suspected for this source, either a wet bulb dry bulb and psychometric chart (correcting for stack pressure) or saturation and vapor pressure table determination will be conducted simultaneously with the moisture sample train, as described in the method.

The stack gas will _____ will not _____ contain moisture droplets.

Calculations

The calculations will be performed in accordance with Section 12.0 of the method.

Proposed deviations from this BTS Template or the Method

(Insert any proposed deviations here)

NEW JERSEY AIR TEST METHOD 1 – DETERMINATION OF PARTICULATE MATTER FROM STATIONARY SOURCES

General Applicability and Principle

Particulate matter determination requires isokinetic sampling of the exhaust stack flue gases and particulate matter is determined gravimetrically after the removal of uncombined water. The measured emission weight will be the combined weight of all particles collected and analyzed in accordance with these sampling and analytical procedures.

Required Test Data

The following test data shall be determined and reported for each run.

- 1) Average dry gas meter temperature (degrees Fahrenheit).
- 2) Average stack temperature (degrees Fahrenheit).
- 3) The root mean square value of differential pressures (inches of water) of all traverse points in the stack during each test run.
- 4) Average differential pressure (inches of water) across the orifice meter during each test run.
- 5) Diameter of the stack cross-sectional area at sampling location.
- 6) Weight (grams) of total solid particles collected during each test run.
- 7) Moisture content by volume in stack gas during each test run, determined by EPA Method 5 moisture procedures.
- 8) Volume of gas (cubic feet) sampled during each test run.
- 9) Source gas emission rate (SCFM).
- 10) Molecular weight of the stack gas, determined by EPA Method 3 or 3A.
- 11) Emission rate for each test run in the following units:

Source Specific Method Application

A minimum sample catch of 50 mg is targeted to calculate the minimum sample times. An absolute minimum of 3 mg catch is required, but any expected catch of less than 50 mg will require the use of EPA Alternative Method 005 to process the samples. Sampling times should be as close to the compliance basis (at a minimum equal to the compliance basis) as possible, while still obtaining a representative sample catch. The following equations will calculate the sample catch and the sample time.

LB/HR (from Permit allowable) =	
DSCFM (from Permit information) =	
Vsamp (variable to change to increase catch) =	cf
gr/DSCF = (LB/HR)*7000/(60*DSCFM) = mg = (gr/DSCF)/7000*Vsamp*453.59*1000 =	
Sample rate (min. of 30 cf/hr) =	cf/hr
Sample time (hr) = Vsamp/Sample rate =	
Based on these calculations, a sample time of ______ minutes/run will be performed.

Sampling Train Components

The sampling train will consist of the following:

- 1) A tapered-edge **sampling nozzle** constructed of stainless steel and a **glass-lined probe**.
- 2) **Pitot tube** with a known coefficient.
- 3) A heating system that includes in-line thermocouples to measure the gas stream temperature across the particulate filter. The temperature will not exceed 225 degrees Fahrenheit and will only be sufficiently hot to prevent condensation of water on the filter.
- 4) A glass fiber filter in a glass holder.
- 5) **Four Greenburg-Smith impingers**, the first two containing 100 ml of distilled water, the third impinger empty and the fourth containing a known quantity of silica gel, all submerged in an ice bath which will ensure that they will be kept below 68 degrees Fahrenheit. The first, third and fourth impingers will be modified Greenburg-Smith and the second will be a Greenburg-Smith impinger with a standard tip, as described in Section 6.1.1.8 of EPA Method 5.
- 6) A **leak-free pump** with flow control adjusters and a vacuum gauge.
- 7) A **dry gas meter** accurate within two percent and temperature sensors to indicate the gas inlet and outlet temperatures
- 8) An **orifice meter** with an inclined manometer (leveled and zeroed prior to use)
- 9) A **thermocouple** equipped with a temperature sensor attached to the probe adjacent to the nozzle to indicate the stack gas temperature.

All glassware will be interconnected with glass fittings having ball joints. All components will meet the specifications of the Method.

Preparation for Sampling

All internal surfaces of the nozzles, probes, impingers, connecting glassware and hoses will be cleaned and dried prior to sampling. The open ends of all sampling components will be covered to prevent contamination. A tared filter (dried at 220-230 degrees Fahrenheit, cooled in a desiccator to room temperature and weighed to the nearest 0.1mg) will be placed in the glass filter holder.

A fourth impinger will be charged with 200-300 grams of silica gel and sealed against contamination. The first and second impingers of the sampling train will be charged with 100 ml of distilled, deionized water and connected to the remaining impingers. The third impinger will be empty. The impingers will be placed in the sampling train and surrounded with crushed ice.

The entire sampling system will then be transported to the sampling site for assembly and leak check. The leak check procedure consists of assembling the sample system, starting the sampling pump and blocking the nozzle inlet. The pump is then adjusted to pull a minimum of 15 inches of mercury vacuum. When the vacuum reading stabilizes, the dry gas meter is observed for one minute to determine the sample system leak rate. If the leak rate is less than 0.02 cubic feet per minute, sampling proceeds. When the heating system reaches the proper temperature, sampling will begin.

Sampling

When the probe and filter are up to temperature, the probe will be inserted into the stack at the first traverse point with the nozzle pointed into the gas stream. The dry gas meter initial volume will be recorded and the vacuum pump will be immediately started and the flow adjusted to isokinetic conditions. For each run, the data required on Figure 5-3 of EPA Method 5 will be recorded using that or a similar data sheet. Readings indicated by Figure 5-3 will be taken at each sample point during each time increment and additional readings when significant changes necessitate additional adjustments in flow rate. Each sampling test will consist of **three separate and valid** test runs, unless otherwise specified by the Department.

Leak checks will be performed when sampling is completed at the end of each test. The dry gas meter volume will be recorded and the sample system will be leak checked. A run will be considered valid if the leak rate upon completion of the run is less than 0.02 cubic feet per minute, the final isokinetic sampling rate is between 90 and 110 percent and the filter temperature was maintained consistent with the method.

Sample Recovery Procedures

Following the completion of each test run the sampling train will be transported to a recovery area onsite. The recovery site will be clean and protected from the wind. The sample recovery will be as follows:

- 1) Container 1 Disassemble the filter housing and transfer the filter to its original glass petri dish. Seal the petri dish with Teflon tape and label it with the appropriate sample information.
- 2) Container 2 The front half of the train; nozzle, probe, and front-half filter housing are rinsed and nylon bristle brushed with acetone into an amber glass container with a Teflon-lined cap. This procedure will be performed three times or more until no visible particulate remains, after which the container is sealed and labeled. The liquid level will be marked on the container to ensure no leakage occurred during transport.
- 3) The contents of the first three impingers are measured for volume and discarded.
- 4) Container 3 Note the color of the silica gel to determine whether it has been completely spent and make a notation of its condition. The silica gel is returned to its original container and weighed to obtain a final weight.
- 5) Container 4 100 ml of acetone will taken directly from the wash bottle being used for sample recovery into a separate glass sample container and labeled "Acetone Blank".
- 6) All containers are checked to ensure proper sealing, labeling, and that all liquid levels are marked. All samples are logged onto a chain-of-custody record.

Analysis

The filter will be dried at the average temperature maintained during the corresponding run and desiccated for 24 hours. The filter will be weighed to a constant weight and the final weight will be recorded to the nearest 0.1 mg. The probe and nozzle washings will be transferred to a clean, tared glass weighing dish. The dish will be evaporated at ambient temperature and pressure, then desiccated for 24 hours and weighed to the nearest 0.1 mg.

A 100 ml sample of acetone will be collected as a field blank. The blank will be evaporated at ambient temperature and pressure, then desiccated for 24 hours and weighed to the nearest 0.1 mg. The maximum blank acetone correction will be $\leq 0.001\%$ residue (by weight). This equates to a maximum blank correction of 0.0079 mg/ml of acetone rinse (0.01 mg/g acetone).

Calculations

All calculations will be carried out in accordance with the method, with all final results reported at one significant figure beyond the allowable.

QA/QC Procedure

All pitot tubes, dry gas meters, orifice meters, and thermocouples that are used in this stack test program will be properly calibrated prior to testing. Calibration records will be made available to the on-site DEP observers and submitted to the Department in the final test report.

Chain-of-custody documentation for all samples will be implemented at the completion of sampling and documented until the samples are received by the laboratory for analysis and submitted to the Department.

Proposed Deviations from this BTS Template or the Method

(Insert any proposed deviations here).

METHOD 6 – DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

Applicability

This method is applicable to the determination of sulfur dioxide emissions from stationary sources. This template will not be used to propose combining particulate sampling with sulfur dioxide sampling. The Method 8 template would be used in that situation.

Principle

A stack sample is withdrawn from a sampling point in the stack and is collected in three midget impingers. The sulfuric acid mist (including sulfur trioxide) is separated and the sulfur dioxide is measured by barium-thorin titration.

Interferences

Possible interferents are free ammonia and fine metallic fumes. Where fine metallic fumes are present, high efficiency glass fiber filters must be used in place of the standard glass wool called for in the method. If free ammonia is present, alternate procedures must be used (see Special Situations Section). The preferred option to avoid interference would be to use Method 6C instead. If Method 6C cannot be used, an explanation will be included below.

Based on the above we (appropriate box checked):

- _____ Do not expect any interference.
- _____ Do expect interference. The description and discussion of the anticipated interference follows.

In–Stack Detection Limits & Sample Times

The minimum detection limit of the method has been determined to be 3.4 mg/m^3 (2.12E-07 lb/ft³) for a standard 60 minute (liter) sample. Actual in-stack method detection limits (ISDL) are based on actual source sampling parameters and analytical results. Actual detection limits can be improved through increased stack gas sampled (sample time). For this source, the instack concentration is:

Allowable = _____ lbs/hr Stack flow = _____ dscfm

 $lb/ft^3 = (lb / hr) / (dscfm x 60) =$ _____lb/ft³

Therefore, the sample time will be ______ minutes.

Sample Train & Recovery Components & Supplies

A schematic of the sampling train is shown in Figure 6-1 of the method. Specifically, the sampling train will be constructed with components specified under EPA Method 6, Section 6.0, with the following highlights.

Sample Train

- 1) **Probe liner** will be constructed of Borosilicate glass or stainless steel and be heated.
- 2) A **filter**, either in-stack or heated out-of-stack. In most cases, a glass wool plug is sufficient. If fine metallic fumes are present (identified above) a high efficiency glass fiber filter will be used.
- 3) The impinger train will consist of three to four midget impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. Silicone grease may be used if necessary. The first impinger or bubbler contains 15 ml 80% isopropanol (IPA) packed with glass wool prevents sulfuric acid mist carryover. The second and third impingers contain 15 ml of 3% H₂O₂. The final impinger is dry. A drying tube packed with silica gel is placed at the exit of the last impinger.
- 4) A Method 8 sampling train may be substituted for the Method 6 sampling train (see Special Situations Section).
- 5) **Pump** leak-free diaphragm pump, or equivalent, with a small surge tank between the pump and rate meter.
- 6) **Rate Meter -** Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of about 1 liter/min (0.035 cfm).
- 7) **Dry Gas Meter** (DGM) sufficiently accurate to measure the sample volume to within 2 percent, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature sensor (dial thermometer, or equivalent) capable of measuring temperature accurately to within 3 °C (5.4 °F). A critical orifice may be used in place of the DGM specified in this section provided that it is selected, calibrated, and used as specified in Section 16.0.

Sample Recovery

- 1) Wash bottles (glass or polyethylene); two 500 ml.
- 2) **Storage bottles** (polyethylene) one 100 ml per sample.

Sampling

Pre-test leak checks and post-test leak checks will be conducted as follows: temporarily attach a suitable rotameter to the outlet of the dry gas meter and place a vacuum gauge at or near the probe inlet. Plug the probe inlet and pull a vacuum of least 10 in. Hg. A leak rate as indicated by the rotameter shall not exceed 2% of the sampling rate. Pump leak checks are recommended.

The sampling train will be assembled as indicated above. Crushed ice and water will be placed around the impingers. The initial DGM reading and barometric pressure will be recorded. The sampling rate will be adjusted to approximately 1.0 liter/min as indicated by rate meter. This flow rate will be maintained ($\pm 10\%$) throughout the run. Readings as required by the method

(DGM volume, temperatures at DGM and at impinger outlet, and rate meter flow rate) will be recorded at least every 5 minutes.

Post Test Purge

At the conclusion of an acceptable post leak check, the sampling train will be purged with clean ambient air (optionally passed through either charcoal or a midget impinger containing 15 ml 3% H_2O_2 if necessary) for 15 minutes at the sampling rate.

Sample Recovery

After disconnecting the impingers from purging, the contents of the midget bubbler/impinger will be discarded. The contents of the midget impingers will be poured into a leak free polyethylene bottle for shipment. The three midget impingers and connecting tubes will be rinsed with water and the washings added to the same container. The fluid level will be marked.

30 ml of 3% H₂O₂ will be collected as a blank in a separate container.

Sample Preparation & Analysis

The level of the fluid in the container will be noted. If significant leakage occurred, either the run will be voided or methods will be used to correct the results, subject to BTS approval. The contents of the storage container will be transferred to a 100 ml volumetric flask and diluted to exactly 100 ml with water. A 20 ml aliquot will be pipetted into a 250 ml Erlenmeyer flask and 80 ml of 100% isopropanol and two to four drops of thorin indicator will be repeated and averaged. A blank will be run with each series of samples. The blank is prepared for analysis in the same manner as the samples, as indicated above. Replicate titrations will agree within 1 % or 0.2 ml, whichever is larger.

Calculations

All calculations will be performed as per Method 6, including blank corrections. Detailed sample calculations will be included in the final report.

Emissions will be presented in the following units:

Audit Samples

If provided, audit samples will be analyzed consistent with Section 11.3 of the method and the results will be provided in the final test report. It is recognized that failure to achieve method acceptance criteria for the audit could result in the requirement to repeat the stack test program.

Proposed Deviations from this BTS Template or the Method

(Insert any proposed deviations here)

Special Situations Section – If either of these two situations is relevant, they will be checked in the checkbox prior to the procedure.

(_) 1. Alternative Procedures when Ammonia is Present:

Sampling and Recovery Procedures

The probe will be maintained at 275° C (527° F) and equipped with a high-efficiency instack filter (glass fiber) to remove particulate matter. The filter material will be unreactive to SO₂. Whatman 934AH (formerly Reeve Angel 934AH) filters treated as described in Reference 10 in Section 17.0 of Method 5 is an example of a filter that has been shown to work. Where alkaline particulate matter and condensed moisture are present in the gas stream, the filter will be heated above the moisture dew point but below 225° C (437° F).

Sample recovery will be the same as above, except the contents of the midget IPA bubbler/impinger, along with water rinses of the bubbler/impinger, will be transferred to a separate polyethylene bottle.

Analysis

Analysis will be as described in Section 16.3.3 of the method.

(_) 2. Substituting Method 8 sampling train for midget impingers:

The sampling equipment described in Method 8 may be substituted for the midget impinger train described herein; however, the train must be modified to include a heated filter between the probe and isopropanol impinger and the operation of the sampling train and sample analysis must be at flow rates and solution volumes defined in Method 8.

METHOD 6C - DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

Applicability and Principle

A sample is continuously extracted from the effluent stream portion of the sample stream and conveyed to an instrumental analyzer using either ultraviolet (UV), nondispersive infrared (NDIR), or fluorescence for the determination of SO₂ concentrations. Performance specifications and test procedures are provided to ensure reliable data.

The 2006 revisions to EPA Method 6C failed to properly address issues related to "quenching" when fluorescence based analyzers are used for the determination of SO_2 emissions. Accordingly, when a fluorescence based analyzer is used, BTS requires the calibrations gas concentrations comply with the specifications of the prior version of Method 6C, with the details specified in the "Proposed Deviations from this BTS Template or the Method" section of this Template. The following paragraph indicates the calibration gas specifications from the prior version of Method 6C:

For fluorescence-based analyzers, the O_2 and CO_2 concentrations of the calibration gases as introduced to the analyzer shall be within 1 percent (absolute) O_2 and 1 percent (absolute) CO_2 of the O_2 and CO_2 concentrations of the effluent samples as introduced to the analyzer. Alternatively, for fluorescence based analyzers, use calibration blends of SO_2 in air and the nomographs provided by the vendor to determine the quenching correction factor (the effluent O_2 and CO_2 concentrations must be known).

The principle of detection to be used during the test program will be _____

<u>Apparatus</u>

A **measurement system** for Sulfur Dioxide that meets the specifications in Section 13.0 of this method will be used. Dual range analyzers may be used when needed, provided that both ranges meet all the quality assurance requirements of this method. When an analyzer is routinely calibrated with a calibration span of less than or equal to 20 ppmv, the manufacturer's stability test (MST) is required (Table 7E-5 of Method 7E) prior to testing and documentation will be included in the test report.

The **Sample Probe** will be glass, stainless steel, or equivalent, of sufficient length to traverse the sample points. The sampling probe shall be heated to prevent condensation.

The **Sample Line** will be heated (sufficient to prevent condensation) stainless steel or Teflon® tubing, to transport the sample gas to the moisture removal system.

The **Moisture Removal System** will be a refrigerator-type condenser or similar device to continuously remove condensate from the sample gas while maintaining minimal contact between the condensate and the sample gas. If a wet-basis system is being proposed, details will be included at the end of this template in the protocol submittal.

The **Sample Transport Lines** will be stainless steel or Teflon tubing, to transport the sample from the moisture removal system to the sample pump, sample flow rate control, and sample gas manifold.

A **Calibration Valve Assembly** with a three-way valve assembly, or equivalent, for introducing calibration gases either directly to the analyzer in direct calibration mode, or into the measurement system at the probe in system calibration mode, will be utilized. When in the system calibration mode, the system must be able to flood the sampling probe and vent excess gas.

A **Particulate Filter**, either an in-stack or heated (sufficient to prevent water condensation) out-ofstack filter will be used. All filters will be fabricated of materials that are nonreactive to the gas being sampled. If an out-of-stack filter is used, it will be included in the system bias test.

A **Leak-free Pump**, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system will be used. The pump will be constructed of any material that is nonreactive to the gas being sampled.

The **Recorder** will be a computerized data acquisition system, digital recorder or data logger used for recording measurement data. The minimum data-recording requirement is one measurement value per minute. A strip-chart recorder can only be used as a back-up system.

Analytical Span and Calibration Gases

The span of the monitoring system is equivalent to the high-level calibration gas value (see below) and will be selected such that to the extent practicable, the measured emissions will be between 20 - 100% of the selected calibration span. In practical terms, the span is selected such that a pollutant gas concentration equivalent to the emission <u>standard</u> is not less than 30% of the span. If at any time during the run the measured gas concentration exceeds the readable range of the analyzer, the run may be considered invalid. If the average of any run exceeds the calibration span value, the run is invalid.

Allowable =	lb/hr	MW = 64 lb/lbmole
Stack Flow =	dscfm	

The in-stack concentration based on the emission standard and stack flow parameters is

 $\frac{(\text{lb/hr}) \times (387 \times 10^6)}{(\text{MW}) \times (\text{dscfm}) \times 60} = \underline{\qquad} \text{ppm.}$

Note: when actual concentrations differ significantly from the standard, the span may need to be modified accordingly, such that the method criteria for span selection are met. Determining the emission standard is the first step in approximating the necessary span.

The **high-level calibration gas sets the calibration span** and results in measurements being (to the extent practicable) equivalent to 20 to 100 percent of the span. Based on the calculation above, the high-level gas (span) concentration to be used during the source test will be

The **mid-level calibration gas** will be equivalent to 40 to 60 percent of the span. The mid-level calibration gas concentration to be used during the source test will be ______.

The **low-level calibration gas** will be less than 20 percent of the calibration span. A zero gas meeting the definition of "zero air material" in 40CFR72.2 may be used. The low-level gas concentration to be used during the source test will be ______.

The calibration gases will be SO_2 in N_2 , SO_2 in air or a calibration gas mixture, as indicated in Section 7.1 of the method. The calibration gases will be (check one):

- Certified within an uncertainty of 2.0 percent in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards" (EPA Protocol gases). If a zero gas is used for the low-level gas, it will meet the definition of "zero air material" in 40CFR72.2, as opposed to being an EPA Protocol gas.
- _____ Blended gas mixtures meeting the protocol above, provided that the additional gas components are shown not to interfere with the analysis.
- Calibration gases will be prepared from EPA Protocol gases using Method 205.
 Separate procedures for Method 205 will be included in the protocol. Note: Part 75 applications require EPA approval for the use of Method 205.

Analyzer Calibration

The **analyzer calibration error check** (or system calibration test for dilution systems) will be conducted by introducing the low-level, mid-level, and high-level gases to the analyzer (or through the entire measurement system, introduced at the probe, for a dilution system). During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow rate at the analyzer. The analyzer responses to each calibration gas will be recorded. The analyzer calibration error check will be considered invalid if the gas concentration displayed by the analyzer exceeds ± 2 percent of the span for any of the calibration gases, or > 0.5 ppmv absolute difference.

The **sampling system bias check** will be performed by introducing first an upscale gas (midrange or high-level, whichever more closely approximates the stack concentration) at the calibration valve assembly installed at the outlet of the sampling probe, and then the low-level gas. During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow at the analyzer. This check will be considered invalid if the difference between the calibration bias check and the calibration error check for the same calibration gas exceeds \pm 5% of the span, or > 0.5 ppmv absolute difference.

During the initial sampling system bias check, the **measurement system response time** is determined, as detailed in Sections 8.2.5 and 8.2.6 of Method 7E.

Documentation that an interference check has been conducted in accordance with Section 8.2.7 of Method 7E will be available on-site and will be included in the final test report. Any specific

technology, equipment or procedures intended to remove interference effects will be operating properly during the stack test. The Alternative Interference Check listed in Section 16.1 of Method 6C may be used instead.

Stratification Determination

Prior to sampling, or as part of the first test run, a stratification check must be performed in accordance with Section 8.1.2 of Method 7E. If more than one instrumental method that requires a stratification check is being performed, the stratification check need only be done on one of them. A stratification check is not required for stacks < 4 inches in diameter.

The stratification check will be conducted at either (1) twelve traverse points selected in a accordance with EPA Method 1, or (2) three traverse points spaced on a line passing through the centroidal area at 16.7, 50.0 and 83.3 percent of the measurement line. Each point will be sampled for a minimum of twice the response time.

The minimum number of traverse points required for sampling will be determined as follows. If each traverse point differs from the mean by no more than the least restrictive of:

Difference from mean	Stratification Class	Number of required sample
		points
<u>+</u> 5% or <u>+</u> 0.5 ppm	Unstratified	A single point that most closely
		matches the mean.
Between \pm 5% and \pm 10%, or between	Minimally stratified	Three (3) sample points spaced
± 0.5 ppm and ± 1.0 ppm		at 16.7, 50.0 and 83.3 percent
		of the measurement line. *
Greater than $\pm 10\%$ and greater than	Stratified	Twelve (12) sample points
<u>+</u> 1.0 ppm		located consistent with EPA
		Method 1 criteria.

* For a minimally stratified stack with an internal diameter greater than 2.4 meters (7.8 feet), the three sampling points may be located at 0.4 meters (1.3 feet), 1.0 meters (3.28 feet) and 2.0 meters (6.56 feet) along the measurement line showing the highest average concentration. This option will only be available if the stratification check consisted of twelve points.

Emission Measurement Test Procedure

The sampling probe will be placed at the first sample point and sampling will begin at the same rate used during the bias check. A constant rate \pm 10 percent will be maintained during the entire sample run. Sampling will commence only after twice the response time has elapsed. Sampling will be conducted for an equal length of time at each traverse point.

Immediately following the completion of the test period and hourly during the test period, the low-level calibration gas and an upscale calibration gas (the mid-level or high-level as appropriate) will be re-introduced one at a time to the measurement system at the calibration valve assembly. No adjustments to the measurement system will be made until both the low-level and upscale bias and drift checks are made. The analyzer response will be recorded. If the

bias values exceed the specified limits, the test results preceding the check will be invalidated and the test will be repeated following corrections to the measurement system and full recalibration. If the drift values exceed the specified limits, the run may be accepted but the test measurement system will be fully recalibrated and the results reported using Eqn. 7E-5b (Eqn. 7E-5a if a non-zero gas is used for the low-level calibration gas) of Method 7E.

Measurement System Performance Specifications

Drift, less than or equal to ± 3 percent of the span value for the low-level or upscale gas. **Sampling System Bias**, less than or equal to ± 5 percent of span value for the low-level or upscale gas. **Calibration Error**, less than or equal to ± 2 percent of span for each calibration gas.

An alternative acceptance criteria for each of these measurements is less than or equal to ± 0.5 ppmv absolute difference.

Emission Calculation

The average gas effluent concentration will be determined from the average gas concentration displayed by the gas analyzer and is adjusted for the zero and upscale sampling system bias checks, as determined in accordance with the procedures specified above. The average gas concentration displayed by the analyzer may be determined by averaging all of the effluent measurements indicated by the data acquisition system for the test run. The minimum frequency for data recording will be one-minute averages during the run. A chart recorder will only be used as a backup to the data acquisition system. The effluent gas concentration will be calculated using Eqn. 7E-5b (Eqn. 7E-5a if a non-zero gas is used for the low-level calibration gas) of Method 7E.

$$C_{gas} = (C_{avg} - C_o) \frac{C_{MA}}{C_M - C_o}$$
Eqn. 7E-5b

$$C_{gas} = (C_{avg} - C_{M}) \frac{C_{MA} - C_{OA}}{C_{M} - C_{O}} + C_{MA} \qquad Eqn. 7E-5a$$

Emissions will be presented in the following units:

Proposed Deviations from this BTS Template or the Method

(Insert any proposed deviations here)

METHOD 7E - DETERMINATION OF NITROGEN OXIDES EMISSIONS FROM STATIONARY SOURCES

Applicability and Principle

A sample is continuously extracted from the effluent stream and is conveyed to an instrumental analyzer for the determination of NOx concentrations. Performance specifications and test procedures are provided to ensure reliable data.

<u>Apparatus</u>

A **measurement system** for Nitrogen Oxides that meets the specifications in Section 13.0 of this method will be used. Analyzers operating on the principle of chemiluminescence, which includes a NO_2 to NO converter that converts NO_2 in the gas stream to NO, have been successfully used in the past. If the analyzer proposed is operated on a principle other than chemiluminescence, the details will be included at the end of this template in the protocol submittal. Dual range analyzers may be used when needed, provided that both ranges meet all the quality assurance requirements of this method. When an analyzer is routinely calibrated with a calibration span of less than or equal to 20 ppmv, the manufacturer's stability test (MST) is required (Table 7E-5 of the method) prior to testing and documentation will be included in the test report.

The **Sample Probe** will be glass, stainless steel, or equivalent, of sufficient length to traverse the sample points. The sampling probe shall be heated to prevent condensation.

The **Sample Line** will be heated (sufficient to prevent condensation) stainless steel or Teflon® tubing, to transport the sample gas to the moisture removal system.

The **Moisture Removal System** will be a refrigerator-type condenser or similar device to continuously remove condensate from the sample gas while maintaining minimal contact between the condensate and the sample gas. If a wet-basis system is being proposed, details will be included at the end of this template in the protocol submittal.

The **Sample Transport Lines** will be stainless steel or Teflon tubing, to transport the sample from the moisture removal system to the sample pump, sample flow rate control, and sample gas manifold.

A **Calibration Valve Assembly** with a three-way valve assembly, or equivalent, for introducing calibration gases either directly to the analyzer in direct calibration mode, or into the measurement system at the probe in system calibration mode, will be utilized. When in the system calibration mode, the system must be able to flood the sampling probe and vent excess gas.

A **Particulate Filter**, either an in-stack or heated (sufficient to prevent water condensation) out-ofstack filter will be used. All filters will be fabricated of materials that are nonreactive to the gas being sampled. If an out-of-stack filter is used, it will be included in the system bias test.

A **Leak-free Pump**, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system will be used. The pump will be constructed of any material that is nonreactive to the gas being sampled.

The **Recorder** will be a computerized data acquisition system, digital recorder or data logger used for recording measurement data. The minimum data-recording requirement is one measurement value per minute. A strip-chart recorder can only be used as a back-up system.

Analytical Span and Calibration Gases

The span of the monitoring system is equivalent to the high-level calibration gas value (see below) and will be selected such that to the extent practicable, the measured emissions will be between 20 - 100% of the selected calibration span. In practical terms, the span is selected such that a pollutant gas concentration equivalent to the emission <u>standard</u> is not less than 30% of the span. If at any time during the run the measured gas concentration exceeds the readable range of the analyzer, the run may be considered invalid. If the average of any run exceeds the calibration span value, the run is invalid.

Allowable = _____ lb/hr MW = 46 lb/lbmole Stack Flow = _____ dscfm

The in-stack concentration based on the emission standard and stack flow parameters is

 $\frac{(\text{lb/hr}) \times (387 \times 10^6)}{(\text{MW}) \times (\text{dscfm}) \times 60} = \underline{\qquad} \text{ppm.}$

Note: when actual concentrations differ significantly from the standard, the span may need to be modified accordingly, such that the method criteria for span selection are met. Determining the emission standard is the first step in approximating the necessary span.

The **high-level calibration gas sets the calibration span** and results in measurements being (to the extent practicable) equivalent to 20 to 100 percent of the span. Based on the calculation above, the high-level gas (span) concentration to be used during the source test will be

The **mid-level calibration gas** will be equivalent to 40 to 60 percent of the span. The mid-level calibration gas concentration to be used during the source test will be _____.

The **low-level calibration gas** will be less than 20 percent of the calibration span. A zero gas meeting the definition of "zero air material" in 40CFR72.2 may be used. The low-level gas concentration to be used during the source test will be ______.

The calibration gases will be NO in N₂. The calibration gases will be (check one):

- Certified within an uncertainty of 2.0 percent in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards" (EPA Protocol gases). If a zero gas is used for the low-level gas, it will meet the definition of "zero air material" in 40CFR72.2, as opposed to being an EPA Protocol gas.
- _____ Blended gas mixtures meeting the protocol above, provided that the additional gas components are shown not to interfere with the analysis.

Calibration gases will be prepared from EPA Protocol gases using Method 205. Separate procedures for Method 205 will be included in the protocol. Note: Part 75 applications require EPA approval for the use of Method 205.

Analyzer Calibration

The **analyzer calibration error check** (or system calibration test for dilution systems) will be conducted by introducing the low-level, mid-level, and high-level gases to the analyzer (or through the entire measurement system, introduced at the probe, for a dilution system). During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow rate at the analyzer. The analyzer responses to each calibration gas will be recorded. The analyzer calibration error check will be considered invalid if the gas concentration displayed by the analyzer exceeds ± 2 percent of the span for any of the calibration gases, or > 0.5 ppmv absolute difference.

The **sampling system bias check** will be performed by introducing first an upscale gas (midrange or high-level, whichever more closely approximates the stack concentration) at the calibration valve assembly installed at the outlet of the sampling probe, and then the low-level gas. During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow at the analyzer. This check will be considered invalid if the difference between the calibration bias check and the calibration error check for the same calibration gas exceeds \pm 5% of the span, or > 0.5 ppmv absolute difference.

During the initial sampling system bias check, the **measurement system response time** is determined, as detailed in Sections 8.2.5 and 8.2.6 of the method.

If the measurement system being used converts NO_2 to NO prior to analyzing for NOx, a NO_2 to NO converter efficiency test will be conducted as part of this test program in accordance with either;

(1) Section 8.2.4.1 (NO₂ cylinder procedure), or

(2) Section 16.2.1 (NOx generator procedure), or

(3) Section 16.2.2 (Tedlar bag procedure using NO calibration gas).

The procedure to be utilized in this test program will be _____

A NOx converter efficiency of \geq 90% is required for Options 1 or 2 above, calculated by Eqn. 7E-7. For Option 3 above, the final NOx reading must not drop more than 2% of the peak value measured to be considered acceptable, as calculated by Eqn. 7E-9. The NOx converter efficiency check will be conducted on each day of testing when the test consists of multiple test dates. Alternatively, the NOx converter check will be conducted, at a minimum, prior to the start of testing on the first day, and at the conclusion of the last day of testing. Should the final NOx converter check fail to meet acceptance criteria, all data since the last passing converter efficiency check will be considered invalid.

Documentation that an interference check has been conducted in accordance with Section 8.2.7 of the method will be available on-site and will be included in the final test report. Any specific technology, equipment or procedures intended to remove interference effects will be operating properly during the stack test.

Stratification Determination

Prior to sampling, or as part of the first test run, a stratification check must be performed in accordance with Section 8.1.2 of the method. If more than one instrumental method that requires a stratification check is being performed, the stratification check need only be done on one of them. A stratification check is not required for stacks < 4 inches in diameter.

The stratification check will be conducted at either (1) twelve traverse points selected in a accordance with EPA Method 1, or (2) three traverse points spaced on a line passing through the centroidal area at 16.7, 50.0 and 83.3 percent of the measurement line. Each point will be sampled for a minimum of twice the response time.

The minimum number of traverse points required for sampling will be determined as follows. If each traverse point differs from the mean by no more than the least restrictive of:

Difference from mean	Stratification Class	Number of required sample
		points
\pm 5% or \pm 0.5 ppm	Unstratified	A single point that most closely
		matches the mean.
Between \pm 5% and \pm 10%, or between	Minimally stratified	Three (3) sample points spaced
± 0.5 ppm and ± 1.0 ppm		at 16.7, 50.0 and 83.3 percent
		of the measurement line. *
Greater than \pm 10% and greater than	Stratified	Twelve (12) sample points
<u>+</u> 1.0 ppm		located consistent with EPA
		Method 1 criteria.

* For a minimally stratified stack with an internal diameter greater than 2.4 meters (7.8 feet), the three sampling points may be located at 0.4 meters (1.3 feet), 1.0 meters (3.28 feet) and 2.0 meters (6.56 feet) along the measurement line showing the highest average concentration. This option will only be available if the stratification check consisted of twelve points.

Emission Measurement Test Procedure

The sampling probe will be placed at the first sample point and sampling will begin at the same rate used during the bias check. A constant rate ± 10 percent will be maintained during the entire sample run. Sampling will commence only after twice the response time has elapsed. Sampling will be conducted for an equal length of time at each traverse point.

Immediately following the completion of the test period and hourly during the test period, the low-level calibration gas and an upscale calibration gas (the mid-level or high-level as appropriate) will be re-introduced one at a time to the measurement system at the calibration

valve assembly. No adjustments to the measurement system will be made until both the lowlevel and upscale bias and drift checks are made. The analyzer response will be recorded. If the bias values exceed the specified limits, the test results preceding the check will be invalidated and the test will be repeated following corrections to the measurement system and full recalibration. If the drift values exceed the specified limits, the run may be accepted but the test measurement system will be fully recalibrated and the results reported using Eqn. 7E-5b (Eqn. 7E-5a if a non-zero gas is used for the low-level calibration gas).

Measurement System Performance Specifications

Drift, less than or equal to ± 3 percent of the span value for the low-level or upscale gas. **Sampling System Bias**, less than or equal to ± 5 percent of span value for the low-level or upscale gas. **Calibration Error**, less than or equal to ± 2 percent of span for each calibration gas.

An alternative acceptance criteria for each of these measurements is less than or equal to ± 0.5 ppmv absolute difference.

Emission Calculation

The average gas effluent concentration will be determined from the average gas concentration displayed by the gas analyzer and is adjusted for the zero and upscale sampling system bias checks, as determined in accordance with the procedures specified above. The average gas concentration displayed by the analyzer may be determined by averaging all of the effluent measurements indicated by the data acquisition system for the test run. The minimum frequency for data recording will be one-minute averages during the run. A chart recorder will only be used as a backup to the data acquisition system. The effluent gas concentration will be calculated using Eqn. 7E-5b (Eqn. 7E-5a if a non-zero gas is used for the low-level calibration gas).

$$C_{gas} = (C_{avg} - C_o) \frac{C_{MA}}{C_M - C_o}$$
 Eqn. 7E-5b

$$C_{gas} = (C_{avg} - C_M) \frac{C_{MA} - C_{OA}}{C_M - C_O} + C_{MA} \qquad Eqn. 7E-5a$$

Emissions will be presented in the following units:

Proposed Deviations from this BTS Template or the Method

(Insert any proposed deviations here)

METHOD 8 – DETERMINATION OF SULFURIC ACID MIST AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

Applicability

This method is applicable to the determination of sulfuric acid (including sulfuric acid mist and sulfur trioxide) and gaseous sulfur dioxide emissions from stationary sources.

Principle

A stack sample is withdrawn isokinetically from the stack. The sulfuric acid mist (including sulfur trioxide) and sulfur dioxide are separated and both fractions are measured separately by barium-thorin titration.

Interferences

Possible interferents are free ammonia, dimethyl aniline and fluorides. If free ammonia is present, alternate procedures must be used (see Special Situations Section).

Based on the above we (appropriate box checked):

_____ Do not expect any interference.

_____ Do expect interference. The description and discussion of the anticipated interference follows.

In-Stack Detection Limits & Sample Times

The minimum detection limit of the method has been determined to be 0.05 mg/m^3 ($0.03\text{E}-07 \text{ lb/ft}^3$) for H₂SO₄/SO₃ and 1.2 mg/m³ ($0.75\text{E}-07 \text{ lb/ft}^3$) for SO₂ for a standard 60-minute sample. Actual in-stack method detection limits (ISDL) are based on actual source sampling parameters and analytical results. Actual detection limits can be improved through increased stack gas sampled (sample time). For this source, the in-stack concentration is:

Allowable(s) = _____ lbs/hr (SO₂), and _____ lbs/hr (H₂SO₄) Stack flow = _____ dscfm

 $lb/ft^3 = (lb / hr) / (dscfm x 60) = _____ lb/ft^3 (SO_2), and _____ lbs/hr (H_2SO_4)$

Therefore, the sample time will be ______ minutes.

Sample Train & Recovery Components & Supplies

A schematic of the sampling train is shown in Figure 8-1 of the method. Specifically, the sampling train will be constructed with components specified under EPA Method 8, Section 6.0, (similar to a Method 5 train), with the following exceptions and/or highlights.

Sample Train

- 1) **Probe liner** will be constructed of Borosilicate or quartz glass and heated.
- 2) **Filter Holder** will be borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other gasket materials (*e.g.*, Teflon or Viton) may be used, subject to BTS approval. The holder design will provide a positive seal against leakage from the outside or around the filter. The filter holder will be placed between the first and second impingers and be unheated.
- 3) The **impinger train** will consist of four Greenburg-Smith impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. Silicone grease may be used if necessary. The first and third impingers must have the standard tips and the second and fourth will be modified to be non-restricted. The first impinger contains 100 ml 80% isopropanol (IPA), 100 ml of 3% H₂O₂ in the second and third impingers and about 200 g of silica gel in the fourth impinger.
- 4) **Pump** leak-free diaphragm pump, or equivalent, with a small surge tank between the pump and rate meter.
- 5) **Dry Gas Meter** (DGM) sufficiently accurate to measure the sample volume to within 2 percent, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature sensor (dial thermometer, or equivalent) capable of measuring temperature accurately to within 3 °C (5.4 °F).
- 6) A **nozzle**, **pitot tube**, **differential pressure gauge** and **metering system** as described in Method 5.
- 7) A Method 8 train may be combined with a particulate matter train (see Special Situations Section).

Sample Recovery

- 1) Wash bottles (glass or polyethylene), two 500 ml.
- 2) **Storage bottles** (polyethylene), two 1000 ml per sample run.
- 3) **Graduated cylinders**, 250 ml and 1 liter.
- 4) **Trip balance** (for moisture determination), accurate to ± 0.5 g.

<u>Sampling</u>

Pre-test leak checks and post-test leak checks will be conducted by following the same basic procedure in Method 5, Section 8.4.2 noting that the probe heater will be adjusted to the minimum temperature to prevent condensation and adjusting the temperature upward if condensation is observed.

The sampling train will be assembled as indicated above. Crushed ice and water will be placed around the impingers. The initial DGM reading and barometric pressure will be recorded. Sample isokinetically, following the general procedures given in Method 5, Section 8.5. For each run, the required data should be recorded on a data sheet such as the one shown in Method 5, Figure 5-3. Maintain sampling rate at or below $0.030 \text{ m}^3/\text{min}$ (1.0 cfm) during the run. Periodically during the test, observe the connecting line between the probe and first impinger for signs of condensation. If condensation does occur, adjust the probe heater setting upward to the minimum temperature required to prevent condensation.

Post-Test Purge

At the conclusion of an acceptable post leak check, remove the probe and purge the remainder of the sampling train with clean ambient air (optionally passed through a charcoal filter) for 15 minutes at the average sampling rate during the test run.

Sample Recovery

Container No. 1

The first impinger (plus contents) will be cleaned and weighed to the nearest 0.5 g, and the weight recorded for the moisture determination. The contents of the first impinger will be transferred to a 250-ml graduated cylinder. The probe, first impinger, all connecting glassware before the filter, and the front half of the filter holder will be rinsed with 80 percent IPA. The IPA rinse solution will be added to the graduated cylinder. The contents of the graduated cylinder will be diluted to 225 ml with 80 percent IPA, and the cylinder contents transferred to the storage container. The graduated cylinder will be rinsed with 25 ml of 80 percent IPA, and the rinse transferred to the same storage container. The filter will be added to the solution in the storage container and the container will be mixed. The container will be sealed to protect the solution against evaporation. The container will be identified.

Container No. 2

The second and third impingers (plus contents) will be cleaned and weighed to the nearest 0.5 g, and the weights recorded for moisture determination. Likewise, the spent silica gel (or silica gel plus impinger) will be weighed to the nearest 0.5 g, and the weight recorded for moisture determination. The solutions from the second and third impingers will be transferred to a 1-liter graduated cylinder. All connecting glassware (including back half of filter holder) between the filter and silica gel impinger will be rinsed with water, and this rinse water will be added to the graduated cylinder. The contents of the graduated cylinder will be diluted to 950 ml with water, and the cylinder contents will be transferred to a storage container. The graduated cylinder will be rinsed with 50 ml of water, and the rinse transferred to the storage container. The container will be sealed, the level of liquid on the container will be marked, and the sample container will be identified.

Sample Preparation & Analysis

The level of the fluid in the container will be noted. If significant leakage occurred, either the run will be voided or methods will be used to correct the results, subject to BTS approval.

Container No. 1

The container holding the IPA solution and the filter will be shaken. If the filter breaks up, the fragments will be allowed to settle for a few minutes before removing a sample aliquot. A 100-ml aliquot of this solution will be pipetted into a 250-ml Erlenmeyer flask, 2 to 4 drops of thorin indicator will be added, then titrated to a pink endpoint using 0.0100 N barium standard solution. The titration will be repeated with a second aliquot of sample, and the titration values averaged. Replicate titrations will agree to within 1 percent or 0.2 ml, whichever is greater.

Container No. 2

The solution in the container holding the contents of the second and third impingers will be thoroughly mixed. A 10-ml aliquot of sample will be pipetted into a 250-ml Erlenmeyer flask. 40 ml of 100 percent IPA and 2 to 4 drops of thorin indicator will be added, then titrated to a pink endpoint using 0.0100 N barium standard solution. The titration will be repeated with a second aliquot of sample, and the titration values averaged. Replicate titrations will agree to within 1 percent or 0.2 ml, whichever is greater.

Blanks

Two blanks are required. The first blank will be 100 ml of 80 percent IPA. 2 to 4 drops of thorin indicator will be added, then titrated as indicated above for Container No. 1.

The second blank will be prepared by combining 2 ml of 3% H_2O_2 , 8 ml of water and 40 ml of 100 percent IPA. 2 to 4 drops of thorin indicator will be added, then titrated as indicated above for Container No. 2.

Calculations

All calculations will be performed as per Method 8, including blank corrections. Please note that the correct Vsoln values in Section 12.1 of the method should be 1000 ml for SO_2 and 250 ml for H₂SO₄. Detailed sample calculations will be included in the final report.

Emissions will be presented in the following units: ______.

Audit Samples

If provided, audit samples will be analyzed consistent with Section 11.3 of the method and the results will be provided in the final test report. It is recognized that failure to achieve method acceptance criteria for the audit could result in the requirement to repeat the stack test program

Proposed Deviations from this BTS Template or the Method

(Insert any proposed deviations here)

<u>Special Situations Section</u> – If either of these two situations is relevant, they will be checked in the checkbox located prior to the procedure.

(_) 1. Alternative Procedures for Method 8 when Ammonia is Present:

Sampling Procedures

SOx must be determined using Method 8, utilizing the sampling procedures specified in Section 16.3.1 of Method 6, which are as follows:

The probe will be maintained at $275 \,^{\circ}$ C ($527 \,^{\circ}$ F) and equipped with a high-efficiency instack filter (glass fiber) to remove particulate matter. The filter material will be unreactive to SO₂. Whatman 934AH (formerly Reeve Angel 934AH) filters treated as described in Reference 10 in Section 17.0 of Method 5 is an example of a filter that has been shown to work. Where alkaline particulate matter and condensed moisture are present in the gas stream, the filter will be heated above the moisture dew point but below $225 \,^{\circ}$ C ($437 \,^{\circ}$ F)."

Procedure for analysis of Container #1

Same as described above.

Procedure for analysis of Container #2

The SO₂ analysis of Container #2 is analyzed per Section 11.2.2 of Method 8 (including replicate titrations), except add 0.5 ml of 0.1N HCl prior to adding the indicator.

The SO_2 concentration determined from Container # 1 is summed with the SO_2 concentration from Container #2 to determine the total SO_2 concentration.

(_) 2. Combining the Method 8 train with the Particulate Matter Sampling Train:

EPA Method 8 allows for determination of particulate in conjunction with SO_x . However, if these procedures are followed the H_2SO_4 mist does not get measured as part of the SO_x .

To address these concerns, the following procedures should be employed.

SAMPLE TRAIN

- 1. A heated glass fiber filter is added to the Method 8 sampling train between the probe and the IPA impinger. This filter is maintained at the temperature specified by NJATM1 and is in addition to (not a replacement for) the filter between the 1st and 2nd impingers called for in Method 8.
- 2. The impingers are as described in Method 8.
- 3. Moisture determination must be made by weight, not volume. The impingers (plus absorbing solutions) and the weight of the silica gel (or silica gel plus

container) must be weighed to the nearest 0.5 g and recorded prior to testing. These items are weighed again at the end of the test to determine the moisture content, prior to sample recovery.

SAMPLE RECOVERY

- 1. The heated filter is recovered per NJATM1 (CONTAINER 1).
- 2. The probe nozzle, fitting, liner and front half of filter holder are recovered with acetone into a container (CONTAINER 2), per NJATM1.
- 3. The above components are then recovered with IPA into a container, with the contents of the first impinger and impinger rinses per Method 8 (CONTAINER 3). The remaining sample recovery continues per Method 8 (CONTAINER 2 from Method 8 becomes CONTAINER 4).
- 4. The post-test 15 minute purge (after a successful leak check) is conducted after removal of the probe AND the heated particulate filter assembly, through the rest of the sample train.

ANALYSIS

- 1. The filter and acetone probe wash are analyzed for particulate per NJATM1.
- 2. The acetone residue is solubilized with the IPA wash (CONTAINER 2). The particulate filter is then added to this container. Analysis for SO_x continues per Method 8.

METHOD 10 - DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

Applicability and Principle

Principle. An integrated or continuous gas sample is extracted from a sampling point and analyzed for carbon monoxide (CO) content. Performance specifications and test procedures are provided to ensure reliable data.

Applicability. This method is applicable for the determination of carbon monoxide emissions from stationary sources. The process will dictate whether a continuous or an integrated sample is required. If the process produces CO spikes that would exceed the span (as determined from the allowable), then an integrated procedure is required.

Interferences

Any substance having a strong absorption of infrared energy will interfere to some extent, for example water and carbon dioxide. The use of silica gel and ascarite traps will alleviate the major interference problems. The measured gas volume will be corrected if these traps are used employing Equation 10-1 listed in Section 12.1 of the method.

Alternatively, the use of GFC NDIR will alleviate this need.

In the sections that follow, the proposed Apparatus, Analytical Method and Sampling Procedure to be used will be noted.

Apparatus (check one)

A **measurement system** for Carbon Monoxide that meets the specifications in Section 13.0 of this method will be used.

The **Sample Probe** will be of sufficient length to traverse the sample points. The sampling probe shall be heated to prevent condensation if also being used to measure SO_2 and/or NOx.

The **Sample Line** will transport the sample gas to the moisture removal system and be heated (sufficient to prevent condensation) if also being used to measure SO_2 and/or NOx.

The **Moisture Removal System** will be a refrigerator-type condenser or similar device to continuously remove condensate from the sample gas while maintaining minimal contact between the condensate and the sample gas. If a wet-basis system is being proposed, details will be included at the end of this template in the protocol submittal.

The **Sample Transport Lines** will transport the sample from the moisture removal system to the sample pump, sample flow rate control, and sample gas manifold.

A **Calibration Valve Assembly** with a three-way valve assembly, or equivalent, for introducing calibration gases either directly to the analyzer in direct calibration mode, or into the measurement system at the probe in system calibration mode, will be utilized. When in the system calibration mode, the system must be able to flood the sampling probe and vent excess gas.

A **Particulate Filter**, either an in-stack or heated (sufficient to prevent water condensation) out-of-stack filter will be used. If an out-of-stack filter is used, it will be included in the system bias test.

A **Leak-free Pump**, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system will be used. The pump will be constructed of any material that is nonreactive to the gas being sampled.

A **Recorder**, a strip-chart recorder, analog computer, or digital recorder for recording measurement data. The minimum data recording requirement is one measurement value per minute. A strip-chart recorder can only be used as a back-up system.

____ Integrated Sample

Probe. Same as above.

Air-Cooled Condenser or Equivalent. To remove any excess moisture.

Valve. Needle valve, or equivalent, to adjust flow rate.

Pump. Leak-free diaphragm type, or equivalent, to transport gas.

Rate Meter. Rotameter, or equivalent, to measure a flow range from 0 to 1.0 liter per minute (0 to 0.035 cfm).

Flexible Bag. Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 ft^3). Leak-test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete, there should be no flow through the meter.

Analysis (check one)

A measurement system for Carbon Monoxide that meets the specifications in Section 13.0 of this method will be used. Dual range analyzers may be used when needed, provided that both ranges meet all the quality assurance requirements of this method. When an analyzer is routinely calibrated with a calibration span of less than or equal to 20 ppmv, the manufacturer's stability test (MST) is required (Table 7E-5 of Method 7E) prior to testing and documentation will be included in the test report. Instruments using a Luft-type nondispersive infrared analyzer (NDIR) or gas filter correlation (GFC) NDIR have successfully been used in the past. The instrument to be used will be:

____NDIR Carbon Monoxide Analyzer. Nondispersive infrared spectrometer, or equivalent.

Silica Gel and Ascarite Traps (check one)

_____ will be used as detailed in the method.

_____ will not be used **because**:

GFC Carbon Monoxide Analyzer. Gas filter correlation NDIR analyzer.

If the analyzer proposed is operated on a principle other than one of the above, the details will be included at the end of this template in the protocol submittal.

Analytical Span and Calibration Gases

The span of the monitoring system is equivalent to the high-level calibration gas value (see below) and will be selected such that to the extent practicable, the measured emissions will be between 20 - 100% of the selected calibration span. In practical terms, the span is selected such that a pollutant gas concentration equivalent to the emission standard is not less than 30% of the span. If at any time during the run the measured gas concentration exceeds the readable range of the analyzer, the run may be considered invalid. If the average of any run exceeds the calibration span value, the run is invalid.

Allowable = _____ lb/hr Stack Flow = _____ dscfm MW = 28 lb/lbmole

The in-stack concentration based on the emission standard and stack flow parameters is

 $\frac{(\text{lb/hr}) \times (387 \times 10^6)}{(\text{MW}) \times (\text{dscfm}) \times 60} = \underline{\qquad} \text{ppm.}$

Note: when actual concentrations differ significantly from the standard, the span may need to be modified accordingly, such that the method criteria for span selection are met. Determining the emission standard is the first step in approximating the necessary span.

The **high-level calibration gas sets the calibration span** and results in measurements being (to the extent practicable) equivalent to 20 to 100 percent of the span. Based on the calculation above, the high-level gas (span) concentration to be used during the source test will be ______.

The **mid-level calibration gas** will be equivalent to 40 to 60 percent of the span. The mid-level calibration gas concentration to be used during the source test will be

_____•

The **low-level calibration gas** will be less than 20 percent of the calibration span. A zero gas meeting the definition of "zero air material" in 40CFR72.2 may be used. The low-level gas concentration to be used during the source test will be ______.

The calibration gases will be CO in N₂. The calibration gases will be (check one):

- Certified within an uncertainty of 2.0 percent in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards" (EPA Protocol gases). If a zero gas is used for the low-level gas, it will meet the definition of "zero air material" in 40CFR72.2, as opposed to being an EPA Protocol gas.
- _____Blended gas mixtures meeting the protocol above, provided that the additional gas components are shown not to interfere with the analysis.
- Calibration gases will be prepared from EPA Protocol gases using Method 205. Separate procedures for Method 205 will be included in the protocol. Note: Part 75 applications require EPA approval for the use of Method 205.

Analyzer Calibration

The **analyzer calibration error check** (or system calibration test for dilution systems) will be conducted by introducing the low-level, mid-level, and high-level gases to the analyzer (or through the entire measurement system, introduced at the probe, for a dilution system). During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow rate at the analyzer. The analyzer responses to each calibration gas will be recorded. The analyzer calibration error check will be considered invalid if the gas concentration displayed by the analyzer exceeds ± 2 percent of the span for any of the calibration gases, or > 0.5 ppmv absolute difference.

The **sampling system bias check** will be performed by introducing first an upscale gas (mid-range or high-level, whichever more closely approximates the stack concentration) at the calibration valve assembly installed at the outlet of the sampling probe, and then the low-level gas. During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow at the analyzer. This check will be considered invalid if the difference between the calibration bias check and the calibration error check for the same calibration gas exceeds \pm 5% of the span, or > 0.5 ppmv absolute difference.

During the initial sampling system bias check, the **measurement system response time** is determined, as detailed in Sections 8.2.5 and 8.2.6 of Method 7E.

Documentation that an interference check has been conducted in accordance with Section 8.2.7 of Method 7E will be available on-site and will be included in the final test report. Any specific technology, equipment or procedures intended to remove interference effects will be operating properly during the stack test. The Alternative Interference Check listed in Section 16.1 of Method 6C may be used instead.

Stratification Determination

Prior to sampling, or as part of the first test run, a stratification check must be performed in accordance with Section 8.1.2 of Method 7E. If more than one instrumental method that requires a stratification check is being performed, the stratification check need only be done on one of them. A stratification check is not required for stacks < 4 inches in diameter.

The stratification check will be conducted at either (1) twelve traverse points selected in a accordance with EPA Method 1, or (2) three traverse points spaced on a line passing through the centroidal area at 16.7, 50.0 and 83.3 percent of the measurement line. Each point will be sampled for a minimum of twice the response time.

The minimum number of traverse points required for sampling will be determined as follows. If each traverse point differs from the mean by no more than the least restrictive of:

Difference from mean	Stratification Class	Number of required sample
		points
\pm 5% or \pm 0.5 ppm	Unstratified	A single point that most closely
		matches the mean.
Between \pm 5% and \pm 10%, or between	Minimally stratified	Three (3) sample points spaced
± 0.5 ppm and ± 1.0 ppm		at 16.7, 50.0 and 83.3 percent
		of the measurement line. *
Greater than $\pm 10\%$ and greater than	Stratified	Twelve (12) sample points
<u>+</u> 1.0 ppm		located consistent with EPA
		Method 1 criteria.

* For a minimally stratified stack with an internal diameter greater than 2.4 meters (7.8 feet), the three sampling points may be located at 0.4 meters (1.3 feet), 1.0 meters (3.28 feet) and 2.0 meters (6.56 feet) along the measurement line showing the highest average concentration. This option will only be available if the stratification check consisted of twelve points.

Emission Measurement Test Procedure

The sampling probe will be placed at the first sample point and sampling will begin at the same rate used during the bias check. A constant rate \pm 10 percent will be maintained during the entire sample run. Sampling will commence only after twice the response time has elapsed. Sampling will be conducted for an equal length of time at each traverse point.

Immediately following the completion of the test period and hourly during the test period, the low-level calibration gas and an upscale calibration gas (the mid-level or high-level as appropriate) will be re-introduced one at a time to the measurement system at the calibration valve assembly. No adjustments to the measurement system will be made until both the low-level and upscale bias and drift checks are made. The analyzer response will be recorded. If the bias values exceed the specified limits, the test results preceding the check will be invalidated and the test will be repeated following corrections to the measurement system and full recalibration. If the drift values exceed the specified limits, the run may be accepted but the test measurement system will be fully recalibrated and the results reported using Eqn. 7E-5b (Eqn. 7E-5a if a non-zero gas is used for the low-level calibration gas) of Method 7E.

Measurement System Performance Specifications

Drift, less than or equal to ± 3 percent of the span value for the low-level or upscale gas. **Sampling System Bias**, less than or equal to ± 5 percent of span value for the low-level or upscale gas. **Calibration Error**, less than or equal to ± 2 percent of span for each calibration gas.

An alternative acceptance criteria for each of these measurements is less than or equal to ± 0.5 ppmv absolute difference.

Sampling Procedure (check one)

_____ Continuous Sampling. See above.

Integrated Sampling. The flexible bag will be evacuated. The equipment will be set up with the bag disconnected. The probe will be placed in the stack, and the sampling line purged. The bag will be connected, making sure that all connections are leak free. The sampling rate will be proportional to the stack velocity. CO₂

content of the gas may be determined by using the Method 3 integrated sample procedures, or by weighing the ascarite CO_2 concentration from the gas volume sampled and the weight gain of the tube, if applicable.

Emission Calculation

The average gas effluent concentration will be determined from the average gas concentration displayed by the gas analyzer and is adjusted for the zero and upscale sampling system bias checks, as determined in accordance with the procedures specified above. The average gas concentration displayed by the analyzer may be determined by averaging all of the effluent measurements indicated by the data acquisition system for the test run. The minimum frequency for data recording will be one-minute averages during the run. A chart recorder will only be used as a backup to the data acquisition system. The effluent gas concentration will be calculated using Eqn. 7E-5b (Eqn. 7E-5a if a non-zero gas is used for the low-level calibration gas) of Method 7E.

$$C_{gas} = (C_{avg} - C_o) \frac{C_{MA}}{C_M - C_o}$$
Eqn. 7E-5b

Cgas = (Cavg - CM)
$$\frac{C_{MA} - C_{OA}}{C_M - C_O} + C_{MA}$$
 Eqn. 7E-5a

Emissions will be presented in the following units:

Proposed Deviations from this BTS Template or the Method

(Insert any proposed deviations here)

METHOD 25A - DETERMINATION OF TOTAL GASEOUS ORGANIC CONCENTRATION USING A FLAME IONIZATION ANALYZER

Applicability

This method is applicable for the determination of total gaseous organic concentration of vapors, **including methane**, consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons). The emissions are **expressed in terms of methane** unless the **Permit** states otherwise or the emissions can be expressed in terms of the specific VOC emitted.

Summary of Method

A gas sample is extracted from the source through a heated sample line and glass fiber filter to a flame ionization analyzer (FIA). Results are reported as volume concentration equivalents of the calibration gas. If the calibration gases are not methane, results will be corrected to a methane basis by use of a response factor, unless the **Permit** limit states a basis other than methane or the emissions can be expressed in terms of the specific VOC emitted.

Apparatus

Measurement System. Any measurement system for total organic concentration that meets the specifications of this method. All sampling components leading to the analyzer will be heated

 \geq 110°C (220°F) throughout the sampling period, unless safety reasons are cited.

Organic Concentration Analyzer. A flame ionization analyzer (FIA) capable of meeting or exceeding the specifications of this method. The flame ionization detector block will be heated >120°C (250°F).

Sample Probe. Stainless steel, or equivalent, three-hole rake type. Sample holes will be 4 mm (0.16-in.) in diameter or smaller and located at 16.7, 50, and 83.3 percent of the equivalent stack diameter. Alternatively, a single opening probe may be used so that a gas sample is collected from the centrally located 10 percent area of the stack cross-section.

Heated Sample Line. Stainless steel or Teflon® tubing to transport the sample gas to the analyzer. The sample line should be heated (≥ 110 °C) to prevent any condensation.

Calibration Valve Assembly. A three-way valve assembly to direct the zero and calibration gases to the analyzers is recommended. Other methods, such as quick-connect lines, to route calibration gas to the analyzers are acceptable.

Particulate Filter. An in-stack or an out-of-stack glass fiber filter is required except as explained and justified in the Proposed Deviation from the Method Section. An out-of-stack filter should be heated to prevent any condensation.

Recorder. A strip-chart recorder, analog computer, or digital recorder for recording measurement data. The minimum data recording requirement is one measurement value per minute.

Analytical Range

The span value should be between 1.5 to 2.5 times the applicable in-stack concentration based on the emission limit.

Allowable = _____ lb/hr MW = _____ lb/lbmole Stack Flow = _____ scfm

The in-stack concentration based on the emission standard and stack flow parameters is

 $\frac{(\text{lb/hr}) \times (387 \times 10^6)}{(\text{MW}) \times (\text{scfm}) \times 60} = \underline{\qquad} \text{ppm. Therefore, the analyzer span will be } \underline{\qquad} \text{ppm.}$

Calibration Gases

The calibration gases for the gas analyzer will be methane in air or methane in nitrogen. Alternatively, organic compounds other than methane can be used; the appropriate corrections for response factor must be made. The calibration gases will be _______. Calibration gases will be EPA Protocol No. 1 gases and will include a recommended shelf-life from the manufacturer over which the concentration does not change more than $\pm 2\%$ from the certified value.

Zero Gas. High purity air with less than 0.1 part per million by volume (ppmv) of organic material (methane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

Low-level Calibration Gas. An organic calibration gas with a concentration equivalent to 25 to 35 percent of the applicable span value. The low-level gas to be used during the source test will be ______.

Mid-level Calibration Gas. An organic calibration gas with a concentration equivalent to 45 to 55 percent of the applicable span value. The mid-level gas to be used during the source test will be ______.

High-level Calibration Gas. An organic calibration gas with a concentration equivalent to 80 to 90 percent of the applicable span value. The high-level gas to be used during the source test will be ______.

Analyzer Calibration

The calibration procedures described in Method 7E (calibration error, bias and drift) will be followed unless deemed not feasible and approved by the on-site BTS observer.

The analyzer calibration error check will be conducted by introducing the zero, lowrange, mid-range, and high-range gases to the analyzer. During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow rate at the analyzer. The analyzer responses to each calibration gas will be recorded. The analyzer calibration error check will be considered invalid if the gas concentration displayed by the analyzer exceeds ± 2 percent of the span for any of the calibration gases.

The sampling system bias check will be performed by introducing first an upscale gas (mid-range) at the calibration valve assembly installed at the outlet of the sampling probe, and then the zero gas. During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow at the analyzer. This check will be considered invalid if the difference between the calibration error check and the calibration bias check for the same calibration gas exceeds \pm 5% of the span.

Alternatively, if approved by the BTS observer, the following calibration error test will be performed. Immediately prior to the test series (within 2 hours of the start of the test), zero gas and high-level calibration gas will be introduced at the calibration valve assembly. The analyzer output will be adjusted to the appropriate levels, if necessary. The predicted response will be calculated for the low-level and mid-level gases based on a linear response line between the zero and high-level response. Then the low-level and mid-level calibration gases will be introduced successively to the measurement system. The analyzer responses will be recorded for low-level and mid-level calibration gases and the differences between the measurement system responses and the predicted responses determined. These differences must be less than 5 percent of the respective calibration gas value. If not, the measurement system is not acceptable and will be replaced or repaired prior to testing. No adjustments to the measurement system will be made after the calibration and before the drift check. If adjustments are necessary before the completion of the test series, drift checks will be performed prior to the required adjustments and the calibration following the adjustments repeated. If multiple electronic ranges are to be used, each additional range will be checked with a mid-level calibration gas to verify the multiplication factor.

Response Time Test. Zero gas will be introduced into the measurement system at the calibration valve assembly. When the system output has stabilized, switch quickly to the high-level calibration gas. The time will be recorded from the concentration change to the measurement system response equivalent to 95 percent of the step change. The test will be repeated three times and the results averaged.

Emission Measurement Test Procedure

Organic Measurement. Sampling will begin at the start of the test period at a sample point that is centrally located in the stack. The time and any required process information will be recorded, as appropriate, in particular, noting on the recording chart, periods of process interruption or cyclic operation.

Bias and Drift Determination. Immediately following the completion of the test period and hourly during the test period, the zero and mid-level calibration gases will be reintroduced one at a time to the measurement system at the calibration valve assembly. No adjustments to the measurement system will be made until both the zero and calibration bias and drift checks are made. The analyzer response will be recorded. If the bias values exceed the specified limits, the test results preceding the check will be invalidated and the test will be repeated following corrections to the measurement system and full recalibration. If the drift values exceed the specified limits, the run may be accepted but the test measurement system will be fully recalibrated and the results reported using Method 7E correction. If the alternative calibration error test was approved, both sets of calibration data (i.e., data determined prior to the test period and data determined following the test period).

Measurement System Performance Specifications

Zero Drift, less than or equal to ± 3 percent of the span value. **Calibration Drift**, less than or equal to ± 3 percent of span value. **Calibration Error**, less than or equal to ± 2 percent of span for Method 7E procedures, less than or equal to ± 5 percent of the calibration gas value for the alternative procedures. **Sampling System Bias**, less than or equal to ± 5 percent of the calibration gas value for Method 7E procedures.

Emission Calculation

Calculations will be done per the method including Method 7E (Equation 7E-5b) drift corrections.

Emissions will be presented in the following units:

Proposed Deviations from this BTS Template or the Method

(Insert any proposed deviations here)

METHOD 25B - DETERMINATION OF TOTAL GASEOUS ORGANIC CONCENTRATION USING A NONDISPERSIVE INFRARED ANALYZER

Applicability

This method is applicable for the determination of total gaseous organic concentration of vapors consisting primarily of alkanes. Other organic materials may be measured using the general procedure in this method, the appropriate calibration gas, and an analyzer set to the appropriate absorption band.

Summary of Method

A gas sample is extracted from the source through a heated sample line, if necessary, and glass fiber filter to a nondispersive infrared analyzer (NDIR). Results are reported as volume concentration equivalents of the calibration gas or as carbon equivalents.

Explosive Atmosphere - This method is often applied in highly explosive areas. Caution and care will be exercised in the choice of equipment and installation.

Apparatus

Measurement System. Any measurement system for total organic concentration that meets the specifications of this method. All sampling components leading to the analyzer will be heated

 \geq 110°C (220°F) throughout the sampling period, unless safety reasons are cited.

Organic Concentration Analyzer. A nondispersive infrared analyzer designed to measure alkane organics and capable of meeting or exceeding the specifications in this method.

Sample Probe. Stainless steel, or equivalent, three-hole rake type. Sample holes will be 4 mm (0.16-in.) in diameter or smaller and located at 16.7, 50, and 83.3 percent of the equivalent stack diameter. Alternatively, a single opening probe may be used so that a gas sample is collected from the centrally located 10 percent area of the stack cross-section.

Heated Sample Line. Stainless steel or Teflon[®] tubing to transport the sample gas to the analyzer. The sample line should be heated (≥ 110 °C) to prevent any condensation.

Calibration Valve Assembly. A three-way valve assembly to direct the zero and calibration gases to the analyzers is recommended. Other methods, such as quick-connect lines, to route calibration gas to the analyzers are acceptable.

Particulate Filter. An in-stack or an out-of-stack glass fiber filter is required except as explained and justified in the Proposed Deviation from the Method Section. An out-of-stack filter should be heated to prevent any condensation.

Recorder. A strip-chart recorder, analog computer, or digital recorder for recording measurement data. The minimum data recording requirement is one measurement value per minute.

Analytical Range

The span value should be between 1.5 to 2.5 times the applicable in-stack concentration based on the emission limit.

Allowable = _____ lb/hr MW = _____ lb/lbmole Stack Flow = _____ scfm

The in-stack concentration based on the emission standard and stack flow parameters is

 $\frac{(\text{lb/hr}) \times (387 \times 10^6)}{(\text{MW}) \times (\text{scfm}) \times 60} = \underline{\qquad} \text{ppm. Therefore, the analyzer span will be } \underline{\qquad} \text{ppm.}$

Calibration Gases

The calibration gases for the gas analyzer will be propane in air or propane in nitrogen. Alternatively, organic compounds other than propane can be used; the appropriate corrections for response factor must be made. The calibration gases will be _______. Calibration gases will be EPA Protocol No. 1 gases and will include a recommended shelf-life from the manufacturer over which the concentration does not change more than $\pm 2\%$ from the certified value.

Zero Gas. High purity air with less than 0.1 part per million by volume (ppmv) of organic material (methane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

Low-level Calibration Gas. An organic calibration gas with a concentration equivalent to 25 to 35 percent of the applicable span value. The low-level gas to be used during the source test will be ______.

Mid-level Calibration Gas. An organic calibration gas with a concentration equivalent to 45 to 55 percent of the applicable span value. The mid-level gas to be used during the source test will be ______.

High-level Calibration Gas. An organic calibration gas with a concentration equivalent to 80 to 90 percent of the applicable span value. The high-level gas to be used during the source test will be ______.
Analyzer Calibration

The calibration procedures described in Method 7E (calibration error, bias and drift) will be followed unless deemed not feasible and approved by the on-site BTS observer.

The analyzer calibration error check will be conducted by introducing the zero, lowrange, mid-range, and high-range gases to the analyzer. During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow rate at the analyzer. The analyzer responses to each calibration gas will be recorded. The analyzer calibration error check will be considered invalid if the gas concentration displayed by the analyzer exceeds ± 2 percent of the span for any of the calibration gases.

The sampling system bias check will be performed by introducing first an upscale gas (mid-range) at the calibration valve assembly installed at the outlet of the sampling probe, and then the zero gas. During this check, no adjustments to the system will be made except those necessary to achieve the correct calibration gas flow at the analyzer. This check will be considered invalid if the difference between the calibration error check and the calibration bias check for the same calibration gas exceeds $\pm 5\%$ of the span.

Alternatively, if approved by the BTS observer, the following calibration error test will be performed. Immediately prior to the test series (within 2 hours of the start of the test), zero gas and high-level calibration gas will be introduced at the calibration valve assembly. The analyzer output will be adjusted to the appropriate levels, if necessary. The predicted response will be calculated for the low-level and mid-level gases based on a linear response line between the zero and high-level response. Then the low-level and mid-level calibration gases will be introduced successively to the measurement system. The analyzer responses will be recorded for low-level and mid-level calibration gases and the differences between the measurement system responses and the predicted responses determined. These differences must be less than 5 percent of the respective calibration gas value. If not, the measurement system is not acceptable and will be replaced or repaired prior to testing. No adjustments to the measurement system will be made after the calibration and before the drift check. If adjustments are necessary before the completion of the test series, drift checks will be performed prior to the required adjustments and the calibration following the adjustments repeated. If multiple electronic ranges are to be used, each additional range will be checked with a mid-level calibration gas to verify the multiplication factor.

Response Time Test. Zero gas will be introduced into the measurement system at the calibration valve assembly. When the system output has stabilized, switch quickly to the high-level calibration gas. The time will be recorded from the concentration change to the measurement system response equivalent to 95 percent of the step change. The test will be repeated three times and the results averaged.

Emission Measurement Test Procedure

Organic Measurement. Sampling will begin at the start of the test period at a sample point that is centrally located in the stack. The time and any required process information will be recorded, as appropriate, in particular, noting on the recording chart, periods of process interruption or cyclic operation.

Bias and Drift Determination. Immediately following the completion of the test period and hourly during the test period, the zero and mid-level calibration gases will be reintroduced one at a time to the measurement system at the calibration valve assembly. No adjustments to the measurement system will be made until both the zero and calibration bias and drift checks are made. The analyzer response will be recorded. If the bias values exceed the specified limits, the test results preceding the check will be invalidated and the test will be repeated following corrections to the measurement system and full recalibration. If the drift values exceed the specified limits, the run may be accepted but the test measurement system will be fully recalibrated and the results reported using Method 7E correction. If the alternative calibration error test was approved, both sets of calibration data (i.e., data determined prior to the test period and data determined following the test period).

Measurement System Performance Specifications

Zero Drift, less than or equal to ± 3 percent of the span value. **Calibration Drift**, less than or equal to ± 3 percent of span value. **Calibration Error**, less than or equal to ± 2 percent of span for Method 7E procedures, less than or equal to ± 5 percent of the calibration gas value for the alternative procedures. **Sampling System Bias**, less than or equal to ± 5 percent of the calibration gas value for Method 7E procedures.

Emission Calculation

Calculations will be done per the method including Method 7E (Equation 7E-5b) drift corrections. Results will be reported (select one):

____ as propane.____ as methane by multiplying concentrations by 3.

Emissions will be presented in the following units:

Proposed Deviations from this BTS Template or the Method

METHOD 26 – DETERMINATION OF HYDROGEN HALIDE AND HALOGEN EMISSIONS FROM STATIONARY SOURCES NON-ISOKINETIC METHOD

Applicability

This method is applicable for the determination of hydrogen halides and halogens emissions from stationary sources (see list below; parameters to be measured have been checked) that do <u>not</u> emit acid particulate matter. Sources that emit acid particulate matter, such as those controlled by wet scrubbers, cannot use this template and should consider using the template for Method 26A.

Hydrogen Halides	Halogens	Halogens		
Hydrogen Chloride (HCl)	Chlorine (Cl ₂)			
Hydrogen Bromide (HBr)	Bromine (Br ₂)			
Hydrogen Fluoride (HF)				

Principle

A stack sample is withdrawn from the source through a pre-purged heated probe and filter into dilute solutions, which separately collect the gaseous hydrogen halides and halogens. The samples are then separately measured by ion chromatography (IC).

Interferences

The simultaneous presence of HBr and Cl_2 can cause a positive bias in HCl and a negative bias in Cl_2 , which would affect the HBr/Br₂ split. High concentrations of nitrogen oxides may interfere with the measurements of very low levels of Br⁻ due to the production of nitrates. In cases where HF is of concern, any new Teflon components will be preconditioned by means of heating since there is evidence that the HF may be outgassed by the Teflon components. The use of a glass wool plug to remove particulate matter could result in a negative bias in the data. Therefore one will <u>not</u> be used.

Based on the above we (appropriate box checked):

_____ Do not expect any interference.

_____ Do expect interference. The description and discussion of the anticipated interference follows.

In-Stack Detection Limits & Sample Times

The minimum detection limit of the method has been determined to be approximately 0.0125 ppm (note: this differs from that stated in the method due to errors in the method) for a standard 60 minute (120 liter) sample. Actual in-stack method detection limits (ISDL) are based on actual source sampling parameters and analytical results. Actual detection limits can be improved through increased stack gas sampled (sample time).

For this source, the in-stack concentration is:

Allowable = ______ lbs/hr Stack flow = ______ dscfm MW = molecular weight. ppm = (lb / hr) x 387E6 / (dscfm x MW x 60) = _____ ppm. Therefore, the sample time will be _____ minutes.

Sample Train & Recovery Components & Supplies

A schematic of the sampling train is shown in Figure 26-1 of the method. Specifically, the sampling train will be constructed with components specified under EPA Method 26, Section 6.0, with the following highlights.

Sample Train

- 1) The **probe liner** will be constructed of glass (Borosilicate or Quartz). To remove particulate matter from the gas stream, a teflon-glass filter in a mat configuration should be used. A glass wool plug is <u>not</u> an acceptable substitution.
- 2) A borosilicate-glass **three-way stopcock** with a heating system will connect to the outlet of the heated filter and the inlet of the first impinger.
- 3) The **impinger train** will consist of five impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. Silicone grease may be used as necessary to prevent leakage. The first and second impingers will be 30 ml midget impingers containing 15 ml of 0.1 N H₂SO₄. The third and fourth impingers will also be 30 ml midget impingers containing 15 ml of 0.1 N NaOH. The fifth impinger will contain 6- to 16-mesh silica gel. When sampling at high moisture stacks, a midget impinger with a shortened stem will be used in front of the first impinger (the stem should be sufficiently short to keep the gas stream from bubbling through the condensate).
- 4) A **Heating System** capable of maintaining a temperature of > 248 °F around the probe and filter holder.
- 5) A 25-mm (1 in) Teflon glass mat **filter** will be used. Other filters may be used but they must contain at least 75% Teflon and be of a mat configuration. Note: If the stack gas temperature exceeds 410 °F and the HCl concentration exceeds 20 ppm, a quartz-fiber filter may be used. This <u>does</u> / <u>does not</u> (circle appropriate) apply to this test program.
- 6) A filter holder made of Teflon or quartz and a filter support made of Teflon.
- 7) A leak-free **sample line**.
- 8) **Rate meter** (rotameter or some equivalent) capable of measuring flow rate to within 2% of the selected flow rate of 2 liters/min (0.07 ft^3/min).
- 9) **Purge Pump** with the capability to purge the sampling probe at 2 liters/min and a rate meter capable of measuring 0 to 5 liters/min (0.2 ft³/min).

Sample Recovery

- 1) Two wash bottles, glass or polyethylene, with capacities of 500-ml or larger.
- 2) Storage bottles, high-density polyethylene bottles, with Teflon screw cap liners.
- 3) Teflon Tape will be used for capping openings and sealing connections, if necessary, on the sampling train.

Sampling

The sample train will be assembled as per Section 8.1.1 and Figure 26-1 of the method. The probe, filter and stopcock will be pre-heated to >248 °F. Once the sample train is assembled a pre-test leak check and purge are required, as stated below.

Leak Checks

Pre-test and post-test leak checks will be conducted as follows: temporarily attach a suitable rotameter to the outlet of the dry gas meter and place a vacuum gauge at or near the probe inlet. Plug the probe inlet and pull a vacuum of at least 10 inches of Hg for pre-test leak checks and 1 to 2 in. Hg higher than the highest vacuum during testing for post-test leak checks. A leak rate as indicated by the rotameter shall not exceed 2% of the sampling rate.

Pre Test Purge

Prior to testing, the probe is placed inside the stack and purged for 5 minutes (during the purge and test run both the probe temperature and filter temperature should be greater than 248°F).

Sample Collection

Once the pre-test purge is completed, the initial meter volume is recorded and sampling begins. Turn on the sampling pump and pull a slight vacuum of approximately 1 in Hg on the impinger train. The sampling rate is adjusted to 2 liters/min (to be maintained to within 10% of this rate during testing). Readings are taken at a minimum of every five minutes for the dry gas meter volume and temperature, vacuum gauge, the probe and filter temperature, stack temperature and exit impinger temperature. At the conclusion of the test run, the probe is removed from the stack and allowed to cool. A post-test leak check is performed, as detailed above.

Sample Recovery

After the post-test leak check, the glassware is disconnected and the contents of the acid impingers (and knockout impinger if used) are poured into a leak-free storage bottle. The impingers and connecting glassware are rinsed with water and these rinses are added to the storage bottle. This procedure is repeated for the alkaline impingers and connecting glassware, using a separate storage bottle. For the alkaline impingers, add 25 mg of sodium thiosulfate per the product of ppm of halogen anticipated to be in the stack gas times the volume (dscm) of stack gas sampled (0.7 mg/ppm-dscf).

Portions of the absorbing reagents, equivalent to the amount used in the sample train, will be saved and water added to dilute to the approximate volume of the collected samples. Also, the same amount of sodium thiosulfate added to the alkaline solution samples will be added to the alkaline solution blank. Finally, a portion of the water used to rinse the sample train will also be saved.

All sample collection bottles are sealed, labeled and the fluid levels marked.

Sample Preparation and Analysis

The liquid levels on the storage containers will be noted at the laboratory and if leakage occurred, the run will either be void or methods will be used, only with the approval of BTS, to correct the final results. Sample solutions will be quantitatively transferred to 100-ml volumetric flasks, and diluted to 100 ml with water. Analysis will be by ion chromatography, per the method. All analysis will include duplicate injections, which must agree within $\pm 5\%$. Analysis will be performed no later than 4 weeks after sample collection.

Calculations

All calculations will be performed as per Method 26, including blank corrections. Detailed sample calculations will be included in the final report.

Emissions will be presented in the following units: ______.

Special Situations

If sampling for Cl₂ and/or Br₂ is not being conducted, analysis of the alkaline impingers may be disregarded.

Audit Samples

If provided, audit samples will be analyzed consistent with Section 11.2 of the method and the results will be provided in the final stack test report. It is understood that failure to meet acceptance criteria for the audit could result in the requirement to repeat the stack test program.

Proposed Deviations from this BTS Template or the Method

METHOD 26A – DETERMINATION OF HYDROGEN HALIDE AND HALOGEN EMISSIONS FROM STATIONARY SOURCES ISOKINETIC METHOD

Applicability

This method is applicable for the determination of hydrogen halides and halogens emissions from stationary sources (see list below; parameters to be measured have been checked), including sources that emit acid particulate matter.

Hydrogen Halides	Halogens	Halogens		
Hydrogen Chloride (HCl)	Chlorine (Cl ₂)			
Hydrogen Bromide (HBr)	Bromine (Br ₂)			
Hydrogen Fluoride (HF)				

Principle

A stack sample is withdrawn from the source isokinetically through a heated probe and filter into dilute solutions, which collect the gaseous hydrogen halides and halogens. The samples are then separately measured by ion chromatography (IC).

Interferences

The simultaneous presence of HBr and Cl_2 can cause a positive bias in HCl and a negative bias in Cl_2 , which would affect the HBr/Br₂ split. High concentrations of nitrogen oxides may interfere with the measurements of very low levels of Br⁻ due to the production of nitrates. In cases where HF is of concern, any new Teflon components will be preconditioned by means of heating since there is evidence that the HF may be outgassed by the Teflon components.

Based on the above we (appropriate box checked):

_____ Do not expect any interference.

_____ Do expect interference. The description and discussion of the anticipated interference follows.

In-Stack Detection Limits & Sample Times

The minimum detection limit of the method has been determined to be approximately 0.02 ppm (note: this differs from that stated in the method due to errors in the method) for a standard 60 minute (1 dscm) sample. Actual in-stack method detection limits (ISDL) are based on actual source sampling parameters and analytical results. Actual detection limits can be improved through increased stack gas sampled (sample time).

For this source, the in-stack concentration is:

Allowable = _____ lbs/hr Stack flow = _____ dscfm MW = molecular weight.

 $ppm = (lb / hr) x 387E6 / (dscfm x MW x 60) = ____ ppm.$

Therefore, the sample time will be _____ minutes.

Sample Train & Recovery Components & Supplies

A schematic of the sampling train is shown in Figure 26A-1 of the method. Specifically, the sampling train will be constructed with components specified under EPA Method 26A, Section 6.0 (similar to a Method 5 train), with the following exceptions and/or highlights.

Sample Train

- The probe nozzle will be constructed of glass (borosilicate or quartz) and calibrated according to Method 5, Sections 6.1.1.1 and 10.1, and joined to the probe liner using a Teflon union. The probe liner will be constructed of glass (borosilicate or quartz). A Teflon probe liner can be used for stack temperatures between 250°F and 410°F. If the stack temperature exceeds 410°F, a one-piece glass nozzle/probe liner assembly must be used.
- To remove particulate matter from the gas stream, a Teflon filter in a mat configuration will be used. Note: If the stack gas temperature exceeds 410°F, a quartz fiber filter may be used. This <u>does</u> / <u>does not</u> (circle appropriate) apply to this test program.
- 3) A **filter holder** made of borosilicate or quartz glass, or Teflon, with a Teflon **filter support** and a Teflon **sealing gasket**.
- 4) An optional cyclone (glass or Teflon) can be used only when the sample gas stream is saturated with moisture. The cyclone is recommended to protect the filter from any liquid droplets that may be present in the gas stream. The cyclone (check one) ____ will ___ will not be needed.
- 5) The **impinger train** will consist of five impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. The first and second impingers will be of the Standard Greenburg-Smith design and will contain 100 ml of 0.1 N H₂SO₄. The third and fourth impingers will be of the modified Greenburg-Smith design and will contain 100 ml of 0.1 N NaOH. The fifth impinger, also a modified Greenburg-Smith impinger, will contain with 6- to 16-mesh silica gel. When sampling at high moisture stacks, a knockout impinger with a shortened stem containing 50 ml of 0.1 N H₂SO₄ will be used in front of the first impinger (the stem should be sufficiently short to keep the gas stream from bubbling through the condensate). Teflon impingers are an acceptable alternative.

- 6) A heating system capable of maintaining a temperature of $> 248^{\circ}$ F around the probe and filter holder.
- 7) An **optional ambient air conditioning tube** tightly packed with approximately 150 g of fresh 8 to 20 mesh sodium hydroxide-coated silica, or equivalent, is used to dry and remove acid gases from the ambient air used to remove moisture from the filter and cyclone, when the cyclone is used.
- 8) A leak-free **sample line**.
- 9) A **pitot tube**, **differential pressure gauge** and **metering system** as described in Method 5.

Sample Recovery

- 1) The wash bottles, graduated cylinder and/or balance and rubber policeman will be the same as in Method 5. Funnels will be glass or high-density polyethylene.
- 2) The storage bottles should be high-density polyethylene bottles with Teflon screw cap liners.
- 3) Polypropylene Tweezers and/or Plastic Gloves will be used for recovery of the filter from the sampling train filter holder.
- 4) Teflon Tape will be used for capping openings and sealing connections, if necessary, on the sampling train.

Sampling

The sample train is assembled as per Section 8.1.3 and Figure 26A-1 of the method. Once the sample train is assembled a pre-test leak check is required, as stated below.

Leak Checks

Pre-test and post-test leak checks will be conducted following the procedures of Method 5. The pre-test leak check will be conducted at 15" Hg vacuum (or a lower rate not to be exceeded during sampling) and the post-test leak check will be conducted at or above the highest vacuum reached during sampling. A leakage rate in excess of 0.02 cfm is unacceptable and will void the test run.

Sample Collection

Prior to testing, both the probe and filter temperature should be greater than 248° F. Once the pre-test leak check is completed, the initial meter volume is recorded and sampling begins. Follow the general procedure given in Method 5, Section 8.5. For each run, the required data should be recorded on a data sheet such as the one shown in Method 5, Figure 5-3. If the optional knockout impinger becomes too full, it may be emptied (and saved for moisture determination and sample analysis) and charged with a new 50-ml solution of 0.1 N H₂SO₄. Before this impinger is removed a post leak check must be conducted in order to ensure that the sample train was collecting the proper sample during the elapsed time period. This procedure may be conducted as many times as necessary. At the conclusion of the test run, the probe is removed from the stack and allowed to cool. A post-test leak check is performed, as mentioned above. If applicable, the post-test moisture removal procedures listed in Section 8.1.6 of the method will be performed.

Sample Recovery

After the post-test leak check, the glassware is disconnected and the contents of the acid impingers (and knockout impinger if used) are poured into a leak-free storage bottle. The impingers and connecting glassware are rinsed with water and these rinses are added to the storage bottle. This procedure is repeated for the alkaline impingers and connecting glassware, using a separate storage bottle. For the alkaline impingers, add 25 mg of sodium thiosulfate per the product of ppm of halogen anticipated to be in the stack gas times the volume (dscm) of stack gas sampled (0.7 mg/ppm-dscf).

Portions of the absorbing reagents, equivalent to the amount used in the sample train, will be saved and water added to dilute to the approximate volume of the collected samples. Also, the same amount of sodium thiosulfate added to the alkaline solution samples will be added to the alkaline solution blank. Finally, a portion of the water used to rinse the sample train will also be saved.

All sample collection bottles are sealed, labeled and the fluid levels marked.

Sample Preparation and Analysis

The liquid levels on the storage containers will be noted at the laboratory and if leakage occurred, the run will either be void or methods will be used, only with the approval of BTS, to correct the final results. Sample solutions will be quantitatively transferred to 100-ml volumetric flasks, and diluted to 100 ml with water. Analysis will be by ion chromatography, per the method. All analysis will include duplicate injections, which must agree within $\pm 5\%$. Analysis will be performed no later than 4 weeks after sample collection.

Calculations

All calculations will be performed as per Method 26A, including blank corrections. Detailed sample calculations will be included in the final report.

Emissions will be presented in the following units: ______.

Special Situations

1) If sampling for Cl₂ and/or Br₂ is not being conducted, analysis of the alkaline impingers may be disregarded.

2) Due to different filter temperature requirements, Method 26A cannot be combined with the NJ Air Test Method 1 (the standard particulate test method used in NJ). However, particulate testing can be combined with Method 26A testing if the Permit states that the allowable(s) for particulate are to be based on EPA Method 5 testing. If this is to be done, an attachment to this Template will be included in the protocol submittal, detailing the procedures.

Audit Samples

If provided, audit samples will be analyzed consistent with Section 11.4 of the method and the results will be provided in the final stack test report. It is understood that failure to meet acceptance criteria for the audit could result in the requirement to repeat the stack test program.

Proposed Deviations from this BTS Template or the Method

Check Applicable	Analyte	Check Applicable Analytical Method Principle			
Analyte	Í	ICAP	AAS	GFAAS	ICAP-MS
	Antimony (Sb)				
	Arsenic (As)				
	Barium (Ba)				
	Beryllium (Be)				
	Cadmium (Cd)				
	Chromium (Cr)				
	Cobalt (Co)				
	Copper (Cu)				
	Lead (Pb)				
	Manganese (Mn)				
	Mercury (Hg)				
	Nickel (Ni)				
	Phosphorus (P)				
	Selenium (Se)				
	Silver (Ag)				
	Thallium (Tl)				
	Zinc (Zn)				

METHOD 29 – DETERMINATION OF METALS EMISSIONS FROM STATIONARY SOURCES

<u>Note</u>: The following is only a summary of Method 29 that highlights important aspects of the test methodology. Unless otherwise noted, procedures (i.e. sample train construction, sample recovery, sample preparation, reagents and standards, analytical technique, standards, QA/QC samples, calculations) will strictly be conducted as specified within the most current EPA Method 29 version.

Applicability

This method is applicable to the determination of metals emissions from stationary sources (See previous list). We are aware that the NJDEP does not allow particulate sampling to be combined with the Method 29 train.

Principle

A stack sample is withdrawn isokinetically from the source, particulate emissions are collected in the probe and on a heated filter, and gaseous emissions are then collected in an aqueous acidic solution of hydrogen peroxide (analyzed for all metals including Hg) and an aqueous acidic solution of potassium permanganate (analyzed only for Hg). The recovered samples are digested, and appropriate fractions are analyzed using an analytical method with a sufficient detection limit to demonstrate compliance, as detailed on the previous page.

Interferences

Iron (Fe) can be a spectral interference during the analysis of As, Cr, and Cd by ICAP. Aluminum (Al) can be a spectral interference during the analysis of As and Pb by ICAP. Generally, diluting the analytical sample can reduce these interferences, but such dilution raises the in-stack detection limits. Background and overlap corrections may be used to adjust for spectral interferences. Refer to Method 6010 of Reference 2 in Section 16.0 or the other analytical methods used for details on potential interferences to this method. For all GFAAS analyses, use matrix modifiers to limit interferences, and matrix match all standards.

Based on the above we (appropriate box checked):

_____ Do not expect any interference.

_____ Do expect interference. The description and discussion of the anticipated

interference follows.

In-Stack Detection Limits & Sample Times

Actual in-stack method detection limits (ISDL) are based on actual source sampling parameters and analytical results. Actual detection limits can be improved through increased stack gas sampled (sample time), reducing the total volume of the digested samples, improving the analytical detection limits or any combination of the three.

The goal of the sample program will be to have the in-stack detection limit at 1/10 the **Permit** allowable expressed as the in-stack concentration limit (ISCL). The ratio of ISCL/ISDL should be ≥ 10 . The following will detail these determinations and will establish the sampling time.

ISDL Calculations for each Analyte

ISDL $(ug/m^3) = A/C \times (B_F + B_B) / 1000$

 $\begin{array}{ll} \mbox{Where:} & A = \mbox{analytical detection limit (ng/ml)} \\ B_F = \mbox{amount of analyte analyzed, front half (ml, default = 300)} \\ B_B = \mbox{amount of analyte analyzed, back half (ml, default = 150)} \\ C = \mbox{volume of stack gas sampled (m^3, 1.25 m^3 for each hour)} \\ \end{array}$

In-Stack Concentration Limit (ISCL)

ISCL $(ug/m^3) = E/F \ge 2.67E8$

Where:	E = Permit allowable (lb/hr)		
	F = stack flow rate (dscfm)		

 $B_F =$ _____ $B_B =$ _____ C =_____ F =

ANALYTE	Α	Ε	ISDL	ISCL	ISCL/ISDL
Antimony (Sb)					
Arsenic (As)					
Barium (Ba)					
Beryllium (Be)					
Cadmium (Cd)					
Chromium (Cr)					
Cobalt (Co)					
Copper (Cu)					
Lead (Pb)					
Manganese (Mn)					
Mercury (Hg)*					
Nickel (Ni)					
Phosphorus (P)					
Selenium (Se)					
Silver (Ag)					
Thallium (Tl)					
Zinc (Zn)					

*Front-half ISDL calculated by (A x B_F)/C. Back-half ISDL estimated at 0.5 ug/m³. See Sections 13.2 and 11.1.3 of Method 29.

Based on the preceding, each sample run will be _____ minutes.

Sample Train & Recovery Components & Supplies

A schematic of the sampling train is shown in Figure 29-1 of the method. It has general similarities to that of EPA Method 5. Specifically, the sampling train will be constructed with components specified under EPA Method 29, Section 6.0, with the following exceptions and highlights.

No metal components will be used, eliminating potential for metal contamination

<u>Sample Train</u>

- 1) **Probe liner** and **nozzle** will be constructed of glass (Borosilicate or Quartz). A single glass piece consisting of a combined probe tip and probe liner may be used.
- 2) Sample **filters** will be constructed of quartz or glass fiber containing less than $1.3 \mu g/in^2$ of each of the metals to be measured. Analytical results provided by filter manufacturers stating metals content of the filters are acceptable.
- 3) The glass filter holder, same as Method 5, Section 6.1.1.5, except a Teflon filter support or other non-metallic support will be used.
- The impinger train (for condensing and collecting gaseous metals and 4) determining the moisture content of the stack gas) will consist of four to seven impingers connected in series with leak-free ground glass fittings or other leakfree, non-contaminating fittings. The first impinger is used as a moisture trap. The second impinger (which is the first HNO_3/H_2O_2 , 5 Percent $HNO_3/10$ Percent H_2O_2 , impinger) will be identical to the first impinger in Method 5. The third impinger (which is the second HNO₃/H₂O₂ impinger) will be a Greenburg Smith impinger with the standard tip as described for the second impinger in Method 5, Section 6.1.1.8. The fourth (empty) impinger and the fifth and sixth (both containing acidified KMnO₄, 4 Percent KMnO₄ (W/V), 10 Percent H₂SO₄ (V/V)) impingers are the same as the first impinger in Method 5. A temperature sensor capable of measuring to within 1°C (2°F) will be placed at the outlet of the last impinger. If no Hg analysis is planned, then the fourth, fifth, and sixth impingers are not used. For this test program, the fourth, fifth and sixth impingers (choose one) will / will not be used.
- 5) **Teflon Tape** will be used for capping openings and sealing connections, if necessary, on the sampling train.

Sample Recovery

- 1) During sample recovery **non-metallic brushes or swabs** for quantitative recovery of materials collected in the front-half of the sampling train.
- Glass bottles with Teflon-lined caps that are non-reactive to the oxidizing solutions, with capacities of 1000- and 500-ml, for storage of acidified KMnO₄containing samples and blanks. Glass or polyethylene bottles may be used for other sample types.
- 3) **Polypropylene Tweezers and/or Plastic Gloves**. Used for recovery of the filter from the sampling train filter holder.

Pre-Test Preparation & Train Assembly

Pre-test preparation will follow the same general procedures given in Method 5, Section 4. All sampling train glassware will be rinsed with hot tap water and then wash in hot soapy water. Next, glassware will be rinsed three times with tap water, followed by three additional rinses with water. Finally, all glassware will be soaked in a 10 percent (V/V) nitric acid solution for a minimum of 4 hours, rinsed three times with water, rinsed a final time with acetone, and allowed to air dry. All glassware openings will be covered where contamination can occur until the sampling train is assembled for sampling.

The sampling train will be set up as shown in Figure 29-1 of the method. The same general procedures given in Method 5 will be followed, except 100 ml of the HNO_3/H_2O_2 solution will be placed in each of the second and third impingers. 100 ml of the acidic KMnO₄ absorbing solution will be placed in each of the fifth and sixth impingers, and approximately 200 to 300 g of pre-weighed silica gel will be transferred from its container to the last impinger.

If Hg analysis will not be performed, the fourth, fifth, and sixth impingers will not be used, if so indicated in the previous section.

Sampling

Pre-test leak checks will be conducted followed by three (3) separate and valid isokinetic test runs performed as specified by the procedures given in EPA Method 5. A leak check will be considered valid if the leakage rate is found to be no greater than 0.020 cfm (0.00057 m³/min) or 4 percent of the average sampling rate (whichever is less). If sampling for Hg, procedures analogous to those described in Section 8.1 of Method 101A, 40 CFR Part 61, Appendix B, will be followed in order to maintain the desired color in the last acidified permanganate impinger. For each run, all required data will be recorded on a data sheet similar to the one shown in Method 5.

Post test leak checks will be conducted at the completion of each test run, with the same acceptance criteria as the pre-test leak check. If the final leak checks are acceptable, then the isokinetic percentages will be calculated as described in Method 5, with an acceptance criteria of 90 to 110 percent.

Sample Recovery

At the completion of each test run the entire sample train will be taken to a **sheltered and contamination free cleanup site**. First, the impingers will be weighed for moisture determination and the following sample fractions will be recovered. All containers will be clearly labeled and the height of all fluid levels will be marked to ensure no leakage occurred during sample transport. We will also refer to Figure 2, entitled Sample Recovery Scheme, as a general outline.

*Note: We understand the use of the exact method specified rinse volumes is necessary for the subsequent blank correction procedures.

- 1) Filter removed carefully and placed into petri dish labeled Container No. 1.
- 2) No Container No. 2 because particulate not determined in this sample train.
- 3) Probe, nozzle, and front-half glassware brush and rinse three times with a total of 100 ml of $0.1N \text{ HNO}_3$ All placed into a single storage Container No. 3.
- 4) Contents of two HNO₃/H₂O₂ impingers and the moisture knockout impinger placed in a container and the volume recorded. Clean each of the three impingers (referring to the 1st originally empty impinger and the 2nd and 3rd impingers containing HNO₃/H₂O₂, when a total of 7 impingers are used), the back half of the filter housing, and connecting glassware rinsed three times with a total volume of 100 ml of 0.1 N HNO₃ and label Container No. 4.

When sampling for Hg, steps 5, 6 and 7 are performed.

- 5) Pour all liquid from the impinger preceding the permanganate impingers (impinger No. 4 when a total of 7 impingers are used) into Container No. 5A. Three rinses using a total of exactly 100 ml of 0.1 N HNO₃ are then placed into the same container.
- 6) Place the liquid from the permanganate impingers (impinger Nos. 5 and 6 when a total of 7 impingers used) into Container No. 5B. Three rinses of the impingers and connecting glassware using a total of exactly 100 ml of KNnO₄ are then placed in the same container. Place three additional rinses of the impingers and connecting glassware, totaling 100 ml, of water into the same container.
- 7) The permanganate impingers are then rinsed using a total of 25 ml of 8 N HCl. This wash will be placed into a container containing 200 ml of water and labeled Container No. 5C. We understand that this fraction is **required** unless specifically waived by an observer from the Department.
- 8) Silica gel will be placed into Container No. 6. The color of the indicating silica gel will be noted and recorded on the data sheet to determine whether it has been completely spent.

Blank samples for QA/QC purposes, as specified in the method, will be collected in the field and labeled appropriately.

Sample Preparation & Analysis

Sample preparation and analysis will be conducted strictly as specified in Method 29 and the selected analytical method(s). Special attention will be paid to all required QA/QC procedures. On at least one sample run in the source test, and for each metal analyzed, repetitive analysis, Method of Standard Additions, serial dilutions or matrix spike addition will be performed to document the quality of the data.

All analytical fractions will be analyzed **separately**, per the method. We are aware that the NJDEP does not allow the combination of Analytical Fractions 1A and 2A.

Calculations

All calculations will be performed as per Method 29, including blank corrections. Detailed sample calculations will be included in the final report.

Emissions will be presented in the following units:

_____·

Proposed Deviations from this BTS Template or the Method

CTM-027 – DETERMINATION OF AMMONIA EMISSIONS IN STATIONARY SOURCES

Applicability

This method is applicable for the determination of ammonia emissions from stationary sources.

Principle

A stack gas sample is withdrawn isokinetically from the source and must be collected at the actual stack temperature, or slightly above, in order to minimize either negative or positive reactions that would bias the ammonia results. Samples pass through an in-stack filter and are collected in impingers containing sulfuric acid solution. All collected sample fractions are analyzed using ion chromatography.

In-Stack Detection Limits & Sample Times

CTM-027 sample recovery procedures are designed such that a sample containing 1 ppmV ammonium can be detected in the sample collection media. Actual in-stack method detection limits (ISDL) are based on actual source sampling parameters and analytical results. Actual detection limits can be improved through increased stack gas sampled (sample time).

For this source, the in-stack concentration is:

Allowable = _____ lbs/hr Stack flow = _____ dscfm MW = molecular weight.

 $ppm = (lb / hr) x 387E6 / (dscfm x MW x 60) = ____ ppm.$

Therefore, the sample time will be _____ minutes.

Sample Train Equipment & Reagents

The CTM-027 sample train is based upon the Reference Method 17 sample train, a schematic of which is shown in Figure 17-1 of that method. The sample train will be constructed of components specified under CTM-027, Section 1.1, with the following highlights.

- 1) **Probe liner** and **nozzle** constructed of borosilicate or quartz glass.
- 2) **In-stack filer holder** –constructed of borosilicate glass or Teflon, a filter support (with gaskets made of either Teflon, Viton, silicone rubber, each capable of handling actual stack temperatures).

- 3) Filters glass fiber filter without organic binders, with collection efficiency of \geq 99.95% for 0.3 um diameter particles.
- 4) Dry Gas Metering System
- 5) Impinger train
 - **Impingers #1 and #2** each of Greenburg-Smith (G-S) construction and each charged with 100 ml of 0.1N H₂SO₄.
 - **Impingers #3** modified Greenburg-Smith (MG-S) construction with the tip removed and is either left empty or charged with 100 ml of $0.1N H_2SO_4$ (if needed to capture possible breakthrough from impinger #2 due to high ammonia concentrations and/or high sample flow rate requirement).

- 6) Silicone grease may be used for impinger assembly.
- 7) Silica Gel
- 8) **DI Water** must be blank-checked for ammonium ion and other constituents of interest prior to testing.
- 9) 0.1N Sulfuric Acid Solution reagent grade

Sample Recovery Equipment

- 1) Wash bottles Polyethylene (2 quantity). One containing DI water, the other reagent-grade acetone.
- 2) **Sample Storage Bottles** Clean high-density polyethylene (HDPE) bottles with 250 or 500 ml capacity, having wide mouth construction and airtight seals. Used to store the 0.1N H₂SO₄ impinger solutions, rinses and spent silica gel.
- 3) Graduated Cylinders glass or HDPE

Sample Train Preparation & Assembly

The sample train is assembled as per Section 2.0 of the method. Once the sample train is assembled and pre-heated (either with the heating system or the stack), a pre-test leak check is required, as stated below.

Leak Checks

Pre-test and post-test leak checks will be conducted following the procedures of Method 17. The pre-test leak check will be conducted at 15" Hg vacuum (or a lower rate not to be exceeded during sampling) and the post-test leak check will be conducted at or above the highest vacuum reached during sampling. A leakage rate in excess of 0.02 cfm is unacceptable and will void the test run.

Sampling

1) **The stack gas temperature will be determined**, then the in-stack filter, probe and area just prior to the entrance of the Impinger #1 will be pre-heated to a temperature at or slightly above the stack gas temperature. Note: The pre-heating is optional and is done to save time

Impinger #4 – modified Greenburg-Smith construction with the tip removed and charged with 200-300 grams of indicating silica gel.

by getting the filter up to stack temperature. Alternatively, the train will be pre-heated in the stack.

- 2) The dry gas meter will be activated and **a pre-test leak check** will be conducted on the sample train as indicated above.
- 3) The preheated and leak checked sample train will be placed into the source stack and **the isokinetic sampling procedures in Method 17 will be followed.**
- 4) At end of sampling period a **post-test leak check** will be conducted as indicated above.
- 5) Three valid test runs will be conducted, consecutively.

Sample Recovery

Note: Acetone will not be used to dry glassware.

- 1) The nozzle is removed and the in-stack filter holder is disassembled. The filter is discarded (not used for analysis). Using DI water, front half of filter holder and nozzle are cleaned and dried to prepare them for the next run.
- 2) **Sample Container #1** (250 ml HDPE bottle):
 - A) The back half of the in-stack filter holder and the glass probe liner with any glassware attaching it to the first impinger are each rinsed three times with 10 ml portions of 0.1 H₂SO₄ and the rinses added to Sample Container #1.
 - B) Next, the same components are each rinsed three times with water and these water rinses are added to Sample Container #1 for analysis. Note: Although the method has conflicting information regarding the fate of these water rinses, since this sample is then diluted with water (see next step), BTS requires the water rinses be recovered in this step and not discarded.
 - C) The final volume of Sample Container #1 will be brought to exactly 230 ml using DI water.
- 3) Sample Container #2, Sample Container #3, and Sample Container #4 (250 ml HDPE bottles):
 - A) The Impinger #1 solution will be poured into a graduated cylinder; the volume recorded, then the contents poured into Sample Container #2. The impinger stem, impinger body and graduated cylinder will be each be rinsed with 5 ml portions of water and the rinses added to Sample Container #2. The final sample volume will be brought to exactly 230 ml using DI water.
 - B) The above procedure will be repeated for Impinger #2 and Impinger #3, placing the impinger contents and rinses into Sample Container #3 and Sample Container #4, respectively.
- 4) **NOTE**: The total volume in each Sample Container should not exceed 230 ml so that 20 ml of DI water can be used when transferring the contents in the laboratory for analysis. The final volume of each Sample Container to be analyzed should be 250 ml. If the final volume of any of the fractions is different from 250 ml, then the CTM-027 Equation 2 conversion factor must be modified accordingly (i.e. if 260 ml is the final volume in one of the sample fractions, the conversion would be 0.26 instead of 0.25).
- 5) Impinger #4 contents (silica gel) will be transferred into a 250 ml HDPE bottle to determine moisture weight gain.
- 6) A field blank of the 0.1N sulfuric acid impinger solution will be collected, recovered and analyzed, per Section 3.6 of CTM-027.

Note: All samples will be kept refrigerated (not frozen) at 39°F and warmed slowly prior to analysis.

Note: Sample analysis must be conducted **within two weeks** after their collection date in the field.

Sample Preparation & Analysis

 Impinger Solutions –The contents of each of the Sample Containers and the field blank will be poured from their HDPE bottles into separate 250 ml volumetric flasks. The interior of each emptied HDPE bottle will be rinsed twice with 10 ml portions of DI water and the rinses added to their respective flasks. The final volume in each flask will be brought to 250 ml with DI water if necessary.

NOTE: Sample Container #4 (Impinger #3 contents and rinses) need not be prepared for analysis unless ammonia breakthrough has occurred as indicated by analysis of Sample Containers #2 and #3. The general rule for determining breakthrough has occurred is when the concentration of Container #3 (Impinger #2 contents and rinses) is greater than 10% of the concentration of Container #2 (Impinger #1 contents and rinses).

2) Follow the conditions outlined in the method for Ion Chromatography conditions, calibration and QA/QC.

Calculations

All calculations must be performed in accordance with CTM-027, including any blank corrections. Detailed sample calculations will be included in the final report.

Special Situations Section

In limited situations (ie: moisture saturated stacks or extremely high temperature stacks) an instack filter may not be viable. **Subject to BTS approval**, an out-of-stack filter will be used in these situations, with the probe and filter temperature maintained:

1) At a temperature sufficient to prevent moisture condensation for a moisture saturated stack.

2) At a temperature of 350 °F or greater for extremely high temperature stacks.

If either of these situations is present and an out-of-stack filter is being proposed, justification **must** be presented and additional details **must** be provided in the following "Proposed Deviations from this BTS Template or the Method" section. Otherwise, an in-stack filter will be used per the method.

Proposed Deviations from this BTS Template or the Method