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Stationary Source Compliance Series

EVALUATION OF STATIONARY SOURCE PERFORMANCE TESTS

Emission Testing Concepts and Special Topics

U S ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF AIR, NOISE AND RADIATION
STATIONARY SOURCE COMPLIANCE DIVISION
WASHINGTON DC 20460

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EVALUATION OF STATIONARY SOURCE PERFORMANCE TESTS

Emission Testing Concepts and Special Problems

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INTENDED PURPOSE

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SECTION A. INTRODUCTION TO SOURCE SAMPLING

Basic Terminology and Nomenclature

There are three terms which are used to describe what exists in a stack:

1. Concentration - The quantity of a pollutant per quantity of effluent gas. An example of this is:

   grains (a weight unit)/cubic foot (a volume unit)

2. Stack gas flow rate - the quantity of effluent gas passing up the stack per length of time. An example of this is:

   cubic feet (a volume unit)/hour (a time unit)

3. Pollutant mass rate - The quantity of pollutant passing up the stack per length of time. An example of this is:

   pounds (a weight unit)/hour (a time unit)

These three terms are related to each other by the equation:

$$\overline{pmr}_s = \overline{c}_s \; \overline{Q}_s$$

where $\overline{pmr}_s$ = average pollutant mass emissions rate

$\overline{c}_s$ = average stack concentration

$\overline{Q}_s$ = average volumetric flow rate from the stack

The objective is to determine $\overline{pmr}_s$, so the general approach is to determine $\overline{c}_s$ and $\overline{Q}_s$ (see Stack Sampling Flow Diagram). $\overline{c}_s$ is determined through sampling train design. $\overline{Q}_s$ is given by the equation

$$\overline{Q}_s = \overline{v}_s \, A_s$$

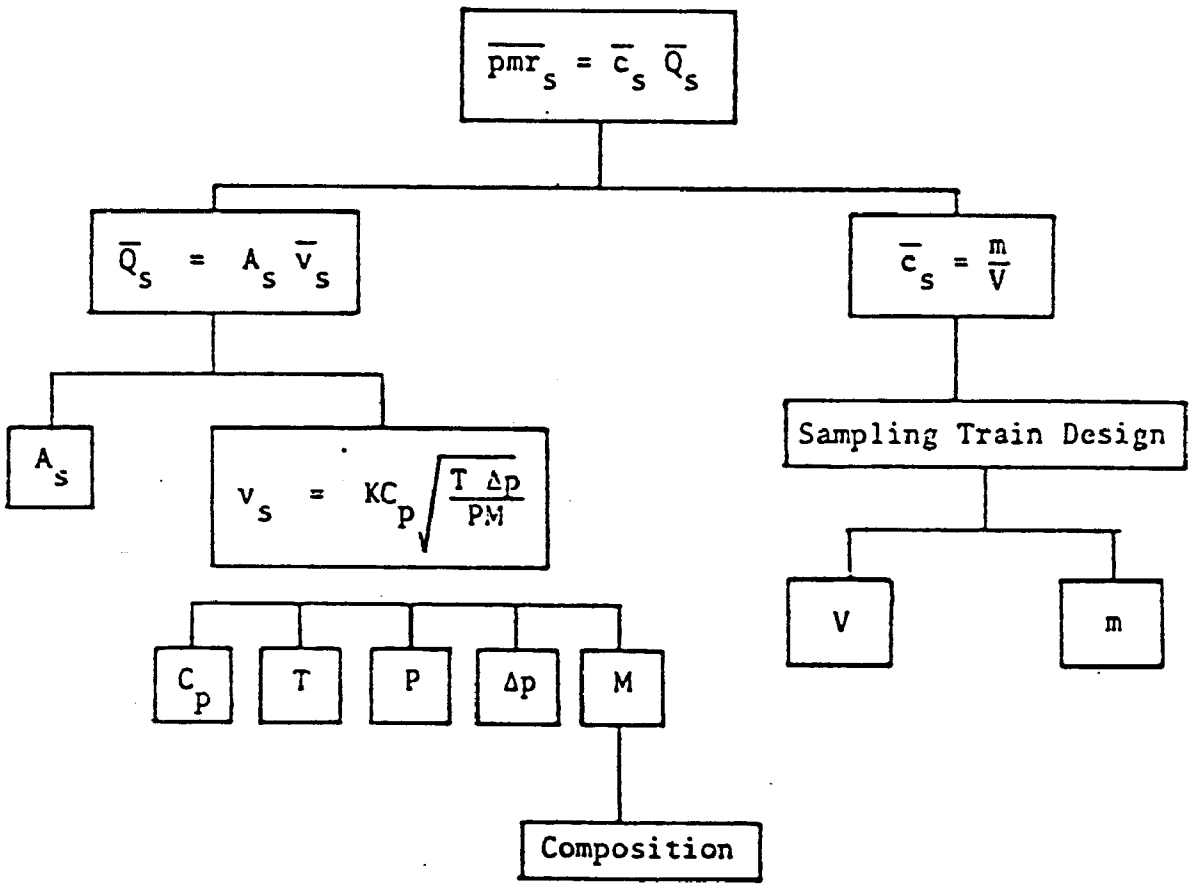
where $\overline{v}_s$ = average stack velocity

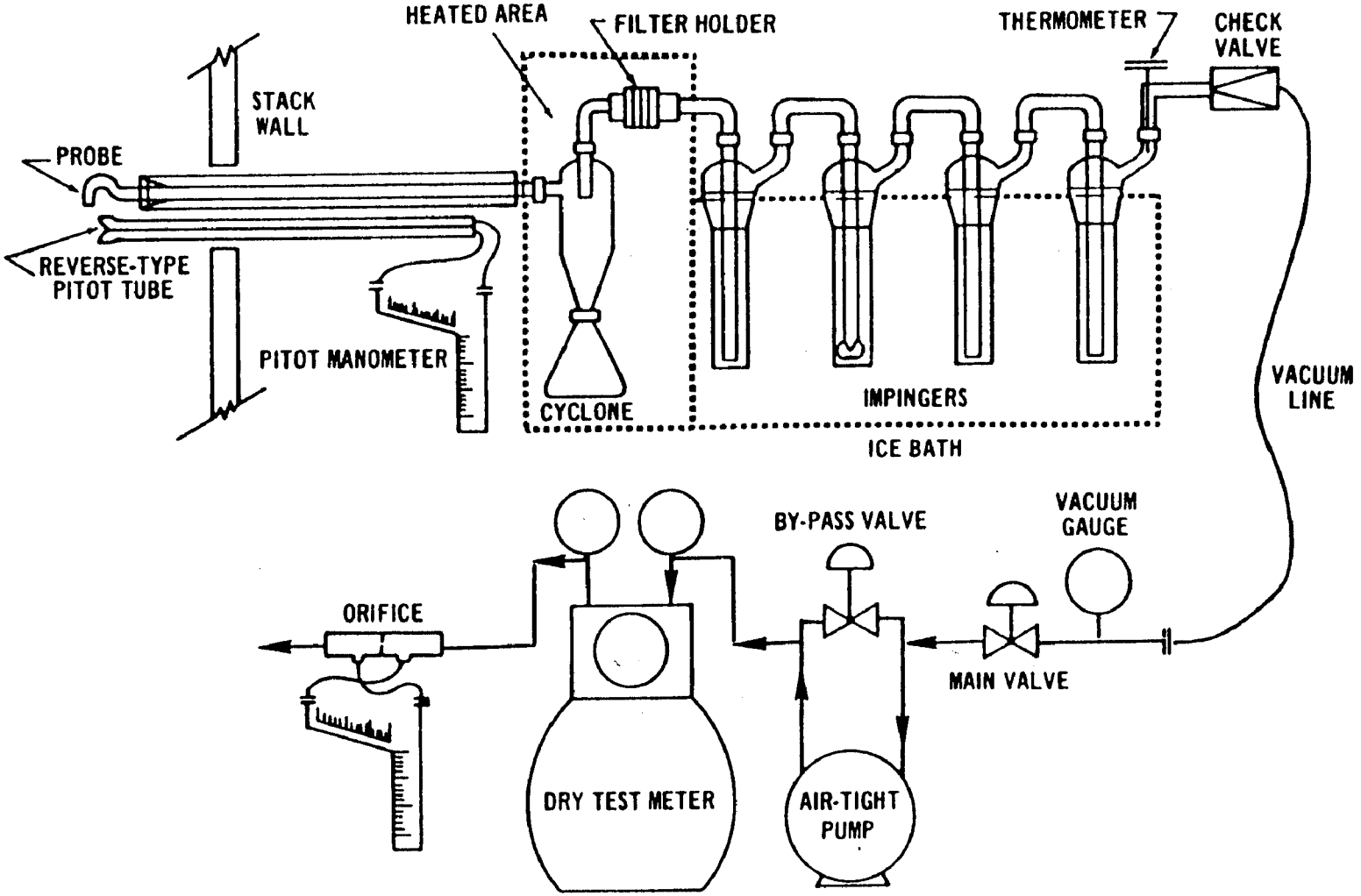
A_s = cross-section area of the stack

The cross-section area A_s is easily determined. The task of determining the average stack velocity, $\overline{v}_s$, is discussed in a following section.

Stack Sampling Flow Diagram

The overall objective of stack sampling is the determination of the average pollution mass emission rate ($\overline{pmr}_s$) and can be summarized by the flow diagram below.





Particulate sampling train.

The Source Test

A source sampling experiment provides data on source emissions parameters. The isokinetic source test extracts a representative gas sample from a gas stream. Although often used only to determine compliance with emissions regulations, the test data can also provide information useful in evaluating control equipment efficiency or design, process economics, or process control effectiveness. Valid source sampling experiments, therefore, yield valuable information to both the industrial and environmental engineer.

The source test is an original scientific experiment and should be organized and executed with the same care taken in performing any analytical experiment. This requires that objectives be decided before starting the experiment and that the procedures and equipment be designed to aid in reaching those objectives. The quantitative or qualitative analysis of the source sample should be incorporated as an integral part of the source test. After all work is done, the results should be evaluated to determine whether objectives have been accomplished. This section contains flow charts and descriptions to assist in the design, planning, and performance of the source test described.

Source Test Objectives

The essential first step in all experiments is the statement of objectives. The source test measures a variety of stack gas variables which are used in evaluating several characteristics of the emissions source. The source experiment should be developed with techniques and equipment specifically designed to give complete, valid data relating to these objectives. Approaching the experiment in this manner increases the possibilities of a representative sampling of the source parameters to be evaluated.

Experiment Design

A well designed experiment incorporates sampling equipment, techniques, and analysis into an integrated procedure to meet test objectives. The source sampling experiment must be based on a sampling technique that can collect the data required. The sampling equipment is then designed to facilitate the sampling procedure. The analysis of the sample taken must be an integral factor in the sampling techniques and equipment design. This approach of achieving test objectives provides the best possible source test program.

Designing a source test experiment requires a knowledge of sampling procedures and industrial processes, a thoroughly researched sampling experiment, and a good basic understanding of the process operation to be tested. This knowledge assists in determining the types of pollutants emitted and test procedures and analysis that

will achieve valid, reliable test results. A literature search of the sampling problem can yield information that may help improve test results or make testing much easier.

Final Test Protocol

The final test protocol clearly defines all aspects of the test program, and incorporates the work done in research, experiment design, and the presurvey. All aspects of this test, from objectives through analysis of the sample and results of the sampling, should be organized into a unified program. This program is then explained to industrial or regulatory personnel involved. The protocol for the entire test procedure should be understood and agreed upon prior to the start of the test. A well organized test protocol saves time and prevents confusion as the work progresses.

Test Equipment Preparations

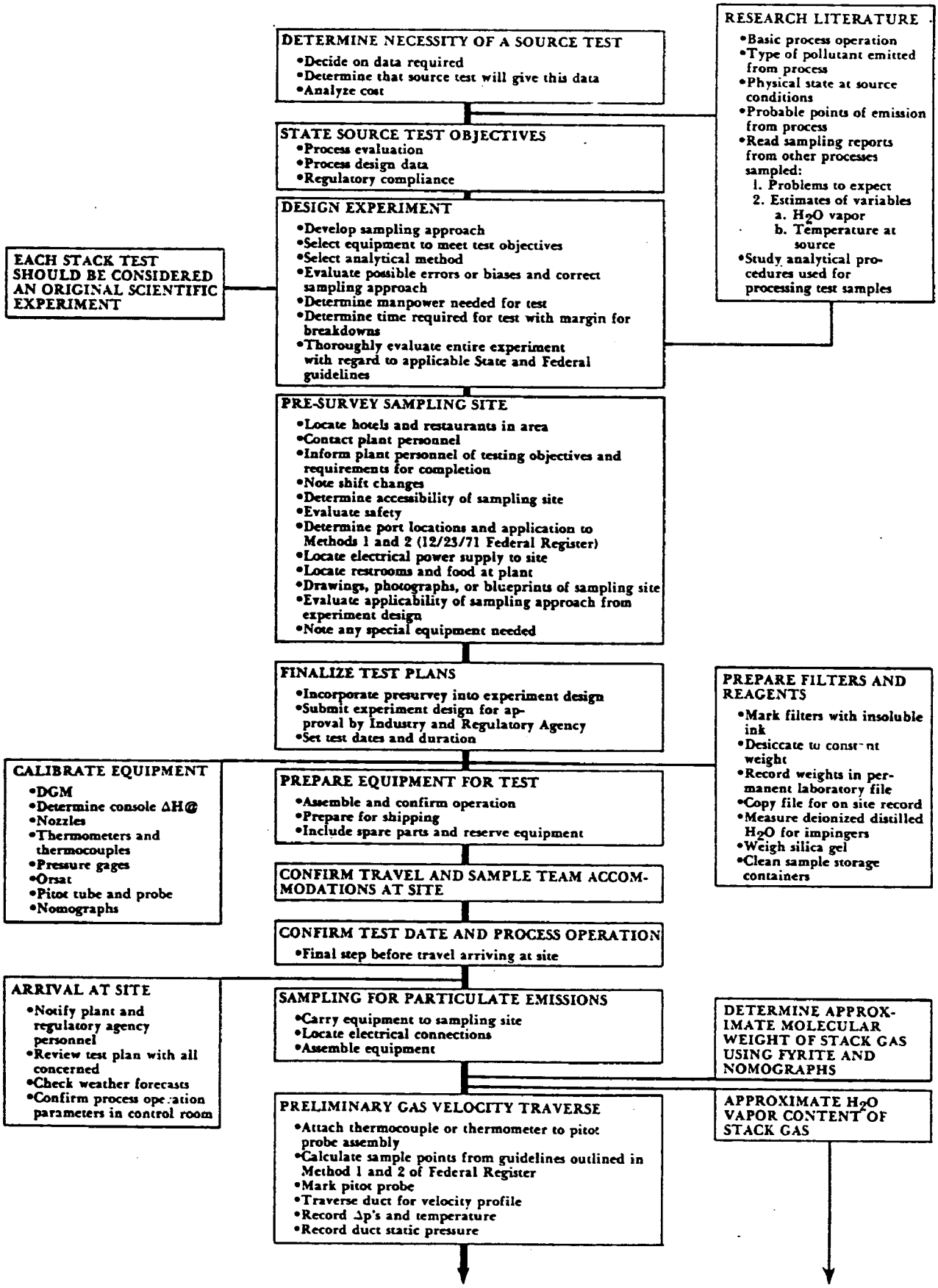
The test equipment must be assembled and checked in advance; it should be calibrated following procedures recommended in the Code of Federal Regulations and this manual. The entire sampling system should be assembled as intended for use during the sampling experiment. This assures proper operation of all the components and points out possible problems that may need special attention during the test. This procedure will assist in making preparations and planning for spare parts. The equipment should then be carefully packed for shipment to the sampling site.

The proper preparation of sampling train reagents is an important part of getting ready for the sampling experiment. The Method 5 sampling train requires well identified, precut, glass mat filters that have been desiccated to a constant weight. These tare weights must be recorded to ensure against errors. Each filter should be inspected for pinholes that could allow particles to pass through. The acetone (or other reagent) used to clean sampling equipment must be a low residue, high purity solvent stored in glass containers. Silica gel desiccant should be dried at 250° to 300°F for 2 hours, then stored in air-tight containers; be sure the indicator has not decomposed (turned black). It is a good procedure, and relatively inexpensive, to use glass-distilled, dionized water in the impingers. Any other needed reagents should be carefully prepared. All pertinent data on the reagents, tare weights, and volumes should be recorded and filed in the laboratory with duplicates for the sampling team leader.

Testing at the Source

The first step in performing the source test is establishing communication among all parties involved in the test program. The source sampling test team should notify the plant and regulatory agency of their arrival. All aspects of the plant operation and sampling experiment should be reviewed and understood by those

Figure 5-1. Planning and performing a stack test.



involved. The proper plant operating parameters and sampling experiment procedures should be recorded in a test log for future reference. The sampling team is then ready to proceed to the sampling site.

The flow diagram outlines the procedures for performing the stack test. The items given are for a basic Method 5 particulate sample. Each item is explained in various sections of this manual. The laboratory training sessions given in Course 450 help to organize the Method 5 test system.

The flow diagram should be of assistance to those having completed the 450 course curriculum and can also serve as a useful guide to anyone performing a stack test.

METHODS FOR SETTING THE ISOKINETIC FLOW RATE IN THE METHOD 5 SAMPLING TRAIN

The commercially available nomograph is often used for the solution of the isokinetic rate equation. These nomographs have based the solution of the isokinetic equation upon the assumptions that the pitot tube coefficient will be 0.85, the stack gas dry molecular weight will be 29.0 lb/lb-mole and will only vary with a change in stack gas moisture content in addition to relying on the use of a drying tube in the train. The nomograph also assumes that changes in other equation variables will be insignificant. Many purchasers are unaware of these assumptions or manufacturer construction errors and use the device without calibrating it or verifying its accuracy. Procedures are presented here to ascertain the precision of nomograph construction and its accuracy. The basic equations employed in constructing a nomograph are given and a calibration form is provided.

The equation for derivation of the isokinetic rate is given below:

(Eq.5-1) $$\Delta H = \left[846.72\ D_n^4\ \Delta H@\ C_p^2\ (1 - B_{ws})^2\ \frac{M_d\ T_m\ P_s}{M_s\ T_s\ P_m} \right] \Delta p$$

where

C_p = *pitot tube coefficient*
D_n = *nozzle diameter (in.)*
ΔH = *pressure difference of orifice meter (in. H_2O)*
$\Delta H@$ = *orifice meter coefficient, ΔH for 0.75 cfm at STP = $0.9244/K_m^2$ (in. H_2O)*
M_s = *apparent stack gas molecular weight* = $M_d(1 - B_w) + 18B_w$ *(lb/lb-mole)*
M_d = *dry gas molecular weight (29) for dry air (lb/lb-mole)*
P_s = *absolute stack pressure (in. Hg)*
P_m = *meter absolute pressure (in. Hg)*
Δp = *pressure difference of pitot tube (in. H_2O)*
T_m = *absolute meter temperature* = °R = °F + 460°

isokinetic $\Delta H = K\Delta p$

K = *Reduced terms in the isokinetic equation.*

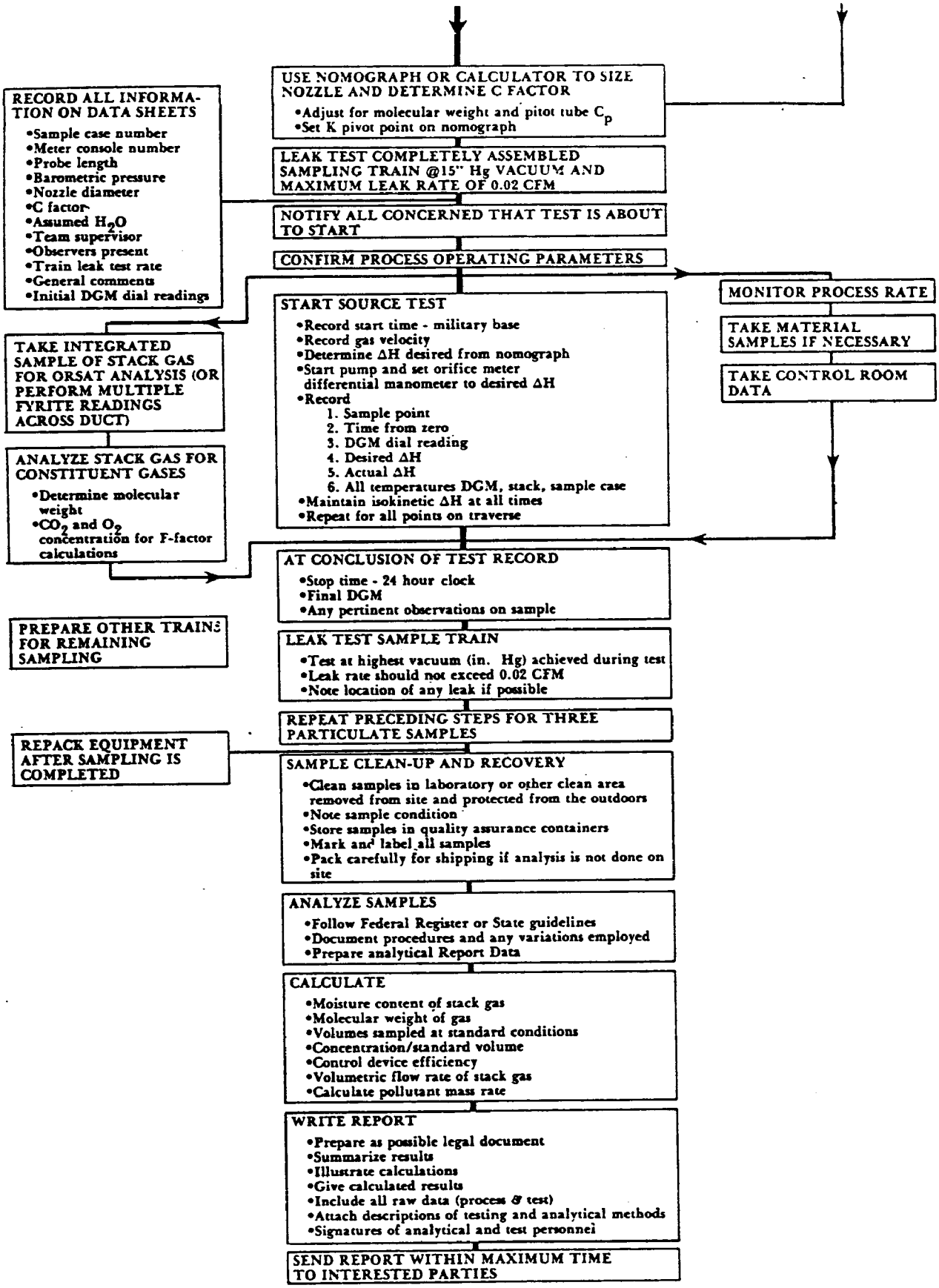
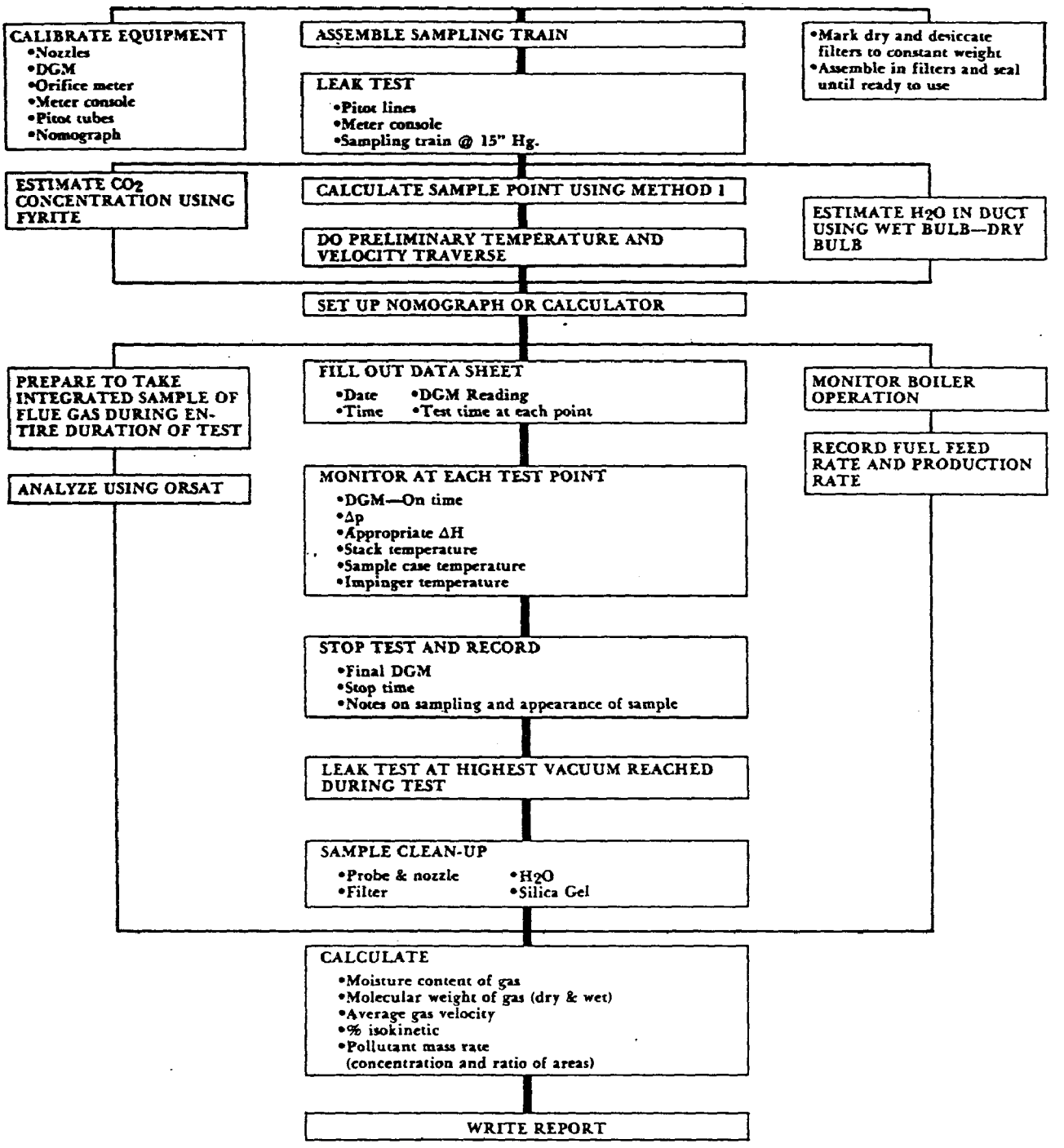

USE NOMOGRAPH OR CALCULATOR TO SIZE NOZZLE AND DETERMINE C FACTOR
•Adjust for molecular weight and pitot tube C_p
•Set K pivot point on nomograph
RECORD ALL INFORMATION ON DATA SHEETS
•Sample case number
•Meter console number
•Probe length
•Barometric pressure
•Nozzle diameter
•C factor
•Assumed H_2O
•Team supervisor
•Observers present
•Train leak test rate
•General comments
•Initial DGM dial readings
LEAK TEST COMPLETELY ASSEMBLED SAMPLING TRAIN @15" Hg VACUUM AND MAXIMUM LEAK RATE OF 0.02 CFM
NOTIFY ALL CONCERNED THAT TEST IS ABOUT TO START
CONFIRM PROCESS OPERATING PARAMETERS
MONITOR PROCESS RATE
TAKE MATERIAL SAMPLES IF NECESSARY
TAKE CONTROL ROOM DATA
START SOURCE TEST
•Record start time - military base
•Record gas velocity
•Determine ΔH desired from nomograph
•Start pump and set orifice meter differential manometer to desired ΔH
•Record
1. Sample point
2. Time from zero
3. DGM dial reading
4. Desired ΔH
5. Actual ΔH
6. All temperatures DGM, stack, sample case
•Maintain isokinetic ΔH at all times
•Repeat for all points on traverse
TAKE INTEGRATED SAMPLE OF STACK GAS FOR ORSAT ANALYSIS (OR PERFORM MULTIPLE FYRITE READINGS ACROSS DUCT)
ANALYZE STACK GAS FOR CONSTITUENT GASES
•Determine molecular weight
•CO_2 and O_2 concentration for F-factor calculations
AT CONCLUSION OF TEST RECORD
•Stop time - 24 hour clock
•Final DGM
•Any pertinent observations on sample
PREPARE OTHER TRAINS FOR REMAINING SAMPLING
LEAK TEST SAMPLE TRAIN
•Test at highest vacuum (in. Hg) achieved during test
•Leak rate should not exceed 0.02 CFM
•Note location of any leak if possible
REPEAT PRECEDING STEPS FOR THREE PARTICULATE SAMPLES
REPACK EQUIPMENT AFTER SAMPLING IS COMPLETED
SAMPLE CLEAN-UP AND RECOVERY
•Clean samples in laboratory or other clean area removed from site and protected from the outdoors
•Note sample condition
•Store samples in quality assurance containers
•Mark and label all samples
•Pack carefully for shipping if analysis is not done on site
ANALYZE SAMPLES
•Follow Federal Register or State guidelines
•Document procedures and any variations employed
•Prepare analytical Report Data
CALCULATE
•Moisture content of stack gas
•Molecular weight of gas
•Volumes sampled at standard conditions
•Concentration/standard volume
•Control device efficiency
•Volumetric flow rate of stack gas
•Calculate pollutant mass rate
WRITE REPORT
•Prepare as possible legal document
•Summarize results
•Illustrate calculations
•Give calculated results
•Include all raw data (process & test)
•Attach descriptions of testing and analytical methods
•Signatures of analytical and test personnel
SEND REPORT WITHIN MAXIMUM TIME TO INTERESTED PARTIES

Figure 5-2. Source test outline.



The Method 5 sampling train is intended to operate at a sampling rate of 0.75 cfm of dry air at 68°F and 29.92 in. Hg. The orifice meter pressure differential that would produce such a sampling rate through the orifice is designated $\Delta H@$.

An additional equation is necessary in order to estimate the nozzle diameter that will give a flow rate of 0.75 cfm at a reasonable pressure drop across the orifice meter.

(Eq.5-2)
$$D_n = \sqrt{\frac{0.0358\ Q_m\ P_m}{T_m\ C_p(1-B_{ws})}\ \sqrt{\frac{T_s\ M_s}{P_s\ \bar{\Delta}p}}}$$

where

D_n = *nozzle diameter (in.)*
Q_m = *volumetric flow rate through meter (ft^3)*
P_m = *absolute pressure at meter (in. Hg)*
P_s = *absolute pressure at stack (in. Hg)*
T_m = *absolute temperature at meter (°R)*
T_s = *absolute temperature at stack (°R)*
C_p = *pitot tube calibration coefficient*
B_{ws} = *water vapor in stack gas, volume fraction*
M_s = *molecular weight of stack gas, wet basis (lb/lb-mole)*
Δp = *average velocity head of stack gas (in. H_2O)*

Once D_n is calculated, the source tester should select the nozzle in his tool box which has a value closest to that calculated. The actual nozzle used should be checked with calipers, and that value of D_n is then substituted in Equation 5-1.

Most of the variables in this equation and the isokinetic ΔH equation are known prior to sampling or can be closely estimated. Often the solution to the equation can be partially calculated before the sampling with the few remaining variables inserted and the equation quickly solved on site. The calculation of isokinetic ΔH using the derived equations allows the sampler to more quickly and easily adjust the sampling rate for changes in the stack gas variables.

SAMPLING METER CONSOLE OPERATION

The sampling meter console must be calibrated and thoroughly leak tested. Meter console operating procedures will differ somewhat according to manufacturer. The procedures discussed here will aid in operating most types of consoles. The objective is to understand console operating procedures for isokinetic source sampling.

Sampling Train Leak Tests

Completely assemble the sampling train as intended for use during the test. Turn on probe and filter heating systems and allow them to reach operating temperatures. Disconnect the umbilical cord vacuum line and turn on the meter

console pump. This allows the pump to lubricate itself and to warm up (this is especially important in cold weather). Leak test the pitot tubes and lines during this warm up.

The pitot tube impact pressure leg is leak tested by applying a positive pressure. Blow into the impact opening until $\geq$ 7.6 cm (3 inches) H_2O is indicated by the differential pressure gage. Seal the impact opening. The pressure should be stable for at least 15 seconds. The static pressure leg of the pitot tube is leak tested in a similar way by drawing a negative pressure $\geq$ 7.6 cm H_2O. Correct any leaks.

The sampling train is leak tested when it has reached operating temperature. Turn off the console pump; connect the umbilical vacuum line. With the coarse control value completely off, turn the fine adjustment (bypass) valve completely counterclockwise. Plug the nozzle inlet and turn on the console pump. Slowly turn the coarse adjustment valve fully open. Gradually turn the fine adjustment valve clockwise until 380 mm (15 inches) Hg vacuum appears on the vacuum gage. If this vacuum is exceeded, do *not* turn the fine adjustment valve back counterclockwise; proceed with the leak test at the vacuum indicated or slowly release the nozzle plug and restart the leak test. At the desired vacuum observe the dry gas meter pointer. Using a stopwatch, time the leak rate for at least 60 seconds. The maximum allowable leak is 0.00057 m^3/min. (0.02 cfm). Having determined the leak rate, slowly release the nozzle plug to bleed air into the train; when the vacuum falls below 130 mm(5 inches) Hg, turn the coarse adjustment valve completely off. If the leak test is unacceptable, trace all sections of the sampling train from the filter holder inlet back. (i.e., leak test from the filter inlet, then the first impinger, etc.) until the leak is found. Correct the leak and retest. Leak test at the highest vacuum reached during the test after the completing the sampling procedure. Testing for leaks should also be done any time the train is serviced (i.e., filter holder change). Record all dry gas meter readings and leak rates for each leak test.

Train Operation

When the leak tests are completed, the sampling console should be prepared for sampling. The sampling console differential pressure gages for the pitot tubes and orifice meter should be checked. Zero and level the gages as required. If the console does not use oil manometers, the gages must agree with an oil manometer within 5 percent for at least 3 Δp readings taken in the stack. This check should be done before testing. Oil manometers should be periodically leveled and re-zeroed during the test if they are used in the console.

The console operator should then determine the source variables used in solving the isokinetic rate equation. The isokinetic ΔH may be determined by using a nomograph, an electronic calculator, or a source sampling slide rule. The variables that need to be determined are: stack gas moisture content, average gas velocity pressure (Δp), stack gas temperature, and estimated average console dry gas meter temperature. The stack gas moisture can be determined by Reference Method 4

sampling or estimated with a wet bulb-dry bulb thermometer technique. The average Δp and stack gas temperature are determined by a preliminary stack traverse. The dry gas meter average temperature can be estimated to be 10°C (25° – 30°F) greater than the ambient temperature at the site. These values are then used in the nomograph or calculator to find the isokinetic ΔH.

The operator can now set up the sampling data sheet. Record the dry gas meter initial reading. Position sampling train at the first sampling point; read the pitot tube Δp and calculate the corresponding ΔH. Record starting time of the test. Turn on the console pump and open the coarse sampling valve while simultaneously starting a stopwatch. Adjust ΔH to the proper value using the fine adjustment valve. Check temperatures and record all data on the data sheet.

The sampling train should be moved to the next sampling point about 15 seconds before the time at point one is over. This allows the pitot tube reading to stabilize. The dry gas meter volume at the point sampled is read when the stopwatch shows the point sample time is over. The operator should quickly read the Δp and calculate ΔH for the next point, then set the proper sampling rate. Record all data and proceed as described for all points on the traverse. At the end of the test, close the coarse valve, stop the pump, and record the stop time. Record the final dry gas meter reading. Remove the sampling train from the stack and test the system for leaks. Record the leak rate. After the train has cooled off, proceed to the cleanup area.

SAMPLING CASE PREPARATION

Inspect and clean the source sampling glassware case before a sampling experiment; remove and clean the sample case glassware. Check the case for needed repairs and calibrate the filter heater. Store the case completely assembled.

Glassware

All glassware including the filter holder and frit should be disassembled and cleaned. Separate the individual glass pieces and check for breaks or cracks. Pieces needing repair are cleaned after repairs have been made. A thorough glass cleaning for simple particulate testing is done with soap and water followed by a distilled water rinse. If analytical work is to be performed on the sample water condensed, clean the glassware by soaking in a methanol-basic hydroxide (NaOH or KOH) solution with $pH \geq 9$. Glass should be left in the base solution until any stains can be easily washed away, but not any longer than 48 hours as the solution can etch the glass. The base should be rinsed away with several portions of distilled water. If ball-joint glassware is used, remove vacuum grease before cleaning with heptane, hexane, or other suitable solvent. Clean the glass frit by pulling several aliquots of HNO_3 through the glass frit with a vacuum pump. It should be rinsed at least three times with double volumes of distilled water and dried before using.

The rubber gasket surrounding the frit should be cleaned, removing any particles imbedded in the rubber, which could prevent proper sealing. The frit and gasket must be constructed such that the glass filter mat does not become compressed in the sealing area. If this is not the case, or the rubber is in poor condition, discard the frit.

The Sample Case

The sample case should be checked thoroughly for needed repairs. All handles, brackets, clamps, and electrical connections must be inspected. Insulation in both the hot and cold areas must be in good condition. The sample case should not leak water from the melting ice into the filter heating compartment. The impinger section should have protective foam padding on the bottom and a good drainage system. The drain plug should be clean.

Calibrate the heater in the filter compartment to maintain a temperature around the filter of 120° ± 14°C (248° ± 25°F)or at other temperatures as specified in the subparts of Title 40 of the Code of Federal Regulations. This calibration should be performed at several conditions (to account for seasonal weather changes) so that the filter compartment temperature can be maintained at the proper level at all times. Often during sampling the filter section is not easy to see, consequently, the filter temperature is difficult to monitor accurately. If the case is calibrated for several conditions, operators can maintain proper temperature control more closely.

Sampling Preparations

The sample case is readied for sampling by filling the impingers with water and silica gel. Impingers 1 and 2 are each filled with 100 ml of water by inserting a funnel in the side arm and slowly pouring in the water. This makes it easy to displace in the impinger and keeps the water from filling the bubbler tube. The third impinger is left dry. The fourth impinger is filled with 200-300 gm of pre-weighed silica gel. The silica gel must be added through the side arm. This prevents dust from collecting on greased ball joints or silica gel from being pulled up the center tube and out of the impinger. After loading the impingers, securely fasten the U-joints. Attach the probe to the sampling case and secure the filter holder in position. Allow the filter compartment and probe to reach operating temperature. Leak test the assembled train from the probe nozzle by pulling 380 mm Hg (15 in. Hg) vacuum on the system. The maximum allowable leak rate is 0.00057 m^3/min (0.02 cfm). After the leak test, fill the impinger section with ice and allow time for all temperatures to stabilize.

SAMPLING PROBE PREPARATION

The sampling probe should be thoroughly inspected before field use. Remove the glass probe liner by loosening the union at the end of the probe. Completely disassemble the probe union and seal gasket, and inspect all the individual components

Probe Sheath and Pitot Tubes

The stainless steel probe sheath should have a small hole drilled near the end of the probe. This prevents a pressure differential inside the sheath from possibly diluting the sample with air drawn down the probe. If the hole is not there, the probe end (fitted into the sample case) should be sealed air tight. Check the weld at the swage fittings for cracks and repair if necessary. Inspect the pitot tubes for damage and proper construction details (see pitot tube calibration section). Pitot tubes should be cleaned, checked for cracks or breaks, and securely fastened to the probe sheath to prevent accidental misalignment in the stack. All pitot tubes and components must be leak tested.

Examine the union and seal gasket for wear. A stainless steel ring should be included in the union-gasket configuration for good compression and an air tight seal. If a rubber o-ring gasket is used (stack temperatures $\leq$ 350°F) it should be inspected for wear and replaced if necessary. Asbestos string gaskets must be replaced each time the union-gasket is disassembled. After inspecting the glass liner-heating element, reassemble the probe in the following manner to prevent leaks:

1. Insert glass liner through probe and swage nut;
2. Place stainless steel ring over glass with flat side facing out;
3. Fit gasket over glass liner and push onto steel ring;
4. Align glass liner end with edge of swage nut closest to pitot tube orifice openings;
5. Screw the union on finger tight;
6. Use probe wrenches to tighten the union. If too much tightening is done here, the end of the glass liner will break.

Glass Liner-Heating Element

The glass liner should be thoroughly cleaned with a probe brush, acetone, and distilled H_2O. If it will not come clean in this manner, it should be cleaned with dilute HCl or replaced. The glass liner-heating element in many sampling probes can not be separated, making thorough cleaning difficult. An easily separated liner-heater is a great advantage.

The heating element should be checked for good electrical insulation; the insulation on a frequently used probe liner heating element will eventually be worn or burned away. This can expose frayed wires, which may short against the probe sheath. These hazards can be avoided with careful inspections and repair. After thorough inspection, check the heating element in the reassembled probe. This procedure is helpful in finding problems before arrival at the sampling site. Attention should be given to the function of the electrical system and wrappings around the glass liner; these wraps help prevent electrical shorts against the probe sheath while minimizing glass liner flexing that can cause a liner break or electrical short.

Summary

A thorough probe check before a sampling experiment helps prevent field problems. Disassemble the probe and inspect all components. Make certain construction details and integrity are correct. Clean the glass liner thoroughly. Check the heating element electrical connections. Test the reassembled probe for leaks and proper heating.

CLEANING AND ANALYTICAL PROCEDURES FOR THE METHOD 5 SAMPLING TRAIN

The clean-up and analysis of the sample taken with the Method 5 Sampling Train is an integral part of the entire experiment. The precise operation of Method 5 Sampling equipment must be complemented by a careful clean-up of the train components. Analysis of the sample using approved procedures and good laboratory technique provides accurate laboratory data. Good testing at the stack must be followed by accurate analysis in the laboratory so that valid data may be presented.

Cleaning the Sampling Train

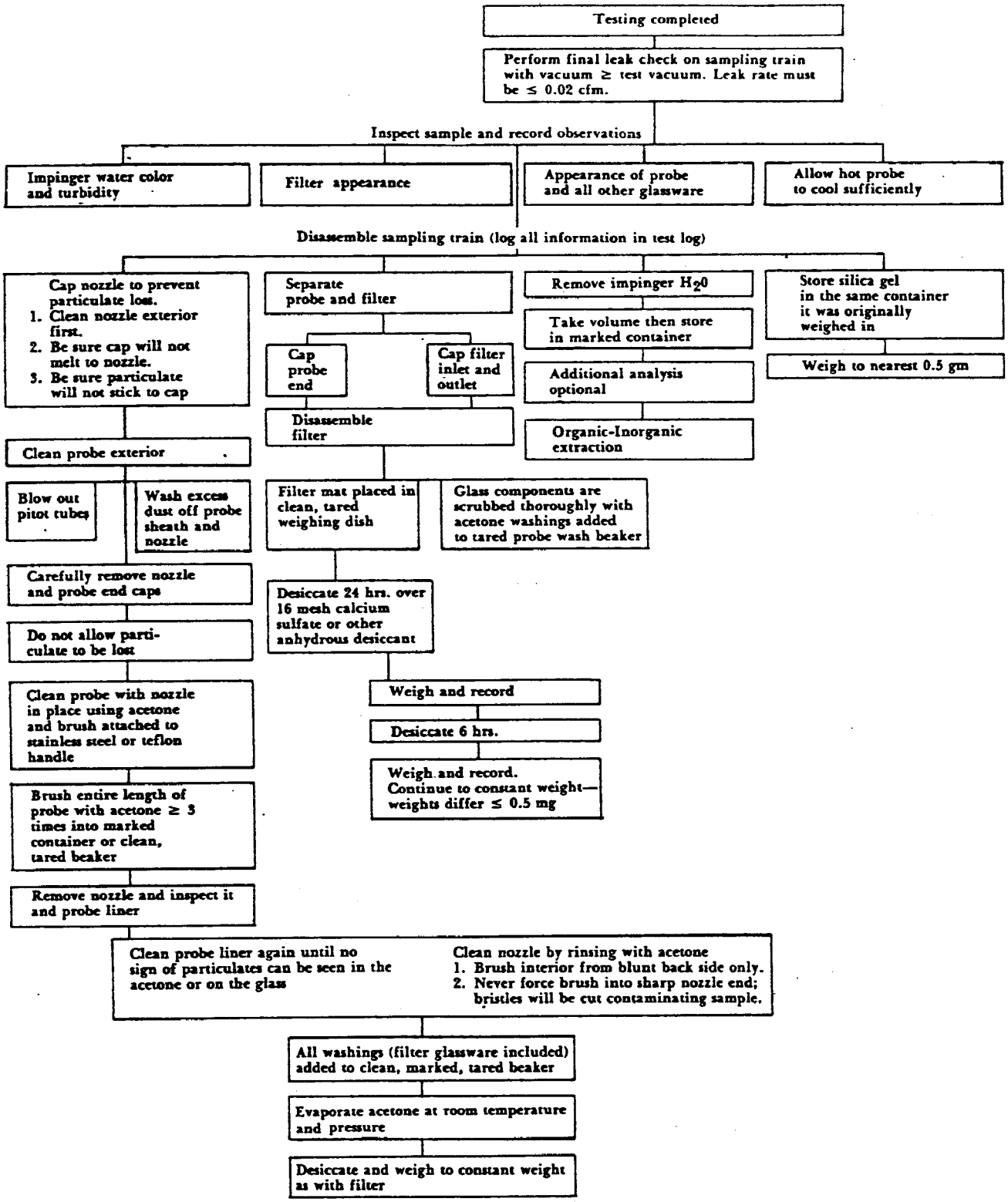
The sequence of procedures in cleaning the sampling train is best presented in an outline-flowchart form. Each step is presented with appropriate comments.

Additional Comments

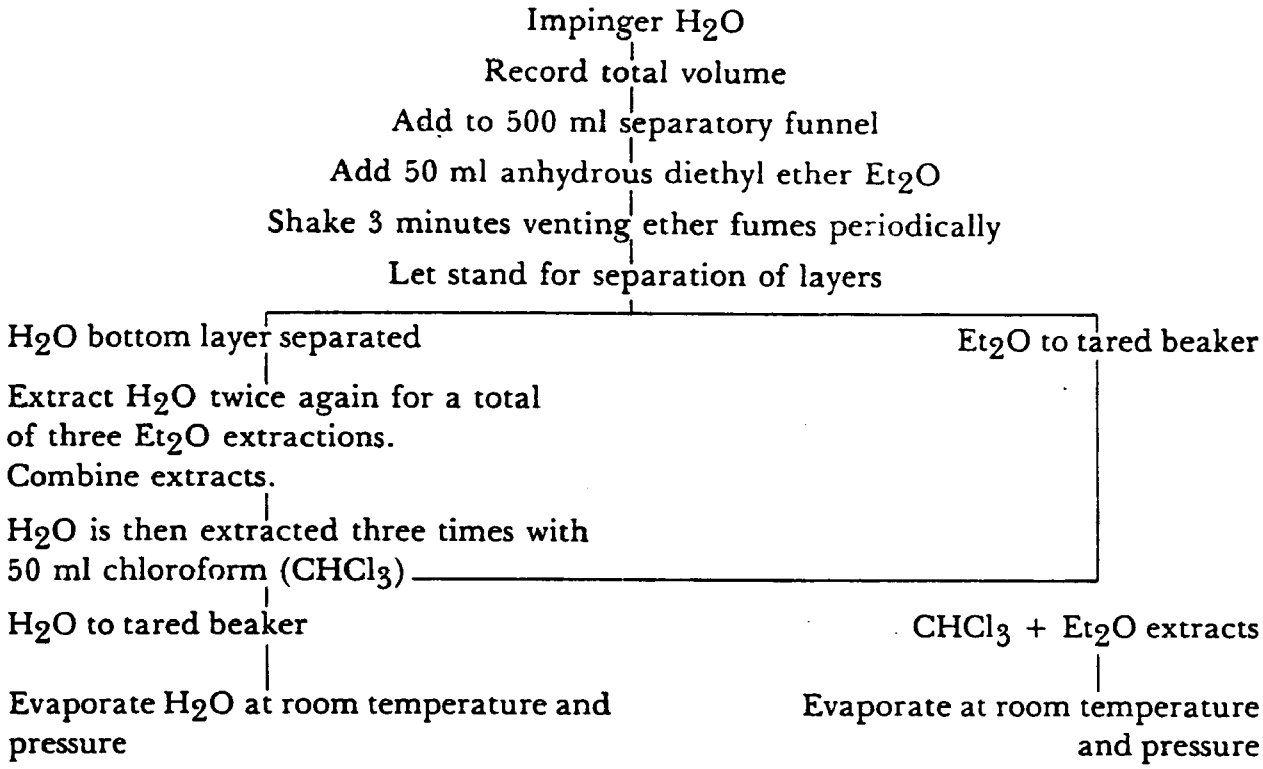
The flowchart (Figure 5-3) gives the general procedure for sample clean-up. Many factors can affect the accuracy of the final sample obtained. Care and experience are very important when cleaning the sample train. A number of helpful tips are given below;

1. Always perform clean-up procedures in a clean, quiet area. The best area is a laboratory.
2. Make a probe holder for the probe cleaning procedure or be sure two people perform the procedure; this prevents spills and accidents.
3. Clean all equipment in an area where an accidental spill may be recovered without contaminating the original sample.
   a. Open and clean the filter holder over clean glassine or waxed paper so that a spill can be recovered.
   b. Clean probe into a container sitting on the same type of glassine paper.
4. Clean the probe equipment thoroughly:
   a. Brush probe a minimum of three times.
   b. Visually inspect the probe interior.
   c. Record appearance and confidence of cleanliness.
   d. Repeat brushing until cleaning is complete.
   e. Confidence $\geq$ 99%. Check with tared cotton swab brushed through probe.

Figure 5-3. Cleaning the sample train.



5. Clean filter equipment thoroughly.
   a. Brush all glassware until clean.
   b. Check with tared cotton swab.
   c. Remove all filter mats adhering to rubber seal ring. This is extremely important for accurate particulate weighing.
   d. Do not scrape glass frit into sample.
6. The laboratory scale accuracy and sensitivity should be checked before each analysis using standard weights. Actual weight and scale reading should agree to ± 0.5 mg.
7. Careful labeling of all train components, tared beakers, and sample containers avoids problems and confusion.
8. Permanently marked weighing glassware with permanent record of their new, clean, reference tare weight allows a check of cleanliness when tared just prior to use. This can also be helpful in checking any weighing discrepancies in the analysis (re-tare reference periodically).
9. Acetone is the solvent recommended for cleaning; however, water washing may be suggested by the type of pollutant sampled and should be added to the procedure if indicated.
10. Adding heat to the evaporation of solvent could evaporate volatile materials and give erroneous data.
11. The laboratory must have:
    a. An analytical balance with minimum precision to 0.5 mg,
    b. Large desiccating container that is air tight.
12. Use only American Chemical Society Reagent grade organic solvent.
13. Use deionized, glass-distilled H_2O.
14. Evaporate a control blank of 100 ml of each solvent used in any part of the analysis in tared beaker at room termperature and pressure.
15. Use only glass wash bottles and glass containers for all procedures that involve analytical workup. Only silica gel may be stored in plastic containers.
16. Organic-inorganic extraction of the impinger may be useful in determining emissions from some sources. Use the flowchart as a guide to this procedure.



17. Procedures given here are only for cleaning Method 5 Train, although, they are good general starting point procedures for cleaning any sampling train.

The most important aspect of cleaning and analyzing the Method 5 Sampling Train is the practice of good laboratory technique. The sampling team may not include an experienced chemist; therefore, good technique may have to be learned by all team members. If an experienced analytical chemist is a member of the sampling team it would probably be best to allow him to assist in cleaning the equipment. This would help to assure good techniques and perhaps save time in preparing samples for more extensive qualitative or quantitative analysis.

SAFETY ON SITE

Source sampling is performed at a variety of industrial sites and under many different conditions. Adequate safety procedures may be different for any given situation; however, generally accepted industrial safety procedures should be helpful to source samplers. The test team must be aware of safe operating methods so that alert discretion may be used for team safety at a particular sampling site. Safety is an attitude that must be instilled in all sample team members. Well thought out and followed procedures will ensure the safety of all team members. The team concept essential to successful testing is vital for *safe* testing. It must be stressed that safety is everyone's responsibility for themselves as well as for other team members.

Key Factors to Good Safety

Knowledge and experience are the major factors in formulating sound safety practice. An individual must draw upon these factors in determining safe methods. A knowledge of standard safety and operating procedures will permit their application in any situation. This basic knowledge in conjunction with understanding of the job tasks and possible dangers assists in planning preventive safety measures. Plans for operating at the job site may be developed around these procedures. If an accident does occur, the people involved must be informed of proper emergency practices and use of first aid. Job experience and analysis of past accidents should be used in developing preventive safety programs.

Accident Analysis

The basic philosophy of a safety program should be that accidents are caused and, therefore, can be avoided or prevented. Accident analysis is a productive tool of this philosophy when it is used as a preventive step. This implies advance examination of a potentially hazardous situation to predict possible accidents and eliminate their causes. Accident analysis is most effective when employed after an accident has taken place. The analysis procedure involves listing the major and contributing causes of the accident. If the real causes of the accident are analyzed in this manner, corrective action will suggest itself. Accident analysis should include preventive suggestions from people involved at the job site or those who have been previously injured.

Common Causes of Accidents

There are a number of items that may be considered common causes of accidents:

1. Failure of supervisory personnel to give adequate instructions or inspections. This includes instructions for performing the job and safety requirements. Inspection of the job site is advisable for all applicable concerns and safety before, during, and after the job.
2. Failure of person in charge to properly plan or conduct the activity. Experiment design and performance are important factors in success and safety of a stack test. This includes providing adequate manpower for the task.
3. Improper design, construction, or layout. Design aspects relate to equipment used and plan of operation.
4. Protective devices or proper tools and equipment not provided. "Jerry rigging" and "making do" should only occur under unusual circumstances, not as standard practice.
5. Failure on the part of any personnel to follow rules or instructions. Safety is the responsiblity of each individual for himself and others around him. Personal disregard for safety rules jeopardizes the safety of all.

6. Neglect or improper use of protective devices, job equipment, or materials.
7. Faulty, improperly maintained devices. Poorly maintained job equipment is inexcusable.
8. Personnel without adequate knowledge or training for performing job. tasks. All present should be capable of performing the job tasks assigned. Trainees should be closely supervised.
9 Personnel in poor physical condition or with a poor mental attitude for task. This can have implications for the attitude of personnel toward each other, the supervisor, the task itself, or working conditions.
10. Unpredictable agents outside the organization. This may mean contract personnel who do not abide by standard rules or something as unpredictable as a biting insect or bad weather.

Accident Prevention

Preventing accidents during a stack test begins with advance planning. Knowledge of process operations and important considerations of the site environment will give insight into chemical, mechanical, or electrical hazards that may be present. This knowledge will be useful in deciding on equipment to be used at the site. Knowledge of the weather conditions and logistical constraints further aid in establishing a safe test program. These items in conjunction with evaluation of site safety and first aid facilities will allow preparation of a source sampling experiment.

The source test program will operate at peak efficiency and safety if plans are properly followed. Thorough planning, including contingency actions, eliminates the confusion that often contributes to accidents. This planning must include allotment of sufficient time for completion of the task, taking into account possible delays. Test personnel should be well informed of the program procedures; their input for test performance and safety suggestions will be useful. Having once established an operating plan, all involved should adhere to it closely.

After thorough planning of the test program, attention focuses upon testing and safety equipment and on site operating practices. General comments on equipment preparation apply to both the sampling and safety apparatus. Experimental design and personnel suggestions should indicate what equipment will be needed on the site for all functions. Equipment should be prepared and assembled in advance; it should be checked for suitable operation or potential problems. Equipment that could handle unexpected situations should also be included. Carry only necessary equipment to the site and use it properly.

Work at the site must be organized following standard rules and work the plan carefully followed. Safety equipment should be used and personnel must remain alert to any changes on the site that could effect safe operation. All present should be made aware of any suspected problems.

Summary

The most important factor in any safety program is common sense. Common sense can, however, be an elusive element. Several steps presented in this section can help in developing sensible safety practices. Thorough advance planning and preparation for the jobs at hand begin the process of good safety practice. Informing involved personnel of all plans and using their suggestions about work safety increases the effectiveness of the planning. Analyzing a work situation for hazards, including past problems, into a coherent, organized safety program, usually results in common sense corrective procedures.

SLIDE 101-0

NOTES

INTRODUCTION TO SOURCE SAMPLING

SLIDE 101-1

PURPOSE OF SOURCE SAMPLING

THE AGENCY

1. Provide data to formulate control strategy.
2. Provide data to evaluate compliance.
3. Provide data upon which regulations can be based.

SLIDE 101-2

PURPOSE OF SOURCE SAMPLING

INDUSTRY

1. Provide information on process operations.
2. Provide information on control device efficiency.
3. Provide information for design of new process and control equipment.

SLIDE 101-3

NOTES

BASIC TERMINOLOGY

CONCENTRATION

Quantity of pollutant per quantity of effluent gas.

grams/cubic foot

STACK GAS FLOW RATE

Quantity of effluent gas per length of time.

cubic feet/hour

POLLUTANT MASS RATE

Quantity of pollutant per length of time.

pounds/hour

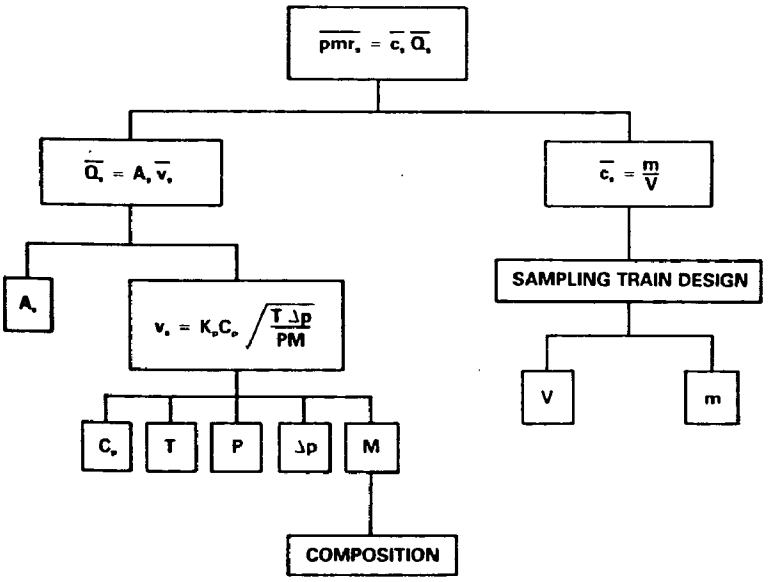
SLIDE 101-4

POLLUTANT MASS RATE EQUATION

$$\overline{Pmr_s} = \overline{C_s}\,\overline{Q_s}$$

SLIDE 101-5

STACK SAMPLING FLOW DIAGRAM



SECTION B. METHOD 1

METHOD 1--SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. To aid in the representative measurement of pollutant emissions and/or total volumetric flow rate from a stationary source, a measurement site where the effluent stream is flowing in a known direction is selected, and the cross-section of the stack is divided into a number of equal areas. A traverse point is then located within each of these equal areas.

1.2 Applicability. This method is applicable to flowing gas streams in ducts, stacks, and flues. The method cannot be used when: (1) flow is cyclonic or swirling (see Section 2.4), (2) a stack is smaller than about 0.30 meter (12 in.) in diameter, or 0.071 m^2 (113 $in.^2$) in cross-sectional area, or (3) the measurement site is less than two stack or duct diameters downstream or less than a half diameter upstream from a flow disturbance.

The requirements of this method must be considered before construction of a new facility from which emissions will be measured; failure to do so may require subsequent alterations to the stack or deviation from the standard procedure. Cases involving variants are subject to approval by the Administrator, U.S. Environmental Protection Agency.

2. Procedure

2.1 Selection of Measurement Site. Sampling or velocity measurement is performed at a site located at least eight stack or duct

diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, or contraction in the stack, or from a visible flame. If necessary, an alternative location may be selected, at a position at least two stack or duct diameters downstream and a half diameter upstream from any flow disturbance. For a rectangular cross section, an equivalent diameter (D_e) shall be calculated from the following equation, to determine the upstream and downstream distances:

$$D_e = \frac{2LW}{L + W}$$

where L = length and W = width.

2.2 Determining the Number of Traverse Points.

2.2.1 Particulate Traverses. When the eight- and two-diameter criterion can be met, the minimum number of traverse points shall be: (1) twelve, for circular or rectangular stacks with diameters (or equivalent diameters) greater than 0.61 meter (24 in.); (2) eight, for circular stacks with diameters between 0.30 and 0.61 meter (12-24 in.); and (3) nine, for rectangular stacks with equivalent diameters between 0.30 and 0.61 meter (12-24 in.).

When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1-1. Before referring to the figure, however, determine the distances from the chosen measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter or equivalent diameter, to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1-1 the minimum number

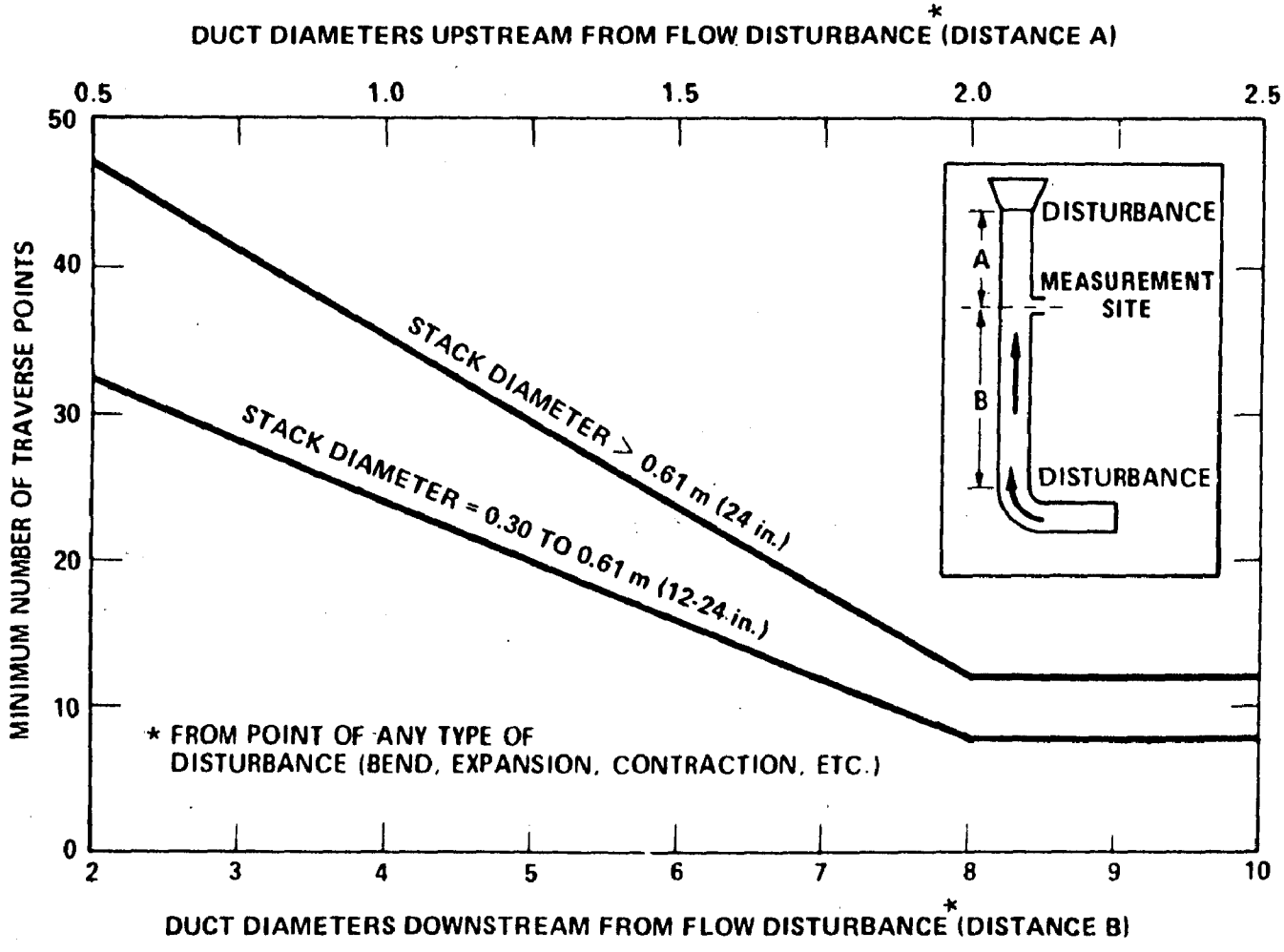


Figure 1-1. Minimum number of traverse points for particulate traverses.

of traverse points that corresponds: (1) to the number of duct diameters upstream; and (2) to the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, so that for circular stacks the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1-1.

Table 1-1. CROSS-SECTIONAL LAYOUT FOR RECTANGULAR STACKS

No. of traverse points	Matrix layout
9	3 x 3
12	4 x 3
16	4 x 4
20	5 x 4
25	5 x 5
30	6 x 5
36	6 x 6
42	7 x 6
49	7 x 7

2.2.2 Velocity (Non-Particulate) Traverses. When velocity or volumetric flow rate is to be determined (but not particulate matter), the same procedure as that for particulate traverses (Section 2.2.1) is followed, except that Figure 1-2 may be used instead of Figure 1-1.

2.3 Cross-Sectional Layout and Location of Traverse Points.

2.3.1 Circular Stacks. Locate the traverse points on two perpendicular diameters according to Table 1-2 and the example shown in Figure 1-3. Any equation (for examples, see Citations 2 and 3 in the

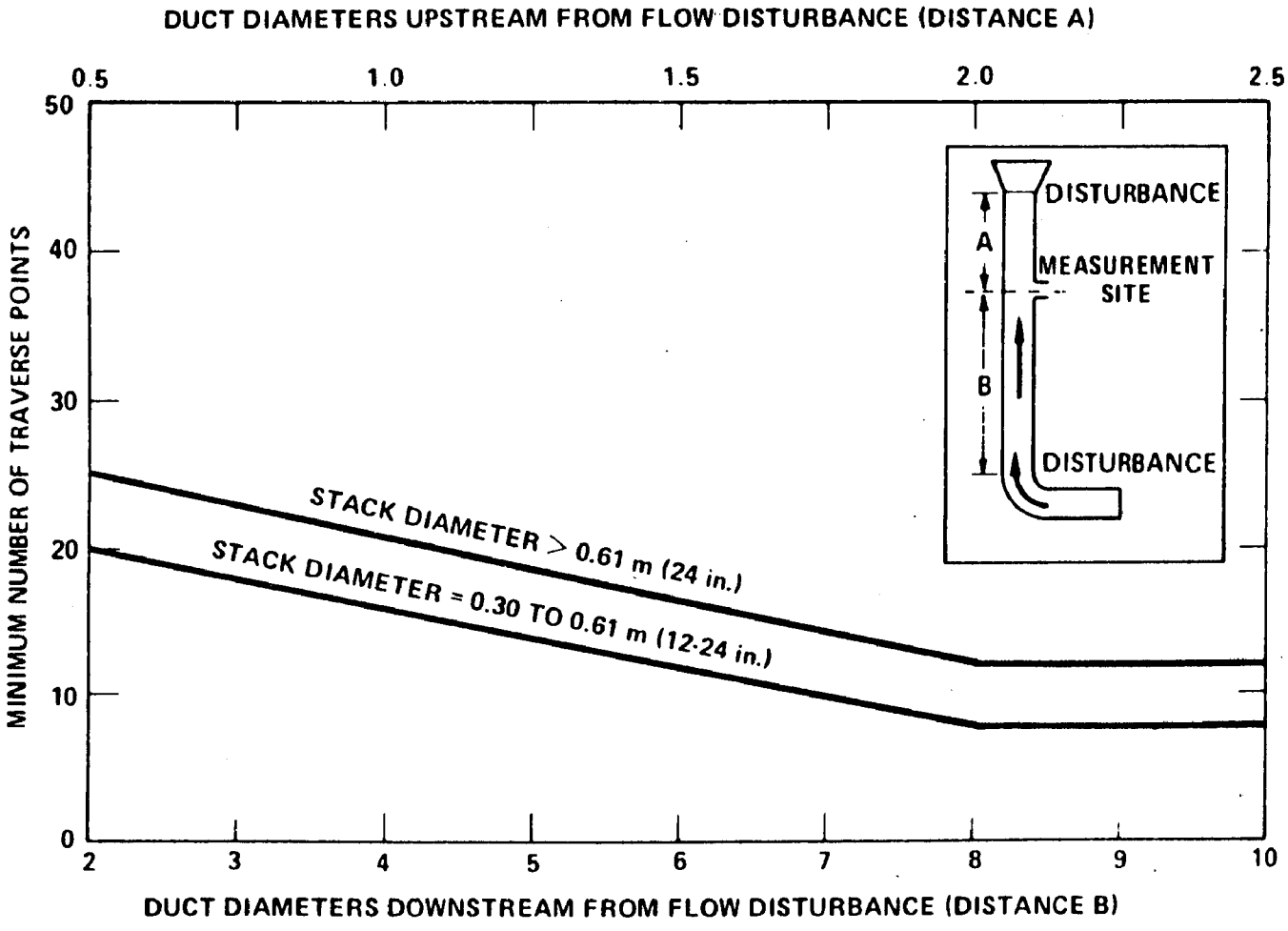


Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.

TRAVERSE POINT	DISTANCE, % of diameter
1	4.4
2	14.7
3	29.5
4	70.5
5	85.3
6	95.6

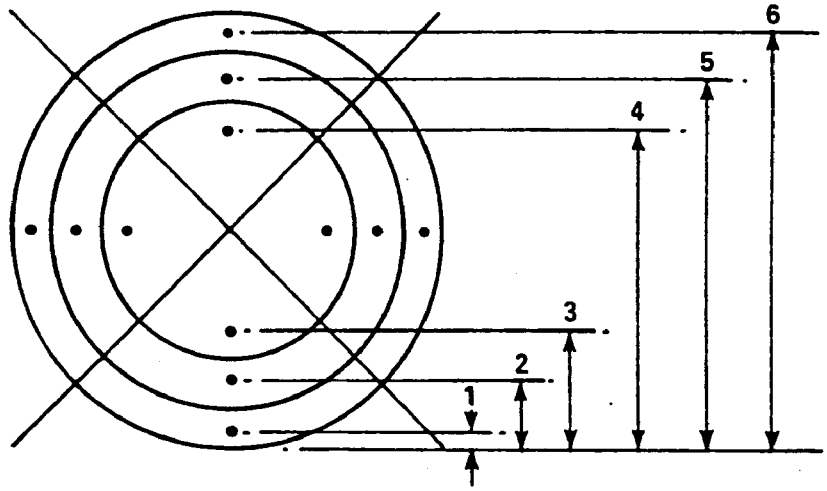


Figure 1-3. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points indicated.

Table 1-2. LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS

(Percent of stack diameter from inside wall to traverse point)

Traverse point number on a diameter	Number of traverse points on a diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.2	2.6	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.6	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.6	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.4	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.4	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5
6			95.6	80.6	65.8	35.6	26.9	22.0	18.8	16.5	14.6	13.2
7				89.5	77.4	64.4	36.6	28.3	23.6	20.4	18.0	16.1
8				96.8	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4
9					91.8	82.3	73.1	62.5	38.2	30.6	26.2	23.0
10					97.4	88.2	79.9	71.7	61.8	38.8	31.5	27.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2
14							98.2	91.5	85.4	79.6	73.8	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.5	92.1
22											98.9	94.5
23												96.8
24												98.9

Bibliography) that gives the same values as those in Table 1-2 may be used in lieu of Table 1-2.

For particulate traverses, one of the diameters must be in a plane containing the greatest expected concentration variation, e.g., after bends, one diameter shall be in the plane of the bend. This requirement becomes less critical as the distance from the disturbance increases; therefore, other diameter locations may be used, subject to approval of the Administrator.

In addition, for stacks having diameters greater than 0.61 m (24 in.) no traverse points shall be located within 2.5 centimeters (1.00 in.) of the stack walls; and for stack diameters equal to or less than 0.61 m (24 in.), no traverse points shall be located within 1.3 cm (0.50 in.) of the stack walls. To meet these criteria, observe the procedures given below.

2.3.1.1 Stacks With Diameters Greater Than 0.61 m (24 in.). When any of the traverse points as located in Section 2.3.1 fall within 2.5 cm (1.00 in.) of the stack walls, relocate them away from the stack walls to: (1) a distance of 2.5 cm (1.00 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger. These relocated traverse points (on each end of a diameter) shall be the "adjusted" traverse points.

Whenever two successive traverse points are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sampling (or velocity measurement) procedure, and in recording the data.

2.3.1.2 Stacks With Diameters Equal to or Less Than 0.61 m (24 in.). Follow the procedure in Section 2.3.1.1, noting only that any "adjusted" points should be relocated away from the stack walls to: (1) a distance of 1.3 cm (0.50 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger.

2.3.2 Rectangular Stacks. Determine the number of traverse points as explained in Sections 2.1 and 2.2 of this method. From Table 1-1, determine the grid configuration. Divide the stack cross-section into as many equal rectangular elemental areas as traverse points, and then locate a traverse point at the centroid of each equal area according to the example in Figure 1-4.

If the tester desires to use more than the minimum number of traverse points, expand the "minimum number of traverse points" matrix (see Table 1-1) by adding the extra traverse points along one or the other or both legs of the matrix; the final matrix need not be balanced. For example, if a 4 x 3 "minimum number of points" matrix were expanded to 36 points, the final matrix could be 9 x 4 or 12 x 3, and would not necessarily have to be 6 x 6. After constructing the final matrix, divide the stack cross-section into as many equal rectangular, elemental areas as traverse points, and locate a traverse point at the centroid of each equal area.

The situation of traverse points being too close to the stack walls is not expected to arise with rectangular stacks. If this problem should ever arise, the Administrator must be contacted for resolution of the matter.

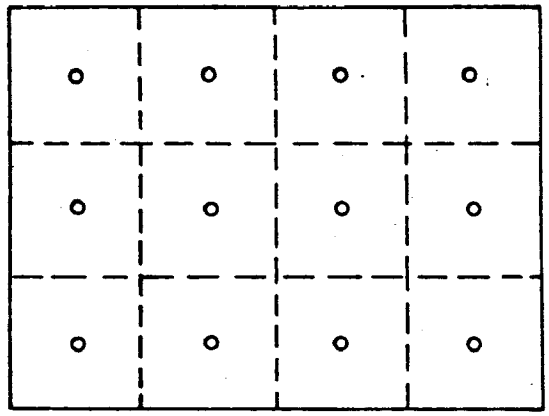


Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with a traverse point at centroid of each area.

2.4 Verification of Absence of Cyclonic Flow. In most stationary sources, the direction of stack gas flow is essentially parallel to the stack walls. However, cyclonic flow may exist (1) after such devices as cyclones and inertial demisters following venturi scrubbers, or (2) in stacks having tangential inlets or other duct configurations which tend to induce swirling; in these instances, the presence or absence of cyclonic flow at the sampling location must be determined. The following techniques are acceptable for this determination.

Level and zero the manometer. Connect a Type S pitot tube to the manometer. Position the Type S pitot tube at each traverse point, in succession, so that the planes of the face openings of the pitot tube are perpendicular to the stack cross-sectional plane: when the Type S pitot tube is in this position, it is at "0° reference." Note the differential pressure (Δp) reading at each traverse point. If a null (zero) pitot reading is obtained at 0° reference at a given traverse point, an acceptable flow condition exists at that point. If the pitot reading is not zero at 0° reference, rotate the pitot tube (up to $\pm 90°$ yaw angle), until a null reading is obtained. Carefully determine and record the value of the rotation angle (α) to the nearest degree. After the null technique has been applied at each traverse point, calculate the average of the absolute values of α; assign α values of 0° to those points for which no rotation was required, and include these in the overall average. If the average value of α is greater than 10°, the overall flow condition in the stack is unacceptable and alternative methodology, subject to the approval of the Administrator, must be used to perform accurate sample and velocity traverses.

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SLIDE 102-0

NOTES

METHOD — 1

Sample and Velocity Traverses
for Stationary Sources

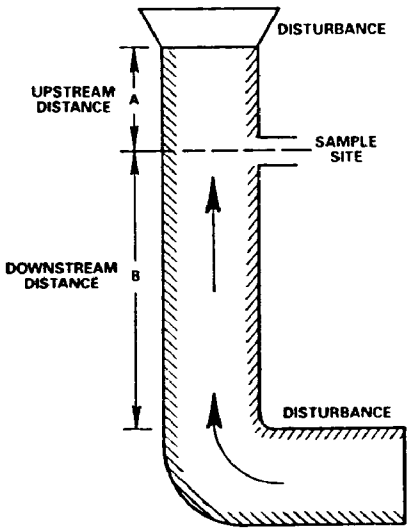
SLIDE 102-1

CRITERIA FOR SELECTION OF MEASUREMENT SITE

- Effluent stream must be flowing in a known direction.
- Ideally, site should be at least eight stack diameters downstream and two diameters upstream from any flow disturbance.
- Alternatively, site must be located at a minimum of two stack diameters downstream and one-half diameter upstream from any flow disturbance.

SLIDE 102-2

UPSTREAM AND DOWNSTREAM FLOW DISTURBANCES



SLIDE 102-3

NOTES

EXAMPLE FOR CIRCULAR STACKS

Inside of far wall to outside of nipple (distance 1)	=	39"
Inside of near wall to outside of nipple (distance 2)	=	3"
Stack ID (distance 1 - distance 2)	=	36"
Nearest upstream disturbance (distance A)	=	74"
Nearest downstream disturbance (distance B)	=	288"

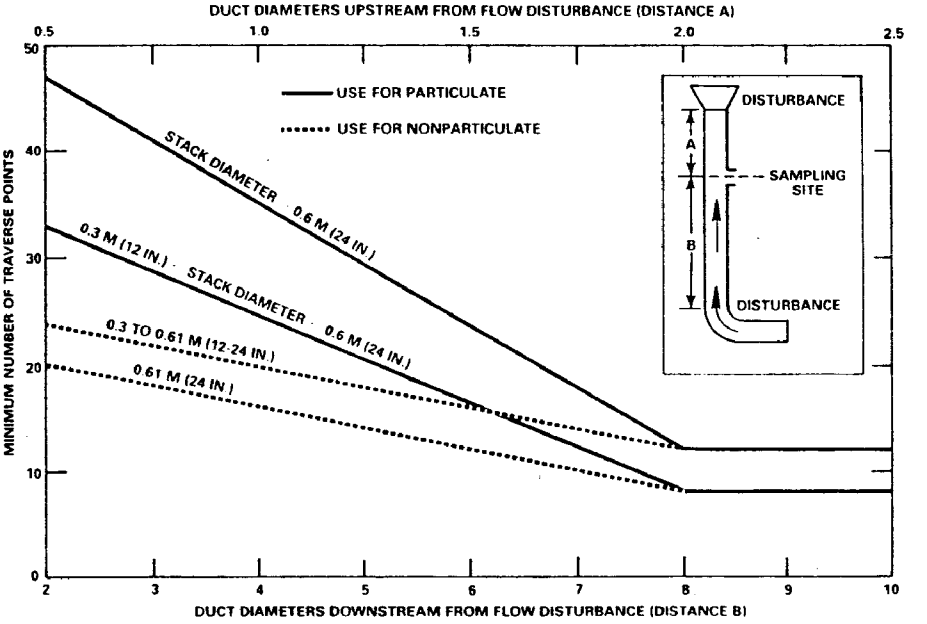
CALCULATION

74" (distance A) ÷ 36" (stack ID) = 2

288" (distance B) ÷ 36" (stack ID) = 8

SLIDE 102-4

MINIMUM NUMBER OF TRAVERSE POINTS



SLIDE 102-5

NOTES

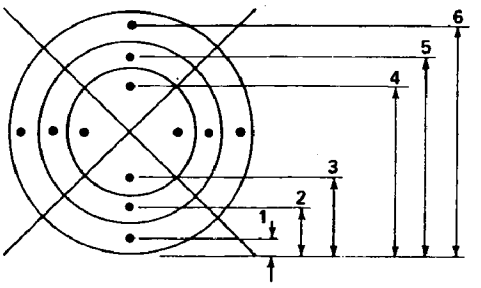
LOCATION OF TRAVERSE POINTS AS A PERCENTAGE OF STACK DIAMETER

Traverse point number on a diameter	Number of traverse points on a diameter					
	2	4	6	8	10	12
1	14.6	6.7	4.4	3.2	2.6	2.1
2	85.4	25.0	14.6	10.5	8.2	6.7
3		75.0	29.6	19.4	14.6	11.8
4		93.3	70.4	32.3	22.6	17.7
5			85.4	67.7	34.2	25.0
6			95.6	80.6	65.8	35.6
7				89.5	77.4	64.4
8				96.8	85.4	75.0
9					91.8	82.3
10					97.4	88.2
11						93.3
12						97.9

SLIDE 102-6

MEASURED LOCATIONS OF TRAVERSE POINTS (CIRCULAR STACK)

TRAVERSE POINT	DISTANCE % DIAMETER	DISTANCE INCHES
1	4.4	1.6
2	14.7	5.3
3	29.5	10.6
4	70.5	25.4
5	85.3	30.7
6	95.6	34.4



SLIDE 102-7

NOTES

CRITERIA FOR LOCATING SAMPLE POINTS IN CIRCULAR STACKS

- One diameter must be in a plane containing greatest expected concentration variation, for particulate traverses.
- No traverse point shall be located within 2.5 cm (1.0 in.) of stack wall for stacks greater than 0.61 m (24 in.) in diameter.
- No traverse point shall be located within 1.3 cm (0.50 in.) of stack wall for stacks with diameters $\leq$ 0.61 m (24 in.).

SLIDE 102-8

(cont.)

- Relocated traverse points shall be defined as "adjusted" traverse points.
- When two traverse points are combined to form a single adjusted traverse point, treat adjusted point as two points.

SLIDE 102-9

BALANCED MATRIX SCHEME FOR RECTANGULAR STACKS

Number of Traverse Points	Matrix Layout
9	3 x 3
12	4 x 3
16	4 x 4
20	5 x 4
25	5 x 5
30	6 x 5
36	6 x 6
42	7 x 6
49	7 x 7

EXAMPLE FOR RECTANGULAR STACKS

Dimensions:

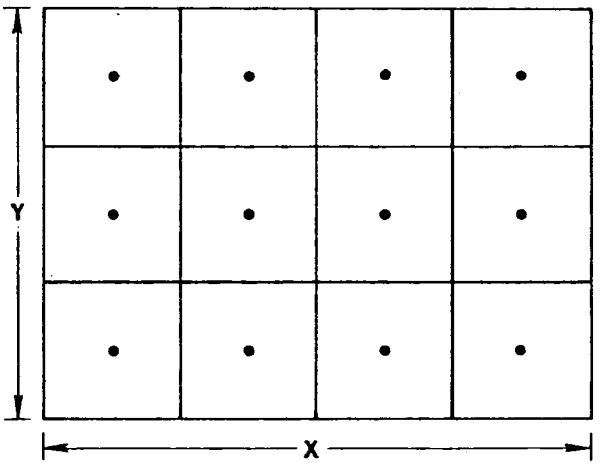
Length of stack (L) (- wall thickness) = 36"
Width of stack (W) (- wall thickness) = 36"
Equivalent Diameter (De)

$$De = \frac{2LW}{L + W} = 2\frac{36 \times 36}{36 + 36} = 36"$$

Nipple Length = 0
Nearest upstream disturbance (distance A) = 74"
Nearest downstream disturbance (distance B) = 288"
74" (distance A) ÷ 36" (stack De) = 2
288" (distance B) ÷ 36" (stack De) = 8

SLIDE 102-11

TRAVERSE POINT LOCATIONS (RECTANGULAR STACK)



SLIDE 102-12

NOTES

MEASURED LOCATIONS OF TRAVERSE POINTS (RECTANGULAR STACK)

Point	Length of nipple	Distance X	Distance Y
1-1	0	4.5"	6"
1-2	0	4.5"	18"
1-3	0	4.5"	30"
2-1	0	13.5"	6"
2-2	0	13.5"	18"
2-3	0	13.5"	30"
3-1	0	22.5"	6"
3-2	0	22.5"	18"
3-3	0	22.5"	30"
4-1	0	31.5"	6"
4-2	0	31.5"	18"
4-3	0	31.5"	30"

SLIDE 102-13

NON PARALLEL FLOW

Presence or absence of non parallel flow must be determined after:

- Cyclones
- Inertial Demisters
- Stacks Having Tangential Inlets

SLIDE 102-14

VERIFY SUITABILITY OF SITE

1. Calculate absolute value of α.
2. If average value of α is $> 10°$, flow conditions are unacceptable.

SECTION C. METHOD 2

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

1. Principle and Applicability

1.1 Principle. The average gas velocity in a stack is determined from the gas density and from measurement of the average velocity head with a Type S (Stausscheibe or reverse type) pitot tube.

1.2 Applicability. This method is applicable for measurement of the average velocity of a gas stream and for quantifying gas flow.

This procedure is not applicable at measurement sites which fail to meet the criteria of Method 1, Section 2.1. Also, the method cannot be used for direct measurement in cyclonic or swirling gas streams; Section 2.4 of Method 1 shows how to determine cyclonic or swirling flow conditions. When unacceptable conditions exist, alternative procedures, subject to the approval of the Administrator, U. S. Environmental Protection Agency, must be employed to make accurate flow rate determinations; examples of such alternative procedures are: (1) to install straightening vanes; (2) to calculate the total volumetric flow rate stoichiometrically, or (3) to move to another measurement site at which the flow is acceptable.

2. Apparatus

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

2.1 Type S Pitot Tube. The Type S pitot tube (Figure 2-1) shall be made of metal tubing (e.g., stainless steel). It is recommended that the external tubing diameter (dimension D_t, Figure 2-2b) be

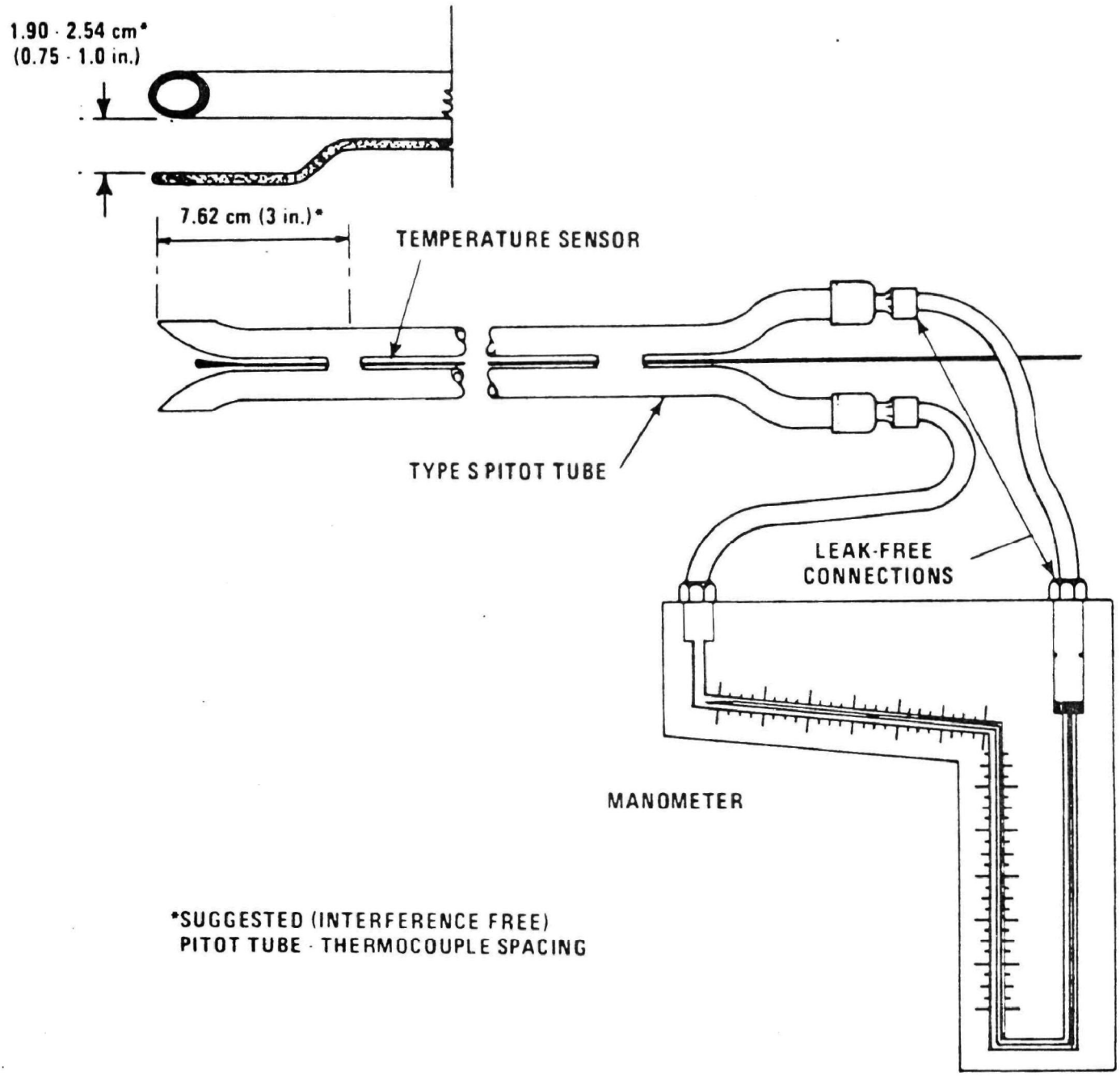


Figure 2-1. Type S pitot tube manometer assembly.

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 tube to its face-opening plane (dimensions P_A and P_B, Figure 2-2b); it is recommended that this distance be between 1.05 and 1.50 times the external tubing diameter. The face openings of the pitot tube shall, preferably, be aligned as shown in Figure 2-2; however, slight misalignments of the openings are permissible (see Figure 2-3).

The Type S pitot tube shall have a known coefficient, determined as outlined in Section 4. An identification number shall be assigned to the pitot tube; this number shall be permanently marked or engraved on the body of the tube.

A standard pitot tube may be used instead of a Type S, provided that it meets the specifications of Sections 2.7 and 4.2; note, however, that the static and impact pressure holes of standard pitot tubes are susceptible to plugging in particulate-laden gas streams. Therefore, whenever a standard pitot tube is used to perform a traverse, adequate proof must be furnished that the openings of the pitot tube have not plugged up during the traverse period; this can be done by taking a velocity head (Δp) reading at the final traverse point, cleaning out the impact and static holes of the standard pitot tube by "back-purging" with pressurized air, and then taking another Δp reading. If the Δp readings made before and after the air purge are the same (± 5 percent), the traverse is acceptable. Otherwise, reject the run. Note that if Δp at the final traverse point is unsuitably low, another point may be selected. If "back-purging" at regular intervals

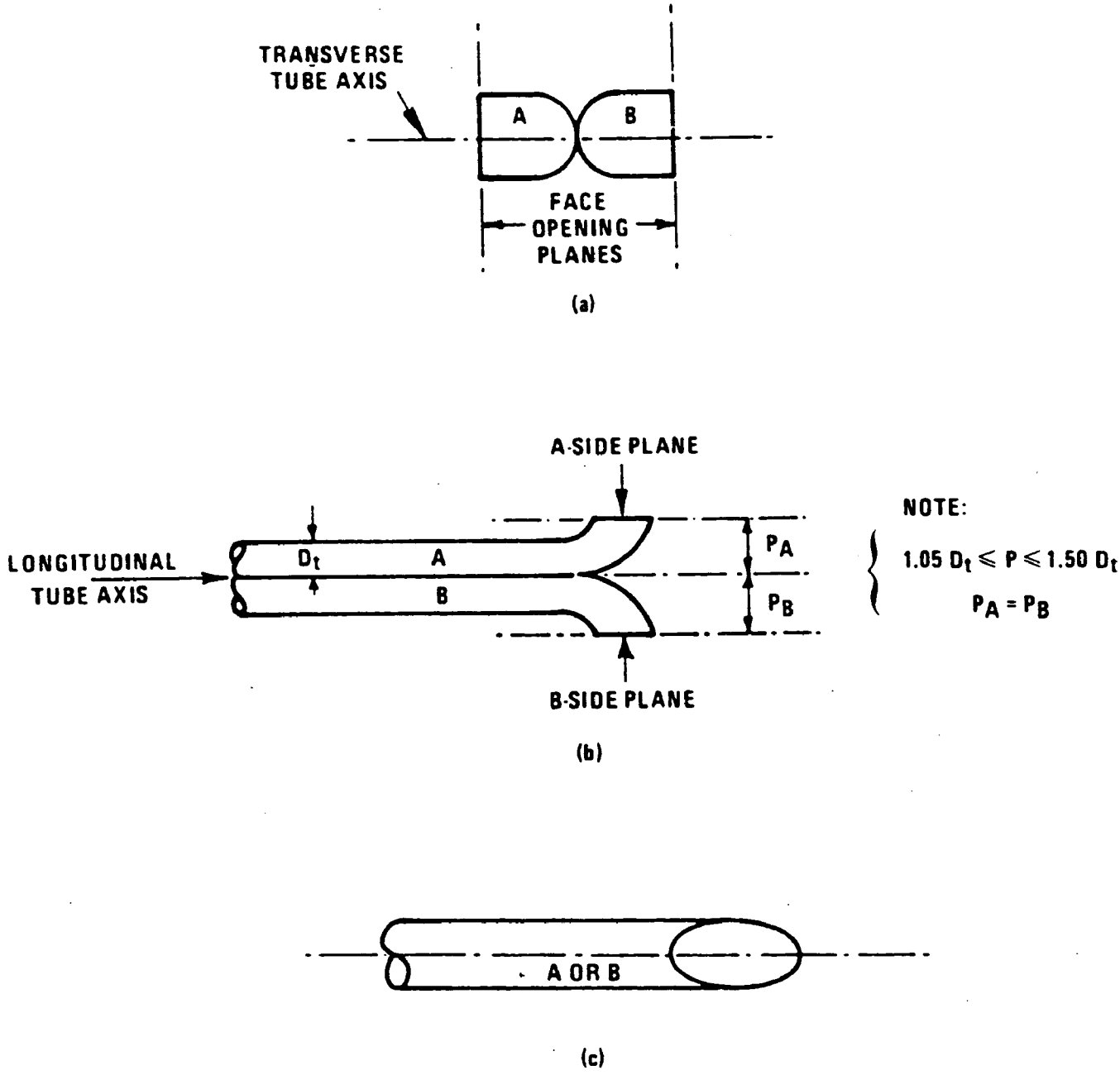


Figure 2-2. Properly constructed Type S pitot tube, shown in: (a) end view; face opening planes perpendicular to transverse axis; (b) top view; face opening planes parallel to longitudinal axis; (c) side view; both legs of equal length and centerlines coincident, when viewed from both sides. Baseline coefficient values of 0.84 may be assigned to pitot tubes constructed this way.

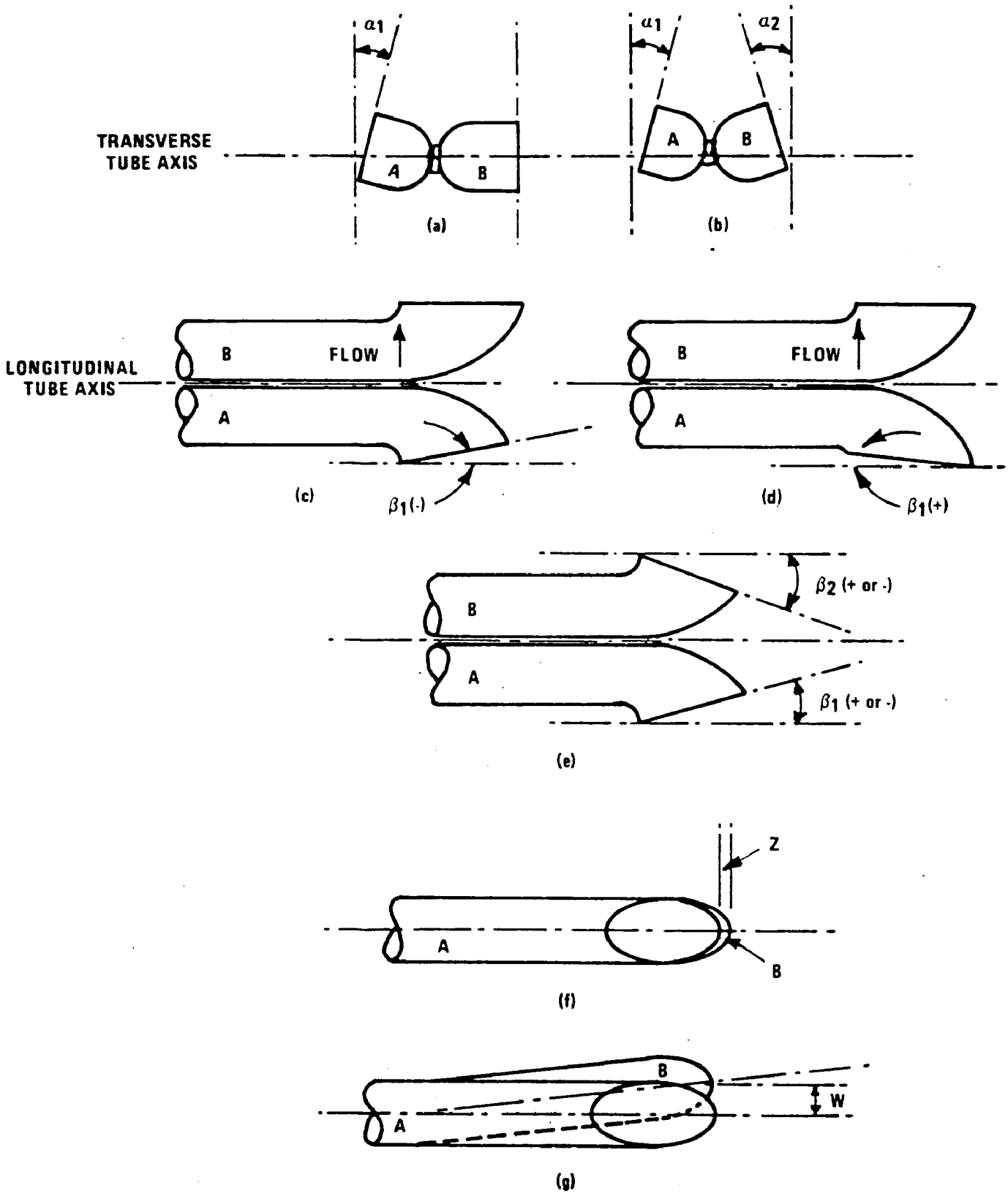


Figure 2-3. Types of face-opening misalignment that can result from field use or improper construction of Type S pitot tubes. These will not affect the baseline value of $\bar{C}p(s)$ so long as α_1 and $\alpha_2 < 10^o$, β_1 and $\beta_2 < 5^o$, $z < 0.32$ cm (1/8 in.) and $w < 0.08$ cm (1/32 in.) (citation 11 in Section 6).

is part of the procedure, then comparative Δp readings shall be taken, as above, for the last two back purges at which suitably high Δp readings are observed.

2.2 Differential Pressure Gauge. An inclined manometer or equivalent device is used. Most sampling trains are equipped with a 10-in. (water column) inclined-vertical manometer, having 0.01-in. H_2O divisions on the 0- to 1-in. inclined scale, and 0.1-in. H_2O divisions on the 1- to 10-in. vertical scale. This type of manometer (or other gauge of equivalent sensitivity) is satisfactory for the measurement of Δp values as low as 1.3 mm (0.05 in.) H_2O. However, a differential pressure gauge of greater sensitivity shall be used (subject to the approval of the Administrator), if any of the following is found to be true: (1) the arithmetic average of all Δp readings at the traverse points in the stack is less than 1.3 mm (0.05 in.) H_2O; (2) for traverses of 12 or more points, more than 10 percent of the individual Δp readings are below 1.3 mm (0.05 in.) H_2O; (3) for traverses of fewer than 12 points, more than one Δp reading is below 1.3 mm (0.05 in.) H_2O. Citation 18 in Section 6 describes commercially available instrumentation for the measurement of low-range gas velocities.

As an alternative to criteria (1) through (3) above, the following calculation may be performed to determine the necessity of using a more sensitive differential pressure gauge:

$$T = \frac{\sum_{i=1}^{n} \sqrt{\Delta p_i + K}}{\sum_{i=1}^{n} \sqrt{\Delta p_i}}$$

where:

Δp_i = Individual velocity head reading at a traverse point, mm H_2O (in. H_2O)

n = Total number of traverse points

K = 0.13 mm H_2O when metric units are used and 0.005 in. H_2O when English units are used

If T is greater than 1.05, the velocity head data are unacceptable and a more sensitive differential pressure gauge must be used.

Note: If differential pressure gauges other than inclined manometers are used (e.g., magnehelic gauges), their calibration must be checked after each test series. To check the calibration of a differential pressure gauge, compare Δp readings of the gauge with those of a gauge-oil manometer at a minimum of three points, approximately representing the range of Δp values in the stack. If, at each point, the values of Δp as read by the differential pressure gauge and gauge-oil manometer agree to within 5 percent, the differential pressure gauge shall be considered to be in proper calibration. Otherwise, the test series shall either be voided, or procedures to adjust the measured Δp values and final results shall be used, subject to the approval of the Administrator.

2.3 Temperature Gauge. A thermocouple, liquid-filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauge capable of measuring temperature to within 1.5 percent of the minimum absolute stack temperature shall be used. The temperature gauge shall be attached to the pitot tube such that the sensor tip does not touch any metal; the gauge shall be in an interference-free arrangement with respect to the pitot tube face openings (see Figure 2-1 and also Figure 2-7 in Section 4). Alternate positions may be used if the pitot tube-temperature gauge system is calibrated according to the procedure

of Section 4. Provided that a difference of not more than 1 percent in the average velocity measurement is introduced, the temperature gauge need not be attached to the pitot tube; this alternative is subject to the approval of the Administrator.

2.4 Pressure Probe and Gauge. A piezometer tube and mercury- or water-filled U-tube manometer capable of measuring stack pressure to within 2.5 mm (0.1 in.) Hg is used. The static tap of a standard type pitot tube or one leg of a Type S pitot tube with the face opening planes positioned parallel to the gas flow may also be used as the pressure probe.

2.5 Barometer. A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg) may be used. In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm (0.1 in.) Hg per 30 meter (100 foot) elevation increase, or vice-versa for elevation decrease.

2.6 Gas Density Determination Equipment. Method 3 equipment, if needed (see Section 3.6), to determine the stack gas dry molecular weight, and Reference Method 4 or Method 5 equipment for moisture content determination; other methods may be used subject to approval of the Administrator.

2.7 Calibration Pitot Tube. When calibration of the Type S pitot tube is necessary (see Section 4), a standard pitot tube is used as a reference. The standard pitot tube shall, preferably, have a known

coefficient, obtained either (1) directly from the National Bureau of Standards, Route 270, Quince Orchard Road, Gaithersburg, Maryland, or (2) by calibration against another standard pitot tube with an NBS-traceable coefficient. Alternatively, a standard pitot tube designed according to the criteria given in 2.7.1 through 2.7.5 below and illustrated in Figure 2-4 (see also Citations 7, 8, and 17 in Section 6) may be used. Pitot tubes designed according to these specifications will have baseline coefficients of about 0.99 $\pm$ 0.01.

2.7.1 Hemispherical (shown in Figure 2-4), ellipsoidal, or conical tip.

2.7.2 A minimum of six diameters straight run (based upon D, the external diameter of the tube) between the tip and the static pressure holes.

2.7.3 A minimum of eight diameters straight run between the static pressure holes and the centerline of the external tube, following the 90 degree bend.

2.7.4 Static pressure holes of equal size (approximately 0.1 D), equally spaced in a piezometer ring configuration.

2.7.5 Ninety degree bend, with curved or mitered junction.

2.8 Differential Pressure Gauge for Type S Pitot Tube Calibration. An inclined manometer or equivalent is used. If the single-velocity calibration technique is employed (see Section 4.1.2.3), the calibration differential pressure gauge shall be readable to the nearest 0.13 mm H_2O (0.005 in. H_2O). For multivelocity calibrations, the gauge shall be readable to the nearest 0.13 mm H_2O (0.005 in H_2O) for Δp values between 1.3 and 25 mm H_2O (0.05 and 1.0 in. H_2O), and to the nearest 1.3 mm H_2O (0.05 in. H_2O) for Δp values above 25 mm H_2O (1.0 in. H_2O). A special, more sensitive gauge will be required to read Δp values below 1.3 mm H_2O [0.05 in. H_2O](see Citation 18 in Section 6).

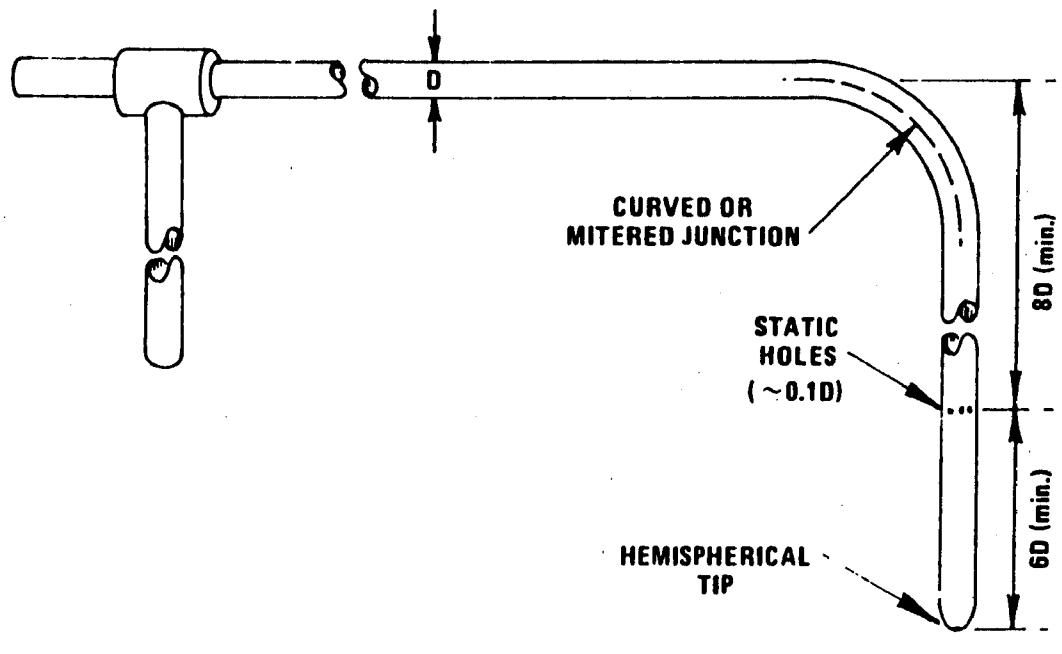


Figure 2-4. Standard pitot tube design specifications.

3. Procedure

3.1 Set up the apparatus as shown in Figure 2-1. Capillary tubing or surge tanks installed between the manometer and pitot tube may be used to dampen Δp fluctuations. It is recommended, but not required, that a pretest leak-check be conducted, as follows: (1) blow through the pitot impact opening until at least 7.6 cm (3 in.) H_2O velocity pressure registers on the manometer; then, close off the impact opening. The pressure shall remain stable for at least 15 seconds; (2) do the same for the static pressure side, except using suction to obtain the minimum of 7.6 cm (3 in.) H_2O. Other leak-check procedures, subject to the approval of the Administrator, may be used.

3.2 Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse. Record all necessary data as shown in the example data sheet (Figure 2-5).

3.3 Measure the velocity head and temperature at the traverse points specified by Method 1. Ensure that the proper differential pressure gauge is being used for the range of Δp values encountered (see Section 2.2). If it is necessary to change to a more sensitive gauge, do so, and remeasure the Δp and temperature readings at each traverse point. Conduct a post-test leak-check (mandatory), as described in Section 3.1 above, to validate the traverse run.

3.4 Measure the static pressure in the stack. One reading is usually adequate.

3.5 Determine the atmospheric pressure.

3.6 Determine the stack gas dry molecular weight. For combustion processes or processes that emit essentially CO_2, O_2, CO, and N_2, use

PLANT ____________________

DATE ____________ RUN NO. ____________

STACK DIAMETER OR DIMENSIONS, m(in.) ____________

BAROMETRIC PRESSURE, mm Hg (in. Hg) ____________

CROSS SECTIONAL AREA, m^2(ft^2) ____________

OPERATORS ____________

PITOT TUBE I.D. NO. ____________

AVG. COEFFICIENT, C_p = ____________

LAST DATE CALIBRATED ____________

SCHEMATIC OF STACK CROSS SECTION

Traverse Pt. No.	Vel. Hd., Δp mm (in.) H_2O	Stack Temperature		P_g mm Hg (in.Hg)	$\sqrt{\Delta p}$
		t_s, °C (°F)	T_s, °K (°R)		
		Average			

Figure 2-5. Velocity traverse data.

Method 3. For processes emitting essentially air, an analysis need not be conducted; use a dry molecular weight of 29.0. For other processes, other methods, subject to the approval of the Administrator, must be used.

3.7 Obtain the moisture content from Reference Method 4 (or equivalent) or from Method 5.

3.8 Determine the cross-sectional area of the stack or duct at the sampling location. Whenever possible, physically measure the stack dimensions rather than using blueprints.

4. Calibration

4.1 Type S Pitot Tube. Before its initial use, carefully examine the Type S pitot tube in top, side, and end views to verify that the face openings of the tube are aligned within the specifications illustrated in Figure 2-2 or 2-3. The pitot tube shall not be used if it fails to meet these alignment specifications.

After verifying the face opening alignment, measure and record the following dimensions of the pitot tube: (a) the external tubing diameter (dimension D_t, Figure 2-2b); and (b) the base-to-opening plane distances (dimensions P_A and P_B, Figure 2-2b). If D_t is between 0.48 and 0.95 cm (3/16 and 3/8 in.), and if P_A and P_B are equal and between 1.05 and 1.50 D_t, there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, or (2) a baseline (isolated tube) coefficient value of 0.84 may be assigned to the pitot tube. Note, however, that if the pitot tube is part of an assembly, calibration may still be required, despite knowledge of the baseline coefficient value (see Section 4.1.1).

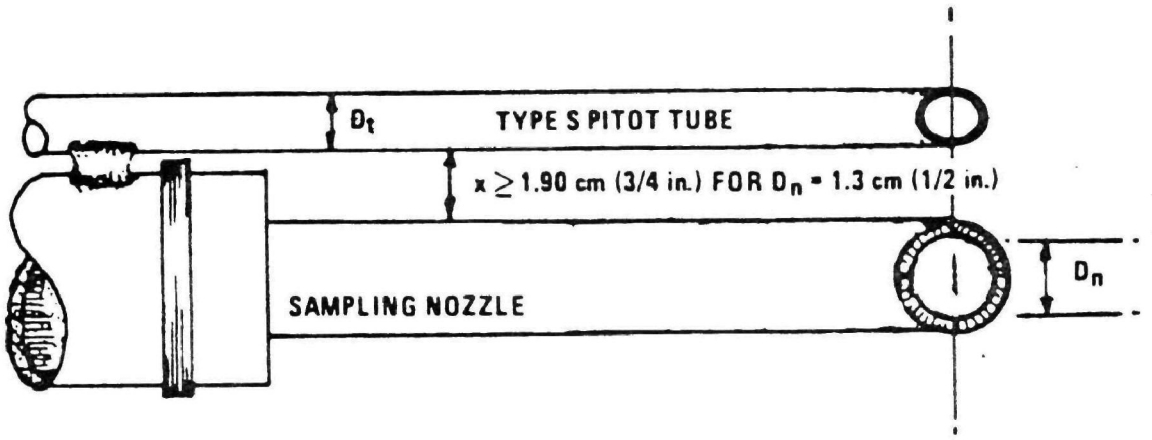
If D_t, P_A, and P_B are outside the specified limits, the pitot tube must be calibrated as outlined in 4.1.2 through 4.1.5 below.

4.1.1 Type S Pitot Tube Assemblies. During sample and velocity traverses, the isolated Type S pitot tube is not always used; in many instances, the pitot tube is used in combination with other source-sampling components (thermocouple, sampling probe, nozzle) as part of an "assembly." The presence of other sampling components can sometimes affect the baseline value of the Type S pitot tube coefficient (Citation 9 in Section 6); therefore an assigned (or otherwise known) baseline coefficient value may or may not be valid for a given assembly. The baseline and assembly coefficient values will be identical only when the relative placement of the components in the assembly is such that aerodynamic interference effects are eliminated. Figures 2-6 through 2-8 illustrate interference-free component arrangements for Type S pitot tubes having external tubing diameters between 0.48 and 0.95 cm (3/16 and 3/8 in.). Type S pitot tube assemblies that fail to meet any or all of the specifications of Figures 2-6 through 2-8 shall be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, and prior to calibration, the values of the intercomponent spacings (pitot-nozzle, pitot-thermocouple, pitot-probe sheath) shall be measured and recorded.

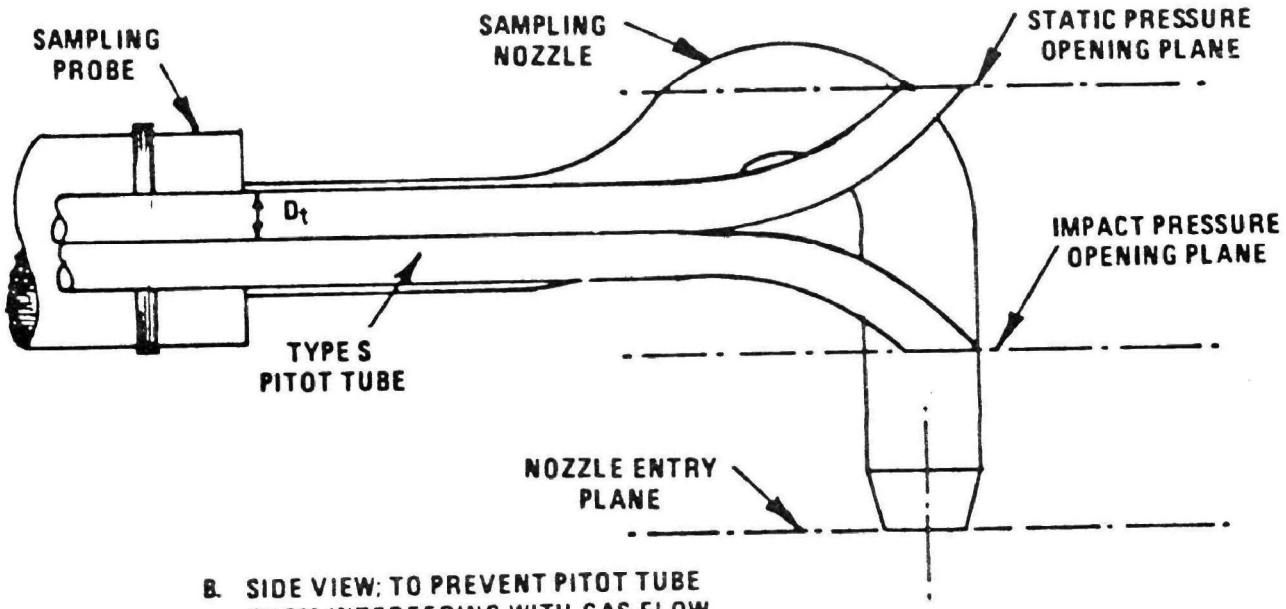
Note: Do not use any Type S pitot tube assembly which is constructed such that the impact pressure opening plane of the pitot tube is below the entry plane of the nozzle (see Figure 2-6b).

4.1.2 Calibration Setup. If the Type S pitot tube is to be calibrated, one leg of the tube shall be permanently marked A, and the other, B. Calibration shall be done in a flow system having the following essential design features:

4.1.2.1 The flowing gas stream must be confined to a duct of definite cross-sectional area, either circular or rectangular. For



A. BOTTOM VIEW; SHOWING MINIMUM PITOT-NOZZLE SEPARATION.



B. SIDE VIEW; TO PREVENT PITOT TUBE FROM INTERFERING WITH GAS FLOW STREAMLINES APPROACHING THE NOZZLE, THE IMPACT PRESSURE OPENING PLANE OF THE PITOT TUBE SHALL BE EVEN WITH OR ABOVE THE NOZZLE ENTRY PLANE.

Figure 2-6. Proper pitot tube - sampling nozzle configuration to prevent aerodynamic interference; buttonhook - type nozzle; centers of nozzle and pitot opening aligned; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

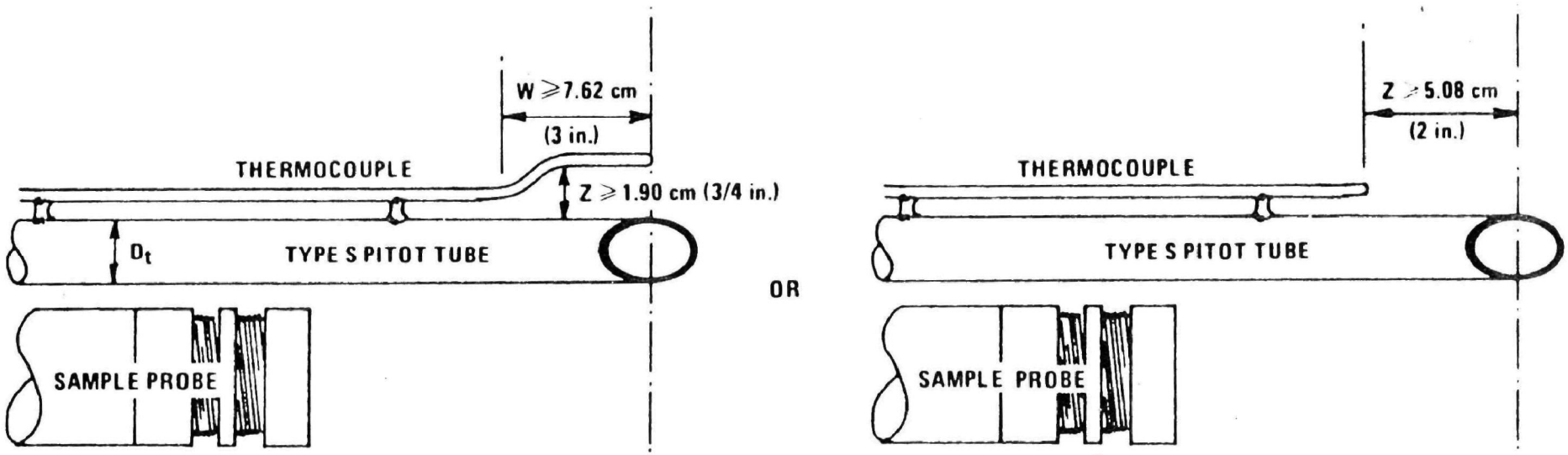


Figure 2-7. Proper thermocouple placement to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

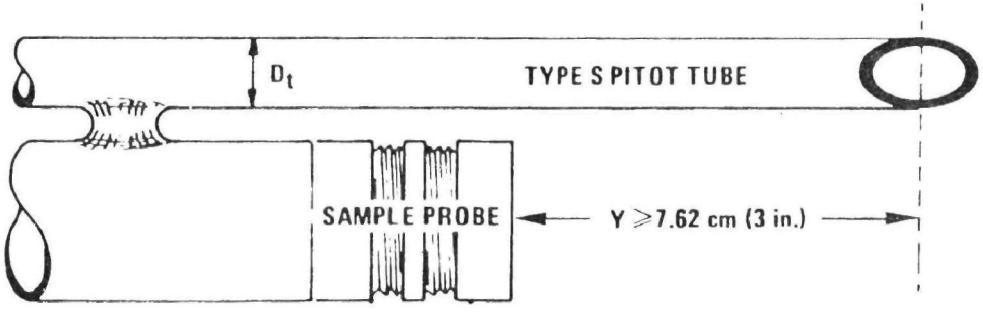


Figure 2-8. Minimum pitot-sample probe separation needed to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

circular cross-sections, the minimum duct diameter shall be 30.5 cm (12 in.); for rectangular cross-sections, the width (shorter side) shall be at least 25.4 cm (10 in.).

4.1.2.2 The cross-sectional area of the calibration duct must be constant over a distance of 10 or more duct diameters. For a rectangular cross-section, use an equivalent diameter, calculated from the following equation, to determine the number of duct diameters:

$$D_e = \frac{2LW}{(L + W)} \qquad \text{Equation 2-1}$$

where:

D_e = Equivalent diameter

L = Length

W = Width

To ensure the presence of stable, fully developed flow patterns at the calibration site, or "test section," the site must be located at least eight diameters downstream and two diameters upstream from the nearest disturbances.

Note: The eight- and two-diameter criteria are not absolute; other test section locations may be used (subject to approval of the Administrator), provided that the flow at the test site is stable and demonstrably parallel to the duct axis.

4.1.2.3 The flow system shall have the capacity to generate a test-section velocity around 915 m/min (3000 ft/min). This velocity must be constant with time to guarantee steady flow during calibration. Note that Type S pitot tube coefficients obtained by single-velocity calibration at 915 m/min (3000 ft/min) will generally be valid to within ± 3 percent for the measurement of velocities above 305 m/min

(1000 ft/min) and to within ±5 to 6 percent for the measurement of velocities between 180 and 305 m/min (600 and 1000 ft/min). If a more precise correlation between C_p and velocity is desired, the flow system shall have the capacity to generate at least four distinct, time-invariant test-section velocities covering the velocity range from 180 to 1525 m/min (600 to 5000 ft/min), and calibration data shall be taken at regular velocity intervals over this range (see Citations 9 and 14 in Section 6 for details).

4.1.2.4 Two entry ports, one each for the standard and Type S pitot tubes, shall be cut in the test section; the standard pitot entry port shall be located slightly downstream of the Type S port, so that the standard and Type S impact openings will lie in the same cross-sectional plane during calibration. To facilitate alignment of the pitot tubes during calibration, it is advisable that the test section be constructed of plexiglas or some other transparent material.

4.1.3 Calibration Procedure. Note that this procedure is a general one and must not be used without first referring to the special considerations presented in Section 4.1.5. Note also that this procedure applies only to single-velocity calibration. To obtain calibration data for the A and B sides of the Type S pitot tube, proceed as follows:

4.1.3.1 Make sure that the manometer is properly filled and that the oil is free from contamination and is of the proper density. Inspect and leak-check all pitot lines; repair or replace if necessary.

4.1.3.2 Level and zero the manometer. Turn on the fan and allow the flow to stabilize. Seal the Type S entry port.

4.1.3.3 Ensure that the manometer is level and zeroed. Position the standard pitot tube at the calibration point (determined as outlined

in Section 4.1.5.1), and align the tube so that its tip is pointed directly into the flow. Particular care should be taken in aligning the tube to avoid yaw and pitch angles. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.4 Read Δp_{std} and record its value in a data table similar to the one shown in Figure 2-9. Remove the standard pitot tube from the duct and disconnect it from the manometer. Seal the standard entry port.

4.1.3.5 Connect the Type S pitot tube to the manometer. Open the Type S entry port. Check the manometer level and zero. Insert and align the Type S pitot tube so that its A side impact opening is at the same point as was the standard pitot tube and is pointed directly into the flow. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.6 Read Δp_s and enter its value in the data table. Remove the Type S pitot tube from the duct and disconnect it from the manometer.

4.1.3.7 Repeat steps 4.1.3.3 through 4.1.3.6 above until three pairs of Δp readings have been obtained.

4.1.3.8 Repeat steps 4.1.3.3 through 4.1.3.7 above for the B side of the Type S pitot tube.

4.1.3.9 Perform calculations, as described in Section 4.1.4 below.

4.1.4 Calculations.

4.1.4.1 For each of the six pairs of Δp readings (i.e., three from side A and three from side B) obtained in Section 4.1.3 above, calculate the value of the Type S pitot tube coefficient as follows:

PITOT TUBE IDENTIFICATION NUMBER: _____ DATE: _____

CALIBRATED BY: _____

	"A" SIDE CALIBRATION			
RUN NO.	Δp_{std} cm H_2O (in. H_2O)	$\Delta p_{(s)}$ cm H_2O (in. H_2O)	$C_{p(s)}$	DEVIATION $C_{p(s)} - \bar{C}_p(A)$
1				
2				
3				
		$\bar{C}_p$ (SIDE A)		

	"B" SIDE CALIBRATION			
RUN NO.	Δp_{std} cm H_2O (in. H_2O)	$\Delta p_{(s)}$ cm H_2O (in. H_2O)	$C_{p(s)}$	DEVIATION $C_{p(s)} - \bar{C}_p(B)$
1				
2				
3				
		$\bar{C}_p$ (SIDE B)		

$$\text{AVERAGE DEVIATION} = \sigma\,(\text{A OR B}) = \frac{\sum_{1}^{3} \left| C_{p(s)} - \bar{C}_p(\text{A OR B}) \right|}{3} \leftarrow \text{MUST BE} \leq 0.01$$

$$\left| \bar{C}_p\,(\text{SIDE A}) - \bar{C}_p\,(\text{SIDE B}) \right| \leftarrow \text{MUST BE} \leq 0.01$$

Figure 2-9. Pitot tube calibration data.

$$C_{p(s)} = C_{p(std)} \sqrt{\frac{\Delta p_{std}}{\Delta p_s}}$$ Equation 2-2

where:

$C_{p(s)}$ = Type S pitot tube coefficient

$C_{p(std)}$ = Standard pitot tube coefficient; use 0.99 if the coefficient is unknown and the tube is designed according to the criteria of Sections 2.7.1 to 2.7.5 of this method.

Δp_{std} = Velocity head measured by the standard pitot tube, cm H_2O (in. H_2O)

Δp_s = Velocity head measured by the Type S pitot tube, cm H_2O (in. H_2O)

4.1.4.2 Calculate $\overline{C}_p$ (side A), the mean A-side coefficient, and $\overline{C}_p$ (side B), the mean B-side coefficient; calculate the difference between these two average values.

4.1.4.3 Calculate the deviation of each of the three A-side values of $C_{p(s)}$ from $\overline{C}_p$ (side A), and the deviation of each B-side value of $C_{p(s)}$ from $\overline{C}_p$ (side B). Use the following equation:

$$\text{Deviation} = C_{p(s)} - \overline{C}_p \text{ (A or B)}$$ Equation 2-3

4.1.4.4 Calculate σ, the average deviation from the mean, for both the A and B sides of the pitot tube. Use the following equation:

$$\sigma \text{ (side A or B)} = \frac{\sum_{1}^{3} \left| C_{p(s)} - \overline{C}_p \text{ (A or B)} \right|}{3}$$ Equation 2-4

4.1.4.5 Use the Type S pitot tube only if the values of σ (side A) and σ (side B) are less than or equal to 0.01 and if the absolute value of the difference between $\overline{C}_p$ (A) and $\overline{C}_p$ (B) is 0.01 or less.

4.1.5 Special considerations.

4.1.5.1 Selection of calibration point.

4.1.5.1.1 When an isolated Type S pitot tube is calibrated, select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The Type S pitot coefficients so obtained, i.e., $\overline{C}_p$ (side A) and $\overline{C}_p$ (side B), will be valid, so long as either: (1) the isolated pitot tube is used; or (2) the pitot tube is used with other components (nozzle, thermocouple, sample probe) in an arrangement that is free from aerodynamic interference effects (see Figures 2-6 through 2-8).

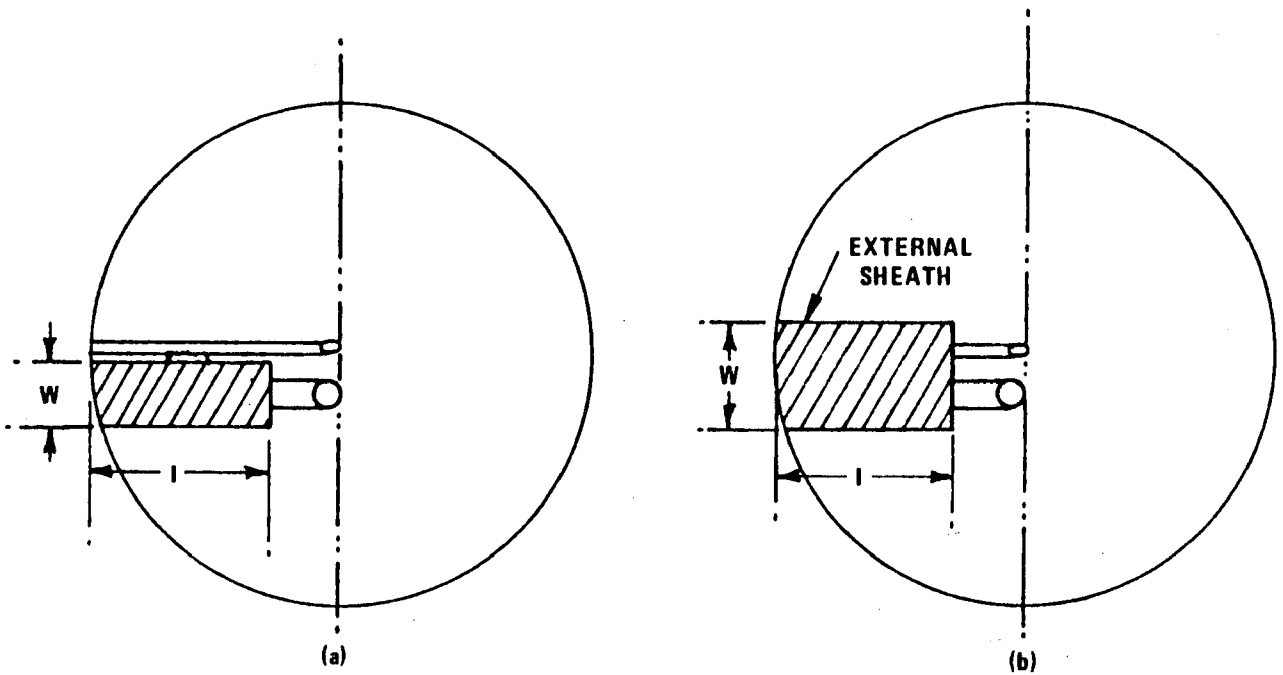
4.1.5.1.2 For Type S pitot tube-thermocouple combinations (without sample probe), select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The coefficients so obtained will be valid so long as the pitot tube-thermocouple combination is used by itself or with other components in an interference-free arrangement (Figures 2-6 and 2-8).

4.1.5.1.3 For assemblies with sample probes, the calibration point should be located at or near the center of the duct; however,

insertion of a probe sheath into a small duct may cause significant cross-sectional area blockage and yield incorrect coefficient values (Citation 9 in Section 6). Therefore, to minimize the blockage effect, the calibration point may be a few inches off-center if necessary. The actual blockage effect will be negligible when the theoretical blockage, as determined by a projected-area model of the probe sheath, is 2 percent or less of the duct cross-sectional area for assemblies without external sheaths (Figure 2-10a), and 3 percent or less for assemblies with external sheaths (Figure 2-10b).

4.1.5.2 For those probe assemblies in which pitot tube-nozzle interference is a factor (i.e., those in which the pitot-nozzle separation distance fails to meet the specification illustrated in Figure 2-6a), the value of $C_{p(s)}$ depends upon the amount of free-space between the tube and nozzle, and therefore is a function of nozzle size. In these instances, separate calibrations shall be performed with each of the commonly used nozzle sizes in place. Note that the single-velocity calibration technique is acceptable for this purpose, even though the larger nozzle sizes (>0.635 cm or 1/4 in.) are not ordinarily used for isokinetic sampling at velocities around 915 m/min (3000 ft/min), which is the calibration velocity; note also that it is not necessary to draw an isokinetic sample during calibration (see Citation 19 in Section 6).

4.1.5.3 For a probe assembly constructed such that its pitot tube is always used in the same orientation, only one side of the pitot tube need be calibrated (the side which will face the flow). The pitot tube must still meet the alignment specifications of Figure 2-2 or 2-3, however, and must have an average deviation (σ) value of 0.01 or less (see Section 4.1.4.4).



$$\text{ESTIMATED SHEATH BLOCKAGE (\%)} = \left[\frac{l \times W}{\text{DUCT AREA}}\right] \times 100$$

Figure 2-10. Projected-area models for typical pitot tube assemblies.

4.1.6 Field Use and Recalibration.

4.1.6.1 Field Use.

4.1.6.1.1 When a Type S pitot tube (isolated tube or assembly) is used in the field, the appropriate coefficient value (whether assigned or obtained by calibration) shall be used to perform velocity calculations. For calibrated Type S pitot tubes, the A side coefficient shall be used when the A side of the tube faces the flow, and the B side coefficient shall be used when the B side faces the flow; alternatively, the arithmetic average of the A and B side coefficient values may be used, irrespective of which side faces the flow.

4.1.6.1.2 When a probe assembly is used to sample a small duct (12 to 36 in. in diameter), the probe sheath sometimes blocks a significant part of the duct cross-section, causing a reduction in the effective value of $\overline{C}_{p(s)}$. Consult Citation 9 in Section 6 for details. Conventional pitot-sampling probe assemblies are not recommended for use in ducts having inside diameters smaller than 12 inches (Citation 16 in Section 6).

4.1.6.2 Recalibration.

4.1.6.2.1 Isolated Pitot Tubes. After each field use, the pitot tube shall be carefully reexamined in top, side, and end views. If the pitot face openings are still aligned within the specifications illustrated in Figure 2-2 or 2-3, it can be assumed that the baseline coefficient of the pitot tube has not changed. If, however, the tube has been damaged to the extent that it no longer meets the specifications of Figure 2-2 or 2-3, the damage shall either be repaired to restore proper alignment of the face openings or the tube shall be discarded.

4.1.6.2.2 Pitot Tube Assemblies. After each field use, check the face opening alignment of the pitot tube, as in Section 4.1.6.2.1; also,

remeasure the intercomponent spacings of the assembly. If the intercomponent spacings have not changed and the face opening alignment is acceptable, it can be assumed that the coefficient of the assembly has not changed. If the face opening alignment is no longer within the specifications of Figures 2-2 or 2-3, either repair the damage or replace the pitot tube (calibrating the new assembly, if necessary). If the intercomponent spacings have changed, restore the original spacings or recalibrate the assembly.

4.2 Standard pitot tube (if applicable). If a standard pitot tube is used for the velocity traverse, the tube shall be constructed according to the criteria of Section 2.7 and shall be assigned a baseline coefficient value of 0.99. If the standard pitot tube is used as part of an assembly, the tube shall be in an interference-free arrangement (subject to the approval of the Administrator).

4.3 Temperature Gauges. After each field use, calibrate dial thermometers, liquid-filled bulb thermometers, thermocouple-potentiometer systems, and other gauges at a temperature within 10 percent of the average absolute stack temperature. For temperatures up to 405°C (761°F), use an ASTM mercury-in-glass reference thermometer, or equivalent, as a reference; alternatively, either a reference thermocouple and potentiometer (calibrated by NBS) or thermometric fixed points, e.g., ice bath and boiling water (corrected for barometric pressure) may be used. For temperatures above 405°C (761°F), use an NBS-calibrated reference thermocouple-potentiometer system or an alternate reference, subject to the approval of the Administrator.

If, during calibration, the absolute temperatures measured with the gauge being calibrated and the reference gauge agree within 1.5 percent, the temperature data taken in the field shall be considered valid. Otherwise, the pollutant emission test shall either be

considered invalid or adjustments (if appropriate) of the test results shall be made, subject to the approval of the Administrator.

4.4 Barometer. Calibrate the barometer used against a mercury barometer.

5. Calculations

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

5.1 Nomenclature.

A = Cross-sectional area of stack, m^2 (ft^2).

B_{ws} = Water vapor in the gas stream (from Method 5 or Reference Method 4), proportion by volume.

C_p = Pitot tube coefficient, dimensionless.

K_p = Pitot tube constant,

$$34.97 \frac{m}{sec} \left[\frac{(g/g\text{-mole})(mm\ Hg)}{(°K)(mm\ H_2O)}\right]^{1/2}$$

for the metric system and

$$85.49 \frac{ft}{sec} \left[\frac{(lb/lb\text{-mole})(in.\ Hg)}{(°R)(in.\ H_2O)}\right]^{1/2}$$

for the English system.

M_d = Molecular weight of stack gas, dry basis (see Section 3.6) g/g-mole (lb/lb-mole).

M_s = Molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole).

$$= M_d(1 - B_{ws}) + 18.0\ B_{ws} \qquad \text{Equation 2-5}$$

P_{bar} = Barometric pressure at measurement site, mm Hg (in. Hg).

P_g = Stack static pressure, mm Hg (in. Hg).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

$= P_{bar} + P_g$ Equation 2-6

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

Q_{sd} = Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr).

t_s = Stack temperature, °C (°F).

T_s = Absolute stack temperature, °K (°R).

$= 273 + t_s$ for metric Equation 2-7

$= 460 + t_s$ for English Equation 2-8

T_{std} = Standard absolute temperature, 293 °K (528°R).

v_s = Average stack gas velocity, m/sec (ft/sec).

Δp = Velocity head of stack gas, mm H_2O (in. H_2O).

3600 = Conversion factor, sec/hr.

18.0 = Molecular weight of water, g/g-mole (lb/lb-mole).

5.2 Average stack gas velocity.

$$v_s = K_p C_p \left(\sqrt{\Delta p}\right)_{avg} \sqrt{\frac{T_{s(avg)}}{P_s M_s}} \qquad \text{Equation 2-9}$$

5.3 Average stack gas dry volumetric flow rate.

$$Q_{sd} = 3600\,(1-B_{ws})\,v_s A \left(\frac{T_{std}}{T_{s(avg)}}\right)\left(\frac{P_s}{P_{std}}\right) \qquad \text{Equation 2-10}$$

6. Bibliography

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12. Vollaro, R. F. Establishment of a Baseline Coefficient Value for Properly Constructed Type S Pitot Tubes. U. S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N. C. November 1976.

13. Vollaro, R. F. An Evaluation of Single-Velocity Calibration Technique as a Means of Determining Type S Pitot Tube Coefficients. U. S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N. C. August 1975.

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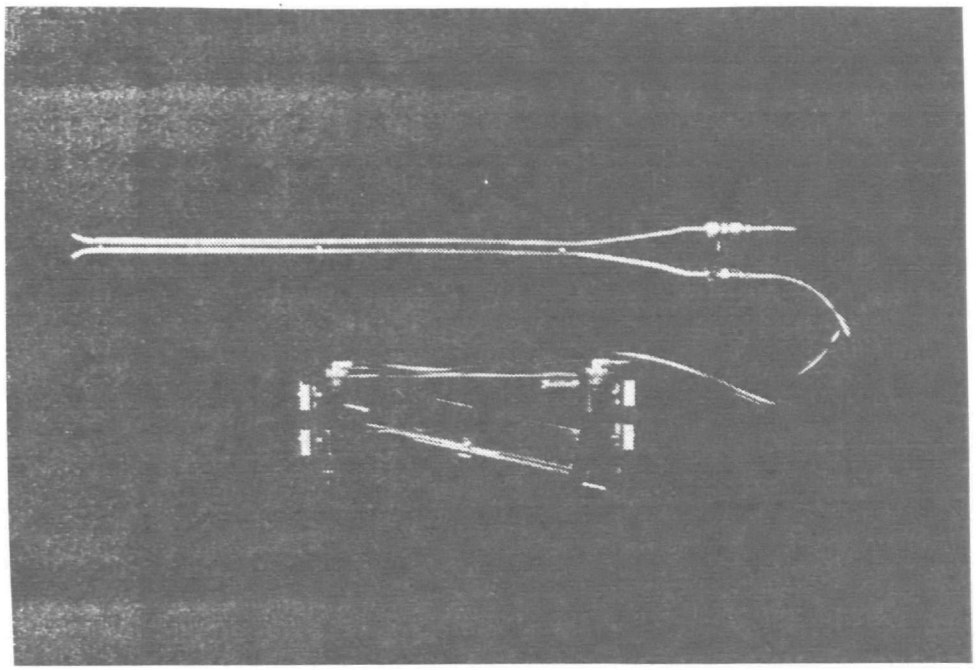
SLIDE 103-0

NOTES

METHOD — 2

Determination of Stack Gas Velocity
and Volumetric Flow Rate

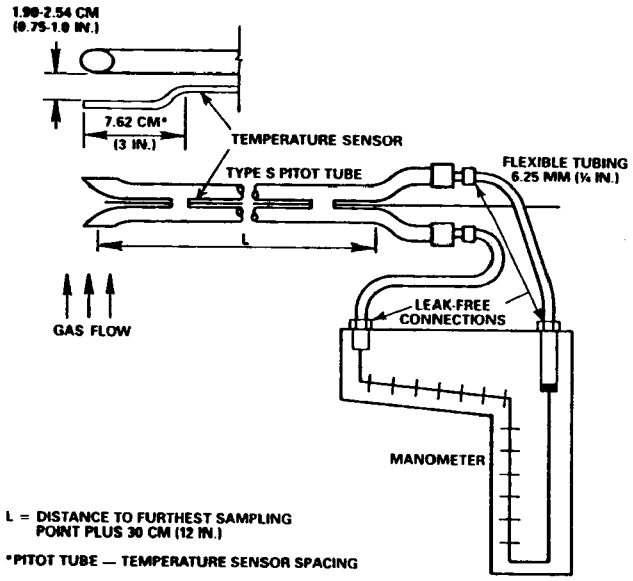
SLIDE 103-1



SLIDE 103-2

NOTES

TYPE S PITOT TUBE MANOMETER ASSEMBLY



SLIDE 103-3

APPLICABILITY OF METHOD 2

This method is only applicable at sites which:

1. Meet the criteria of method 1
2. Do not contain cyclonic or non parallel flow

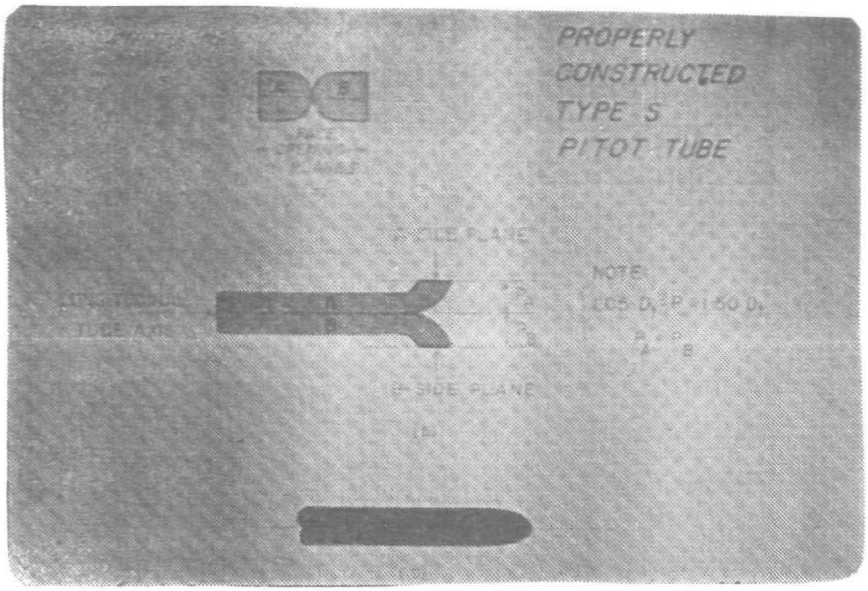
ALTERNATIVES WHEN UNACCEPTABLE CONDITIONS EXIST

(Subject to Approval of the Administrator)

1. Install straightening vanes.
2. Calculate total volumetric flow rate stoichiometrically.
3. Move to a measurement site at which flow is acceptable.
4. Use procedures as described in the cyclonic flow special problems session.

SLIDE 103-4

NOTES



SLIDE 103-5

DIFFERENTIAL PRESSURE GAUGE

- An inclined manometer or equivalent device is required to measure ΔP.
- A differential pressure gauge of greater sensitivity shall be used if any of the following conditions exist:
  1. arithmetic average of all ΔP readings is < 0.05 in. H_2O
  2. traverses of ≥ 12 pts., and 10% of ΔP readings are below 0.05 in. H_2O
  3. traverses of < 12 pts., and more than 1 ΔP reading is below 0.05 in. H_2O

Note: Standard manometer used in source testing is a 10 in. inclined vertical manometer (0.01 in. divisions on a 0 to 1 in. scale, and 0.1 in. divisions on a 1 to 10 in. scale).

NOTES

(cont.)

- Alternative approach to determine if a more sensitive differential pressure guage is required:

$$T = \frac{\sum_{i=1}^{n} \sqrt{\Delta P_i + K}}{\sum_{i=1}^{n} \sqrt{\Delta P_i}}$$

- If T is $>$ 1.05, a more sensitive differential pressure guage must be used.

Where:

ΔP_i = individual velocity head reading at a traverse point

n = total number of points

K = 0.13 mm H_2O for metric units and 0.005 in. H_2O for English units

TEMPERATURE SENSOR

The temperature sensor must be accurate to within 1.5% of Minimum Absolute Stack Temperature.

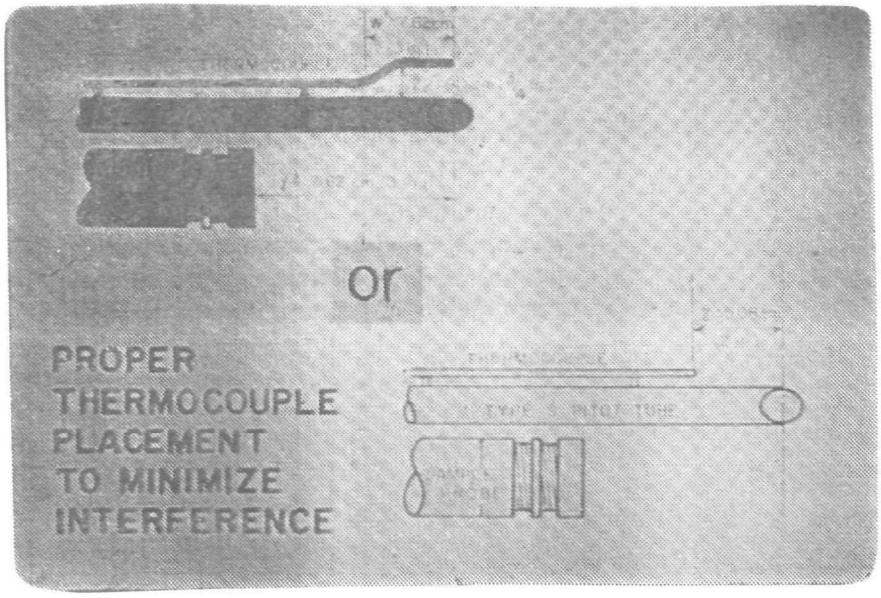
Temperature sensors typically used in testing include:

- Thermocouple
- Bimetallic Thermometer
- Mercury-in-Glass Thermometer
- Liquid-Filled Bulb Thermometer

Note: When the stack temperature measurement is used to calculate moisture, the sensitivity is ± 2°F.

SLIDE 103-8

NOTES



SLIDE 103-9

PRESSURE PROBE AND GUAGE

Static pressure measurement must be accurate to within 0.1 in. Hg (1.36 in. H:O).

Pressure sensors typically used to measure static pressure during testing include:

- a piezometer tube and mercury or water-filled U-tube manometer
- the static tap of a standard pitot tube
- one leg of the type S pitot tube

SLIDE 103-10

NOTES

BAROMETRIC PRESSURE

Barometric pressure measurement must be accurate to within 0.1 in. Hg.

Barometric pressure during testing is obtained by:

INSTRUMENT

Mercury, aneroid or other barometer (with required sensitivity)

OTHER

Obtain barometric pressure from nearby National Weather Service station (station pressure) and adjust for elevation differences between sampling site and weather station.

Note: The station pressure must be used not the pressure corrected to sea level.

SLIDE 103-11

PITOT TUBE CALIBRATION

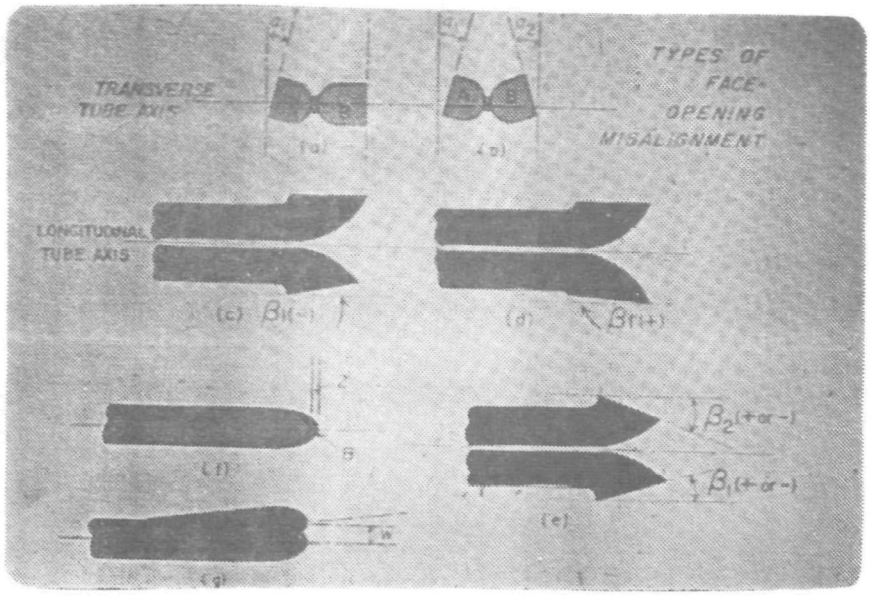
Perform Dimensional Specification Test

and/or

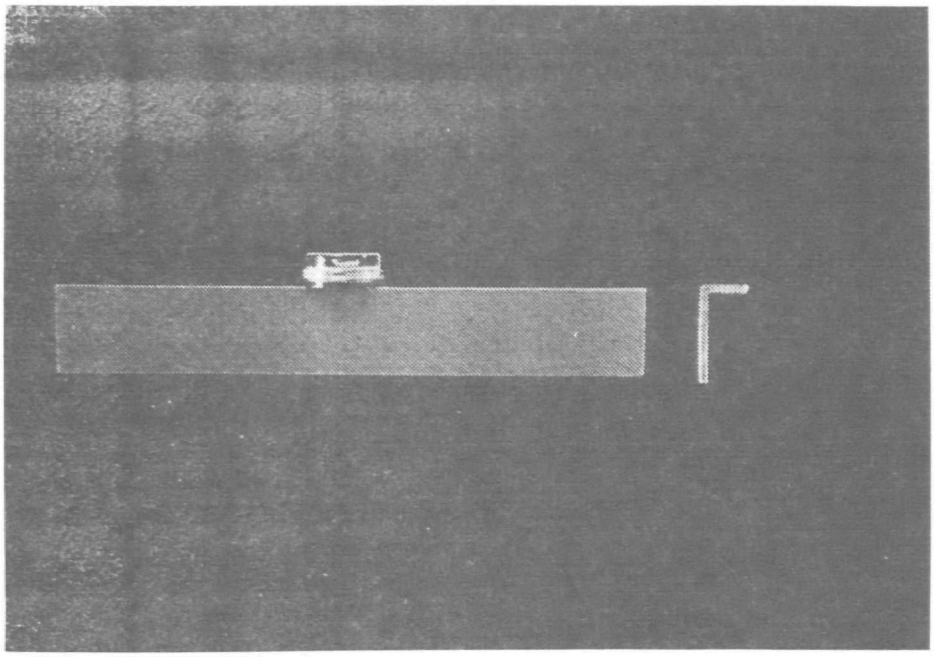
Calibrate in wind tunnel against standard pitot tube (preferably with NBS traceable coefficient)

SLIDE 103-12

NOTES



SLIDE 103-13



SLIDE 103-14

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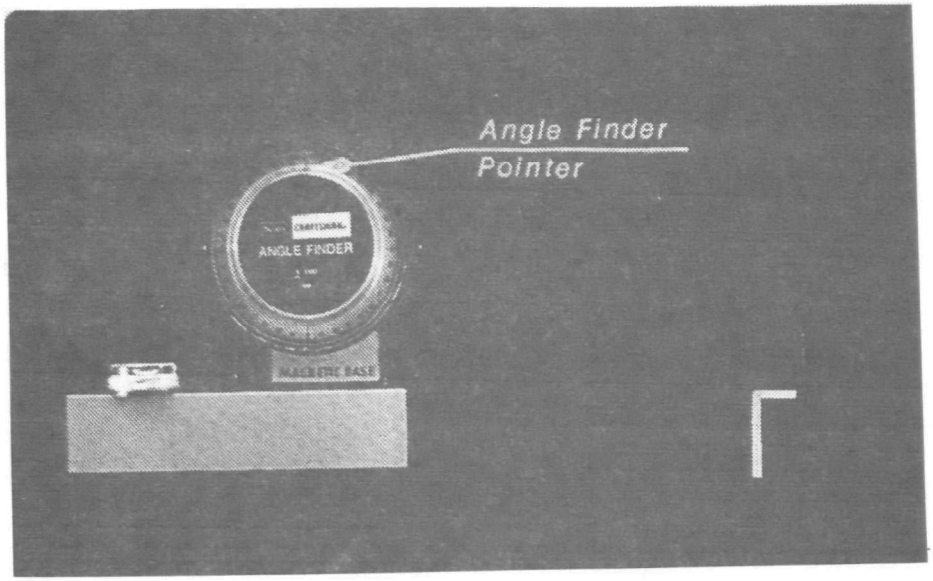
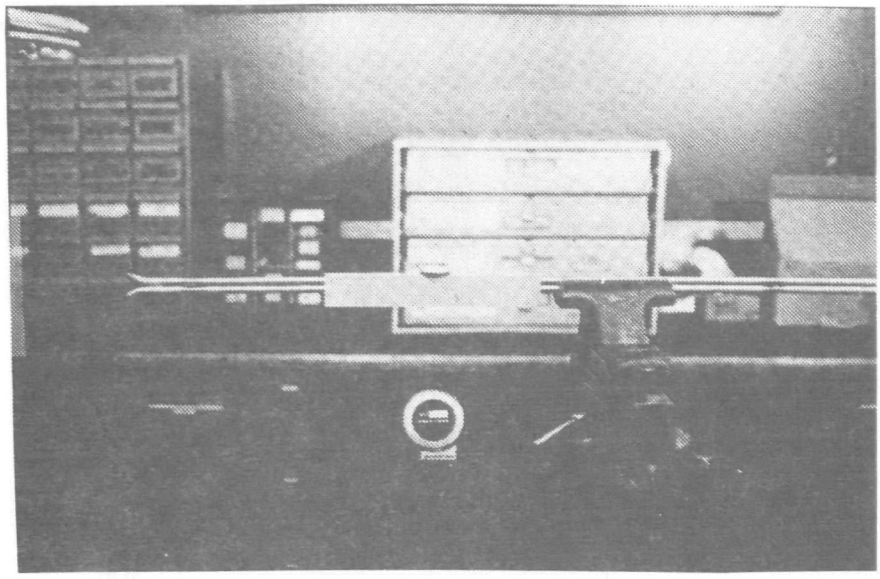


SLIDE 103-15



SLIDE 103-16

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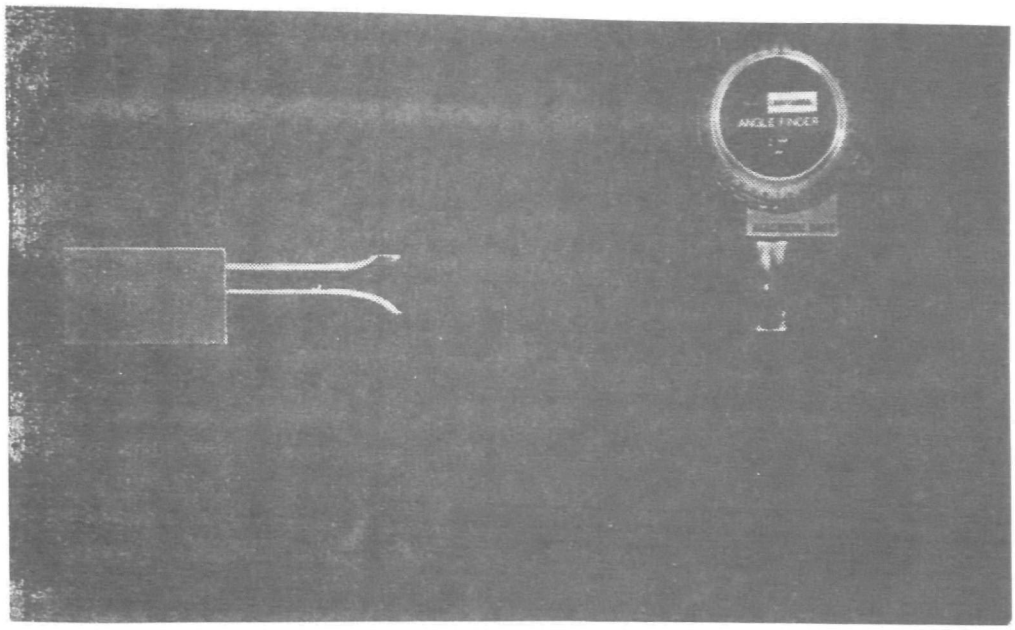
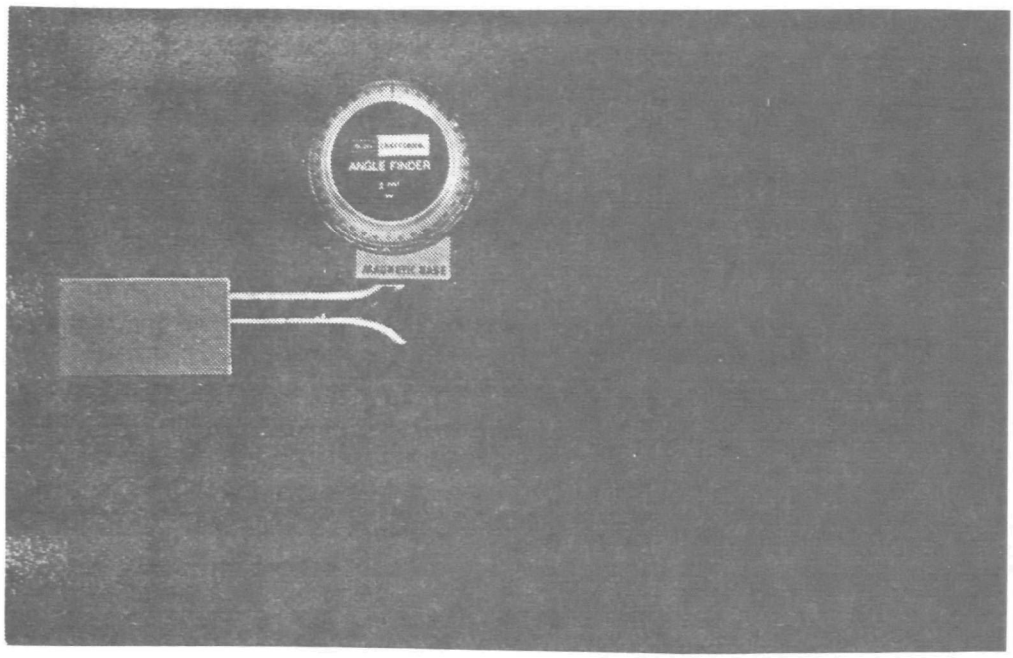


SLIDE 103-17



SLIDE 103-18

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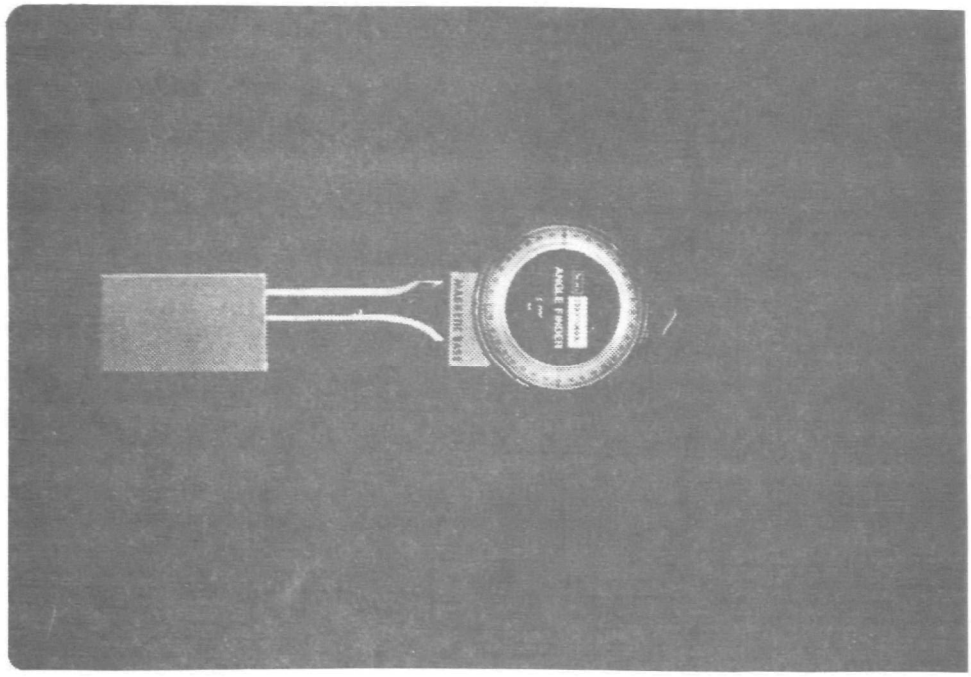
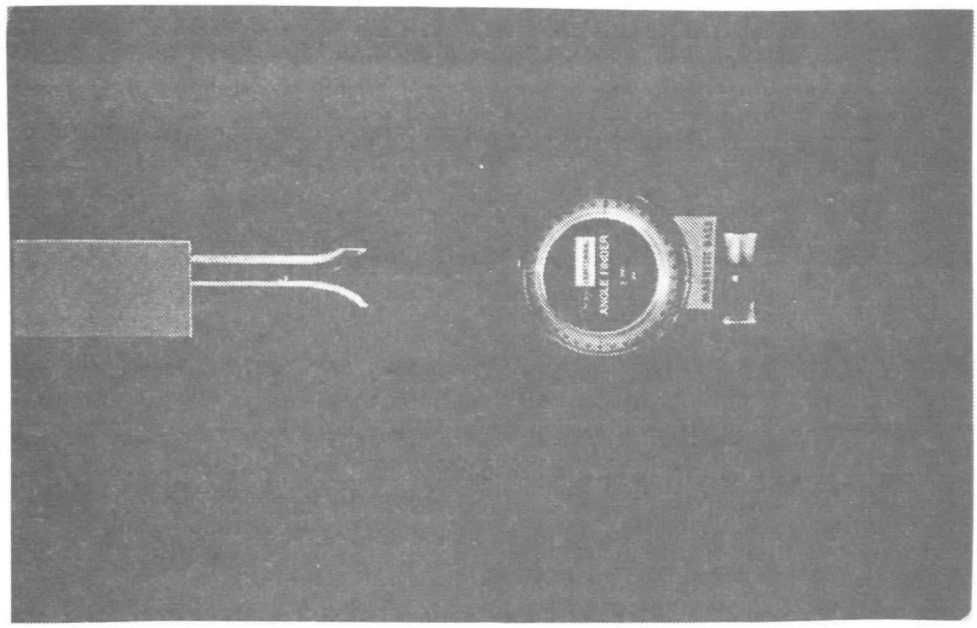


SLIDE 103-19



SLIDE 103-20

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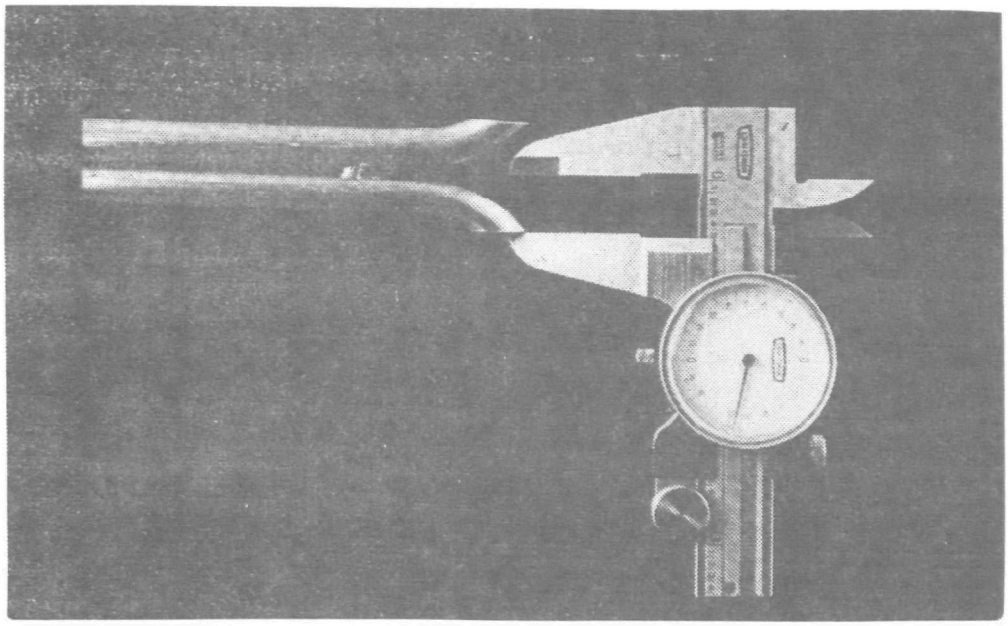
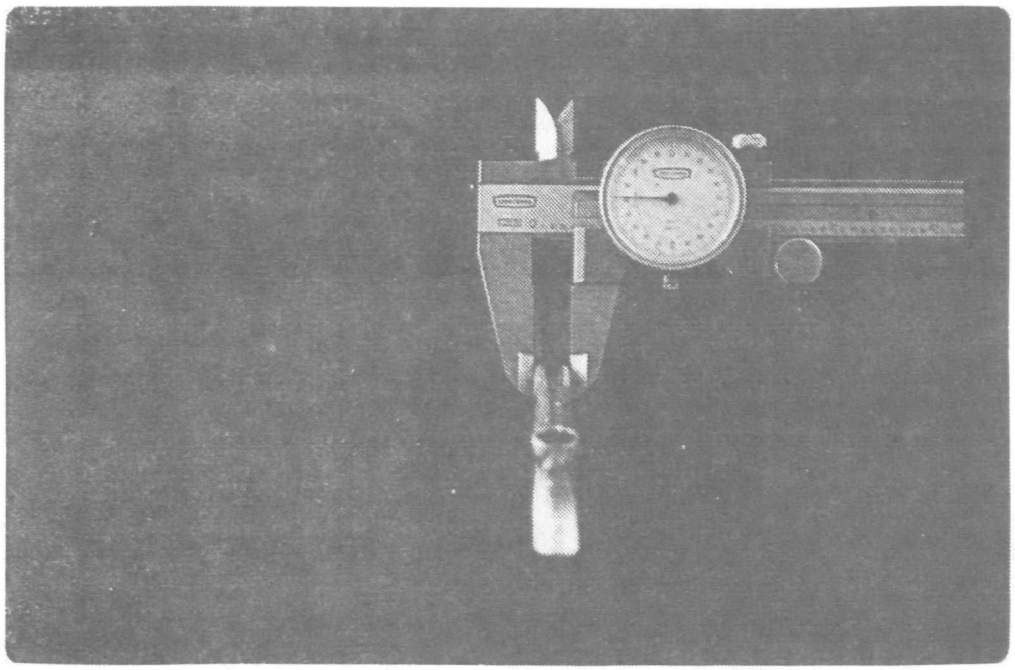


SLIDE 103-21



SLIDE 103-22

NOTES

TYPE-S PITOT TUBE INSPECTION DATA SHEET

PITOT TUBE ASSEMBLY LEVEL? _____ YES _____ NO

PITOT TUBE OPENINGS DAMAGED? _____ YES (EXPLAIN BELOW) _____ NO

α_1 = ____° (≤10°), α_2 = ____° (≤10°), β_1 = ____° (≤5°), β_2 = ____° (≤5°)

γ = ____°, θ = ____°, A = ____ CM (IN.)

z = A sin γ = ____ CM (IN.); ≤0.32 CM (≤⅛ IN.),

w = A sin θ = ____CM (IN.); ≤0.08 CM (≤1/32 IN.)

P_A _____ CM (IN.) P_b _____ CM (IN.)

D_t = _____ CM (IN.) $\frac{P}{D_t}$ = _____ (1.05 ≤ and ≤ 1.50)

$P_a = P_b = P$

COMMENTS: _____

_____

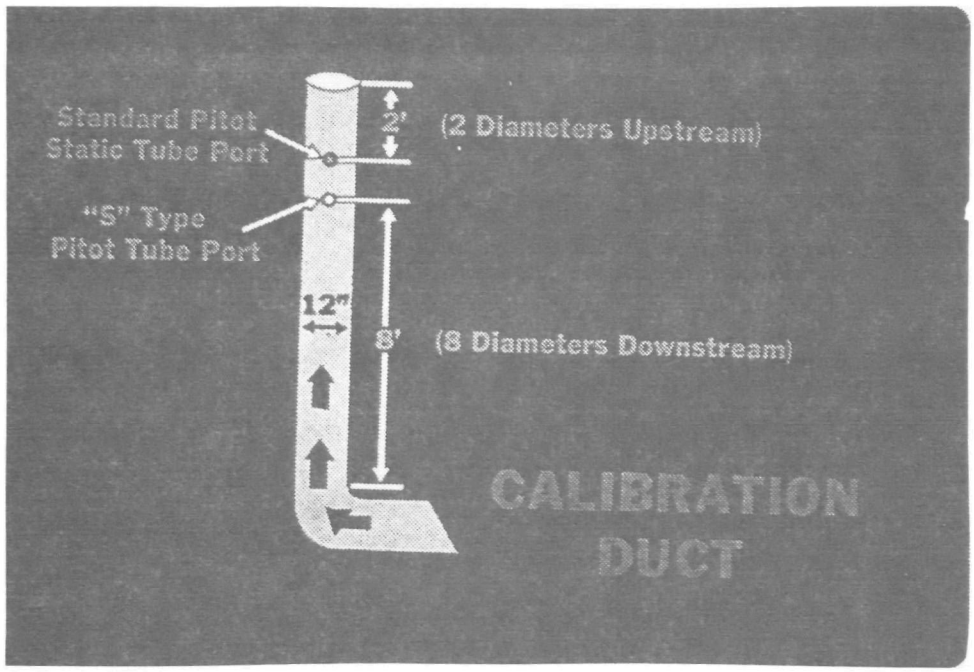
_____

CALIBRATION REQUIRED? _____

_____

DATE: _____ CONDUCTED BY: _____

SLIDE 103-23



SLIDE 103-24

NOTES

PITOT TUBE CALIBRATION DATA SHEET

CALIBRATION PITOT TUBE: TYPE STD. SIZE (OD) ¼" ID NUMBER 2
TYPE S PITOT TUBE ID NUMBER 8S $C_{p(std)}$ = 1.0
CALIBRATION: DATE 3/10/76 PERFORMED BY F.M.

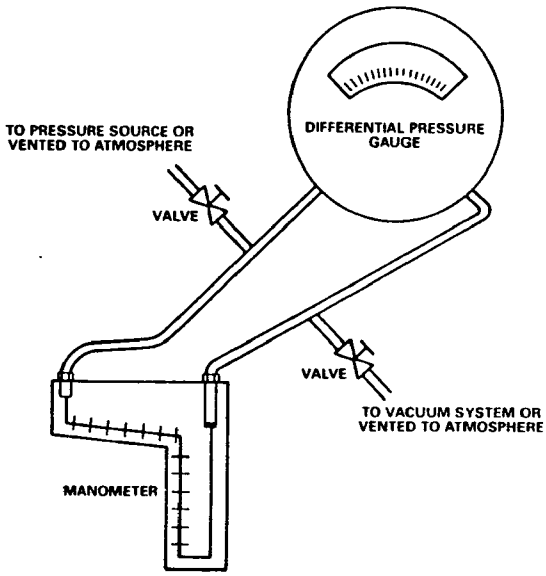
"A" SIDE CALIBRATION			
Δp_{std} cm H_2O (IN. H_2O)	Δp_s cm H_2O (IN. H_2O)	[a] $C_{p(S)}$	[b] DEV.
0.20	0.28	0.845	−0.005
0.30	0.41	0.856	0.006
0.50	0.69	0.851	−0.001
1.00	1.38	0.850	0
	AVERAGE	0.850	

a $C_{p(S)} = C_{p(std)} \sqrt{\frac{\Delta p_{std}}{\Delta p_S}} = 1.0 \sqrt{\frac{0.20}{0.28}} = 1.0 \sqrt{0.714} = 0.845$

b DEV = $C_{p(S)} = \bar{C}_{p'}$ (MUST BE ≤0.01).
$\bar{C}_p(A) = \bar{C}_p(B) =$ 0.005 (MUST BE ≤0.01).

SLIDE 103-25

DIFFERENTIAL PRESSURE GAUGE CALIBRATION



SLIDE 103-26

NOTES

TEMPERATURE SENSOR CALIBRATION

Calibrate initially and after each field use.

Post test calibration must be performed at a temperature within 10% of the average absolute stack temperature.

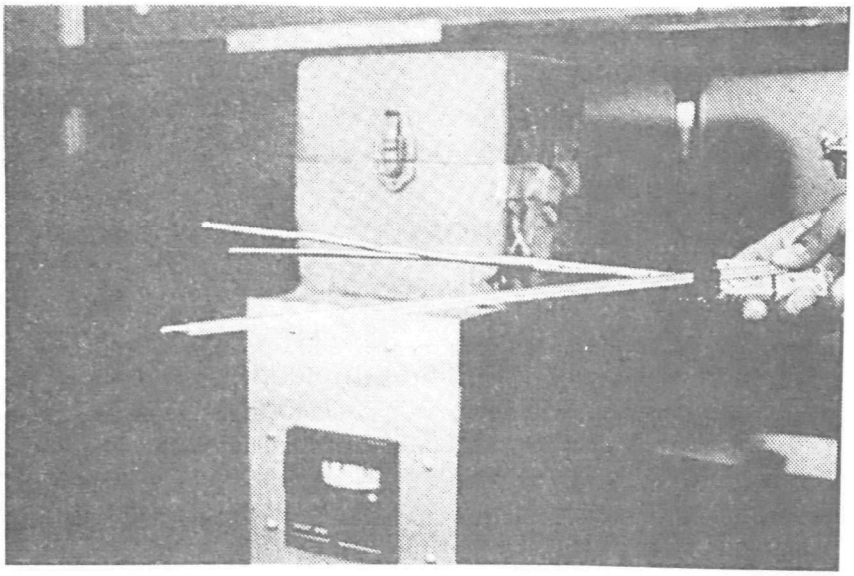
CALIBRATION REFERENCES

UP TO 761°F	ABOVE 761°F
ASTM Mercury-in-glass reference thermometer	NBS-Calibrated reference thermocouple

ALTERNATIVE CALIBRATION REFERENCES

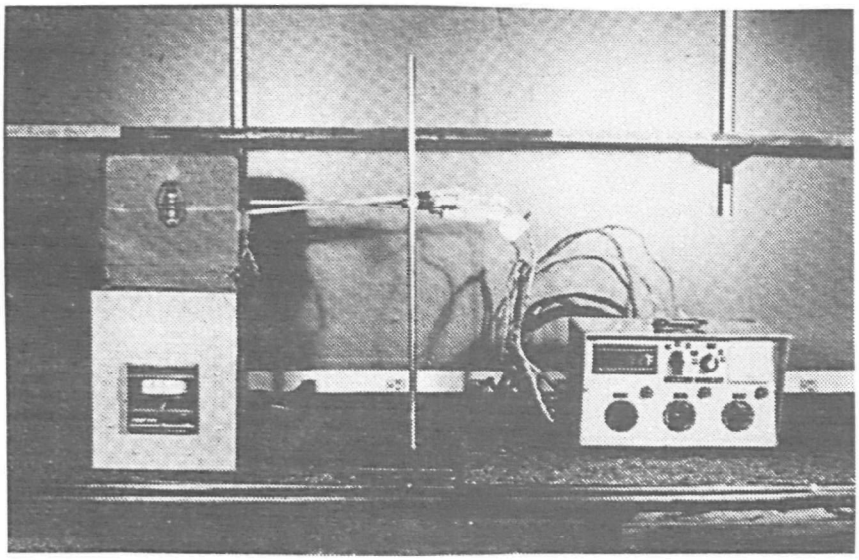
Thermometric fixed points (ice bath and boiling water)

SLIDE 103-27



SLIDE 103-28

NOTES



SLIDE 103-29

TEMPERATURE SENSOR CALIBRATION EVALUATION

THERMOCOUPLE

Absolute temperature values must agree within ± 1.5% at each point.

- Plot data on linear graph paper, draw best fit line.
- Data may be extrapolated above and below calibration points.

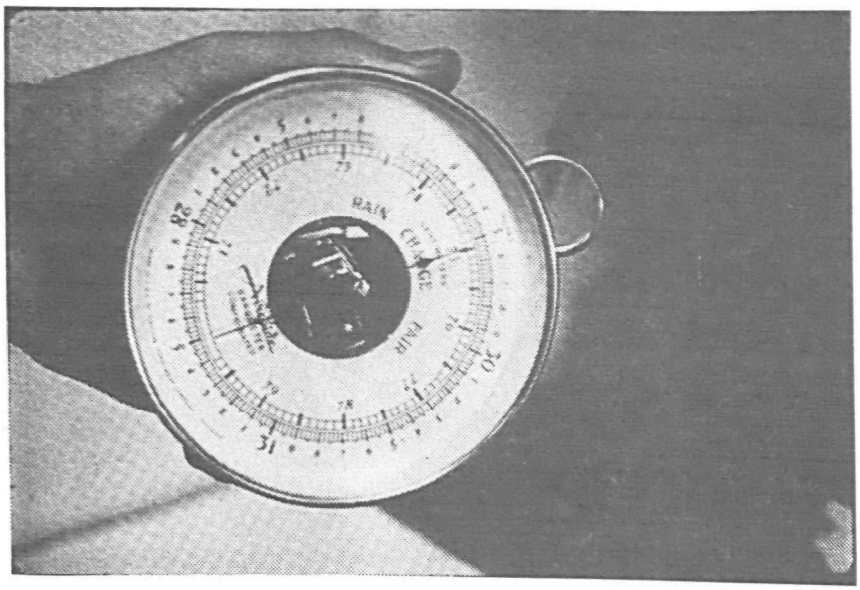
THERMOMETER

Absolute temperature values must agree within ± 1.5% at each point.

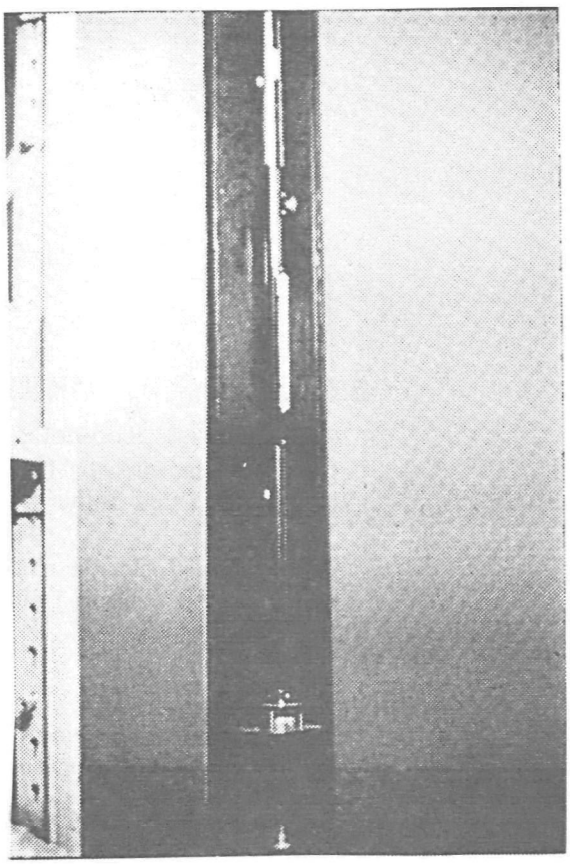
- Thermometer may be used over the range of calibration points.
- If correction factor is needed it must be affixed to thermometer.

SLIDE 103-30

NOTES

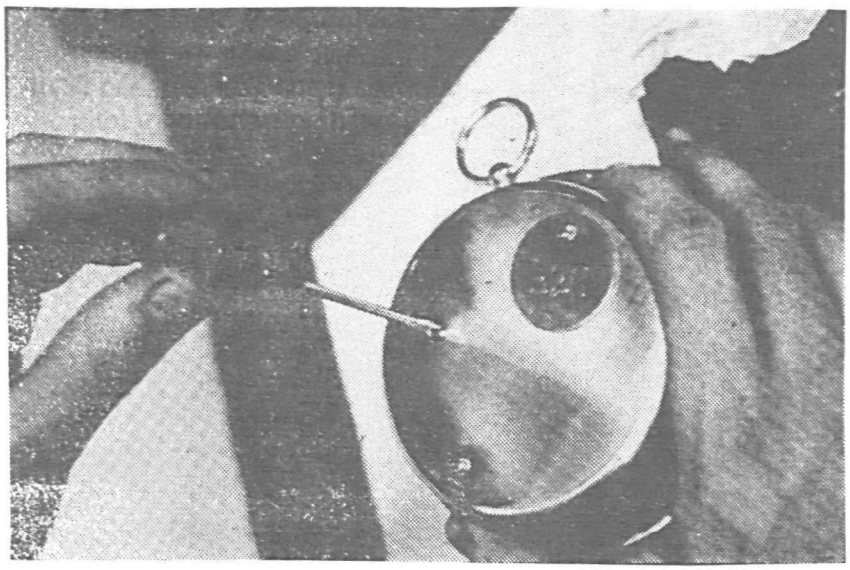


SLIDE 103-31



SLIDE 103-32

NOTES



SLIDE 103-33

VELOCITY MEASUREMENT PROCEDURES

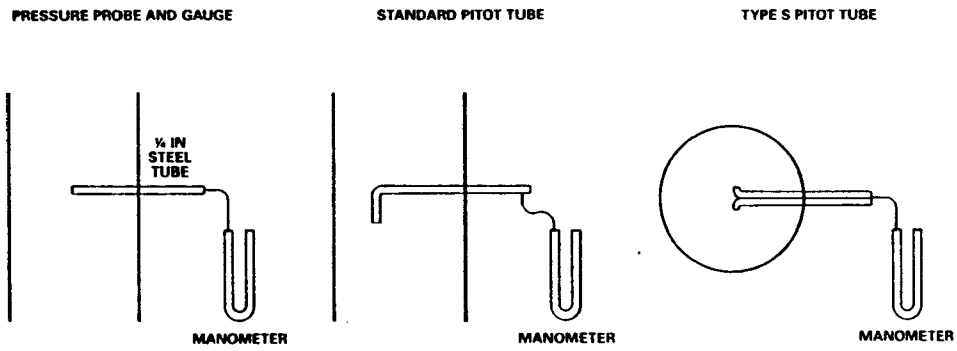
1. Leak-check pitot tube and differential pressure gauge.
2. For circular stacks less than 10 ft. in diameter, two ports are sufficient. Use four ports when stack diameter is greater than 10 ft.
3. Pitot tubes longer than 10 ft. should be structurally reinforced to prevent bending of tube and misalignment errors.
4. Identify each sample port and traverse point with a letter or number.
5. Read velocity head and temperature at least twice at each point and record the average.

(cont.)

6. Care should be taken to prevent touching the pitot tube tip to the side of the stack.
7. Plug unused sampling ports and seal port being used as tightly as possible.
8. After traverse, check differential pressure gauge zero; repeat traverse if zero has shifted.
9. If liquid droplets are present, use a liquid trap in positive pressure leg of pitot tube.
10. A post-test leak check is required after each run of the pitot tube and velocity pressure system.

SLIDE 103-35

STATIC PRESSURE MEASUREMENT



SECTION D. METHOD 3

METHOD 3--GAS ANALYSIS FOR CARBON DIOXIDE, OXYGEN, EXCESS AIR, AND DRY MOLECULAR WEIGHT

1. Principle and Applicability

1.1 Principle. A gas sample is extracted from a stack, by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent carbon dioxide (CO_2), percent oxygen (O_2), and, if necessary, percent carbon monoxide (CO). If a dry molecular weight determination is to be made, either an Orsat or a Fyrite[1] analyzer may be used for the analysis; for excess air or emission rate correction factor determination, an Orsat analyzer must be used.

1.2 Applicability. This method is applicable for determining CO_2 and O_2 concentrations, excess air, and dry molecular weight of a sample from a gas stream of a fossil-fuel combustion process. The method may also be applicable to other processes where it has been determined that compounds other than CO_2, O_2, CO, and nitrogen (N_2) are not present in concentrations sufficient to affect the results.

Other methods, as well as modifications to the procedure described herein, are also applicable for some or all of the above determinations. Examples of specific methods and modifications include: (1) a multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point; (2) a method using CO_2 or O_2 and stoichiometric calculations to determine dry molecular weight and excess air; (3) assigning a value of 30.0

[1]Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator, U.S. Environmental Protection Agency.

2. Apparatus

As an alternative to the sampling apparatus and systems described herein, other sampling systems (e.g., liquid displacement) may be used provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are otherwise capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

2.1 Grab Sampling (Figure 3-1).

2.1.1 Probe. The probe should be made of stainless steel or borosilicate glass tubing and should be equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other material inert to O_2, CO_2, CO, and N_2 and resistant to temperature at sampling conditions may be used for the probe; examples of such material are aluminum, copper, quartz glass and Teflon.

2.1.2 Pump. A one-way squeeze bulb, or equivalent, is used to transport the gas sample to the analyzer.

2.2 Integrated Sampling (Figure 3-2).

2.2.1 Probe. A probe such as that described in Section 2.1.1 is suitable.

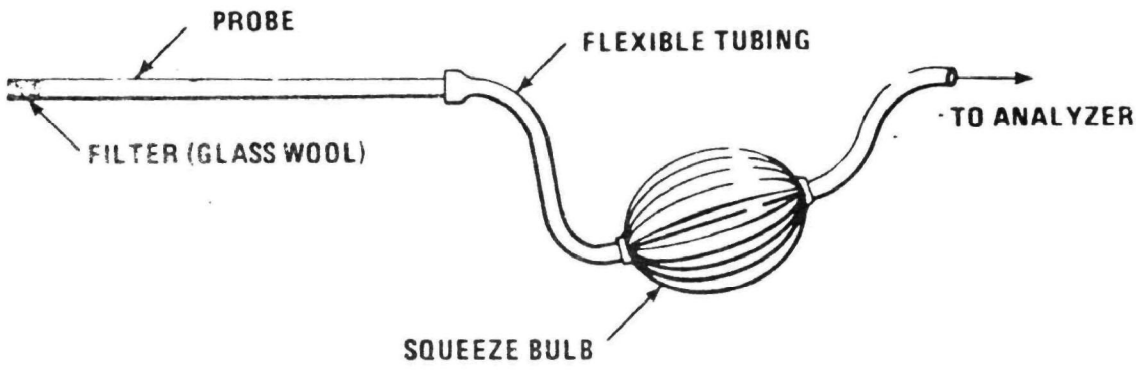


Figure 3-1. Grab-sampling train.

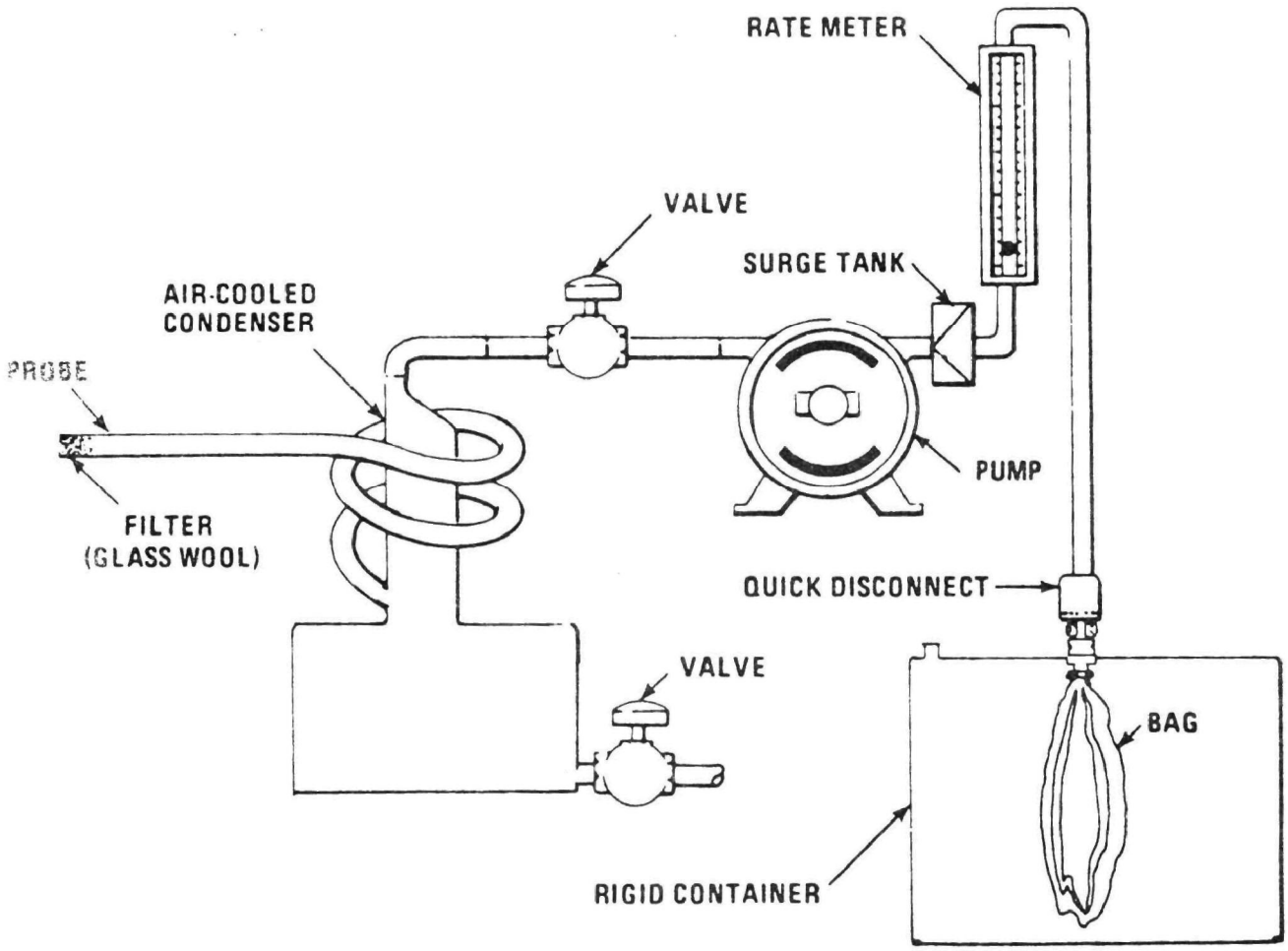


Figure 3-2. Integrated gas-sampling train.

2.2.2 Condenser. An air-cooled or water-cooled condenser, or other condenser that will not remove O_2, CO_2, CO, and N_2, may be used to remove excess moisture which would interfere with the operation of the pump and flow meter.

2.2.3 Valve. A needle valve is used to adjust sample gas flow rate.

2.2.4 Pump. A leak-free, diaphragm-type pump, or equivalent, is used to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.2.5 Rate Meter. The rotameter, or equivalent rate meter, used should be capable of measuring flow rate to within ± 2 percent of the selected flow rate. A flow rate range of 500 to 1000 cm^3/min is suggested.

2.2.6 Flexible Bag. Any leak-free plastic (e.g., Tedlar, Mylar, Teflon) or plastic-coated aluminum (e.g., aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and time length of the test run, may be used. A capacity in the range of 55 to 90 liters is suggested.

To leak-check the bag, connect it to a water manometer and pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. An alternative leak-check method is to pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O) and allow to stand overnight. A deflated bag indicates a leak.

2.2.7 Pressure Gauge. A water-filled U-tube manometer, or equivalent, of about 28 cm (12 in.) is used for the flexible bag leak-check.

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

2.3 Analysis. For Orsat and Fyrite analyzer maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

2.3.1 Dry Molecular Weight Determination. An Orsat analyzer or Fyrite type combustion gas analyzer may be used.

2.3.2 Emission Rate Correction Factor or Excess Air Determination. An Orsat analyzer must be used. For low CO_2 (less than 4.0 percent) or high O_2 (greater than 15.0 percent) concentrations, the measuring burette of the Orsat must have at least 0.1 percent subdivisions.

3. Dry Molecular Weight Determination

Any of the three sampling and analytical procedures described below may be used for determining the dry molecular weight.

3.1 Single-Point, Grab Sampling and Analytical Procedure.

3.1.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

3.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. If an Orsat analyzer is used, it is recommended that the analyzer be leak-checked by following the procedure in Section 5; however, the leak-check is optional.

3.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer and immediately analyze it for percent CO_2 and percent O_2. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.1.4 Repeat the sampling, analysis, and calculation procedures, until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (lb/lb-mole).

3.2 Single-Point, Integrated Sampling and Analytical Procedure.

3.2.1 The sampling point in the duct shall be located as specified in Section 3.1.1.

3.2.2 Leak-check (optional) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (optional) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

3.2.3 Sample at a constant rate. The sampling run should be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collection of at least 30 liters (1.00 ft^3) of sample gas is recommended; however, smaller volumes may be collected, if desired.

3.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent CO_2 and percent O_2 using either an Orsat analyzer or a Fyrite type combustion gas analyzer. If an Orsat analyzer is used, it is recommended that the Orsat leak-check described in Section 5 be performed before this determination; however, the check is optional. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.2.5 Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

3.3. Multi-Point, Integrated Sampling and Analytical Procedure.

3.3.1 Unless otherwise specified by the Administrator, EPA, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used for all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to the approval of the Administrator.

3.3.2 Follow the procedures outlined in Sections 3.2.2 through 3.2.5, except for the following: traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

4. Emission Rate Correction Factor or Excess Air Determination

Note: A Fyrite type combustion gas analyzer is not acceptable for excess air or emission rate correction factor determination, unless approved by the Administrator. If both percent CO_2 and percent O_2 are measured, the analytical results of any of the three procedures given below may also be used for calculating the dry molecular weight.

Each of the three procedures below shall be used only when specified in an applicable subpart of the standards. The use of these procedures for other purposes must have specific prior approval of the Administrator.

4.1 Single-Point, Grab Sampling and Analytical Procedure.

4.1.1 The sampling point in the duct shall either be at the centroid of the cross-section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

4.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. Leak-check the Orsat analyzer according to the procedure described in Section 5. This leak-check is mandatory.

4.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw

TIME	TRAVERSE PT.	Q 1pm	% DEV.[a]
	AVERAGE		

[a] % DEV = $\left(\frac{Q - Q_{avg}}{Q_{avg}}\right) 100$ (MUST BE $\leq 10\%$)

Figure 3-3. Sampling rate data.

a sample into the analyzer. For emission rate correction factor determination, immediately analyze the sample, as outlined in Sections 4.1.4 and 4.1.5, for percent CO_2 or percent O_2. If excess air is desired, proceed as follows: (1) immediately analyze the sample, as in Sections 4.1.4 and 4.1.5, for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; and (3) calculate percent excess air as outlined in Section 6.2.

4.1.4 To ensure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.1.5 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis. Note: Since this single-point, grab sampling and analytical procedure is normally conducted in conjunction with a single-point, grab sampling and analytical procedure for a pollutant, only one analysis is ordinarily conducted. Therefore, great care must be taken to obtain a valid sample and analysis. Although in most cases only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Citation 5 in the Bibliography be used to validate the analytical data.

4.2 Single-Point, Integrated Sampling and Analytical Procedure.

4.2.1 The sampling point in the duct shall be located as specified in Section 4.1.1.

4.2.2 Leak-check (mandatory) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (mandatory) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum shall remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

4.2.3 Sample at a constant rate, or as specified by the Administrator. The sampling run must be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collect at least 30 liters (1.00 ft^3) of sample gas. Smaller volumes may be collected, subject to approval of the Administrator.

4.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. For emission rate correction factor determination, analyze the sample within 4 hours after it is taken for percent CO_2 or percent O_2 (as outlined in Sections 4.2.5 through 4.2.7). The Orsat analyzer must be leak-checked (see Section 5) before the analysis. If excess air is desired, proceed as follows: (1) within 4 hours after the sample is taken, analyze it (as in Sections 4.2.5

through 4.2.7) for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; (3) calculate percent excess air, as outlined in Section 6.2.

4.2.5 To ensure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.2.6 Repeat the analysis until the following criteria are met:

4.2.6.1 For percent CO_2, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when CO_2 is greater than 4.0 percent or (b) 0.2 percent by volume when CO_2 is less than or equal to 4.0 percent. Average the three acceptable values of percent CO_2 and report the results to the nearest 0.1 percent.

4.2.6.2 For percent O_2, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when O_2 is less than 15.0 percent or (b) 0.2 percent by volume when O_2 is greater than or equal to 15.0 percent. Average the three acceptable values of percent O_2 and report the results to the nearest 0.1 percent.

4.2.6.3 For percent CO, repeat the analytical procedure until the results of any three analyses differ by no more than 0.3 percent. Average the three acceptable values of percent CO and report the results to the nearest 0.1 percent.

4.2.7 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis. Note: Although in most instances only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Citation 5 in the Bibliography be used to validate the analytical data.

4.3 Multi-Point, Integrated Sampling and Analytical Procedure.

4.3.1 Both the minimum number of sampling points and the sampling point location shall be as specified in Section 3.3.1 of this method. The use of fewer points than specified is subject to the approval of the Administrator.

4.3.2 Follow the procedures outlined in Sections 4.2.2 through 4.2.7, except for the following: Traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

5. Leak-Check Procedure for Orsat Analyzers

Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak-checked on site before the flue gas sample is introduced into it. The procedure for leak-checking an Orsat analyzer is:

5.1.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing and then close the pipette stopcock.

5.1.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette and then close the manifold stopcock.

5.1.3 Record the meniscus position.

5.1.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

5.1.5 For the Orsat analyzer to pass the leak-check, two conditions must be met:

5.1.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

5.1.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

5.1.6 If the analyzer fails the leak-check procedure, all rubber connections and stopcocks should be checked until the cause of the leak is identified. Leaking stopcocks must be disassembled, cleaned, and regreased. Leaking rubber connections must be replaced. After the analyzer is reassembled, the leak-check procedure must be repeated.

6. Calculations

6.1 Nomenclature.

M_d = Dry molecular weight, g/g-mole (lb/lb-mole).

%EA = Percent excess air.

$\%CO_2$ = Percent CO_2 by volume (dry basis).

$\%O_2$ = Percent O_2 by volume (dry basis).

%CO = Percent CO by volume (dry basis).

$\%N_2$ = Percent N_2 by volume (dry basis).

0.264 = Ratio of O_2 to N_2 in air, v/v.

0.280 = Molecular weight of N_2 or CO, divided by 100.

0.320 = Molecular weight of O_2 divided by 100.

0.440 = Molecular weight of CO_2 divided by 100.

6.2 Percent Excess Air. Calculate the percent excess air (if applicable), by substituting the appropriate values of percent O_2, CO, and N_2 (obtained from Section 4.1.3 or 4.2.4) into Equation 3-1.

$$\%EA = \left[\frac{\%O_2 - 0.5\%\ CO}{0.264\ \%N_2 - (\%O_2 - 0.5\%\ CO)} \right] 100 \qquad \text{Equation 3-1}$$

Note: The equation above assumes that ambient air is used as the source of O_2 and that the fuel does not contain appreciable amounts of N_2 (as do coke oven or blast furnace gases). For those cases when appreciable amounts of N_2 are present (coal, oil, and natural gas do not contain appreciable amounts of N_2) or when oxygen enrichment is used, alternate methods, subject to approval of the Administrator, are required.

6.3 Dry Molecular Weight. Use Equation 3-2 to calculate the dry molecular weight of the stack gas.

$$M_d = 0.440(\%CO_2) + 0.320(\%O_2) + 0.280(\%N_2 + \%CO) \qquad \text{Equation 3-2}$$

Note: The above equation does not consider argon in air (about 0.9 percent, molecular weight of 37.7). A negative error of about 0.4 percent is introduced. The tester may opt to include argon in the analysis using procedures subject to approval of the Administrator.

7. Bibliography

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2. Conner, William D. and J. S. Nader. Air Sampling with Plastic Bags. Journal of the American Industrial Hygiene Association. 25:291-297. 1964.

3. Burrell Manual for Gas Analysts, Seventh edition. Burrell Corporation, 2223 Fifth Avenue, Pittsburgh, Pa. 15219. 1951.

4. Mitchell, W.J. and M.R. Midgett. Field Reliability of the Orsat Analyzer. Journal of Air Pollution Control Association. 26:491-495. May 1976.

5. Shigehara, R.T., R.M. Neulicht, and W.S. Smith. Validating Orsat Analysis Data from Fossil Fuel-Fired Units. Stack Sampling News. 4(2):21-26. August, 1976.

SLIDE 104-0

NOTES

METHOD — 3

Gas Analysis for
Carbon Dioxide, Excess Air and
Dry Molecular Weight

SLIDE 104-1

EPA METHOD 3

PRINCIPLE

A gas sample is collected by one of the following methods:

- Single-point grab sampling
- Single-point integrated sampling
- Multi-point integrated sampling

The sample is analyzed for the following components:

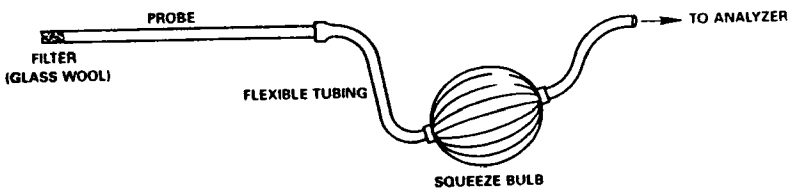
- Carbon dioxide (CO_2)
- Oxygen (O_2)
- Carbon monoxide (CO) (if necessary)

APPLICABILITY

This method is applicable for determining dry molecular weight and excess air correction factor from fossil-fuel combustion sources.

SLIDE 104-2

GRAB SAMPLING TRAIN



SLIDE 104-3

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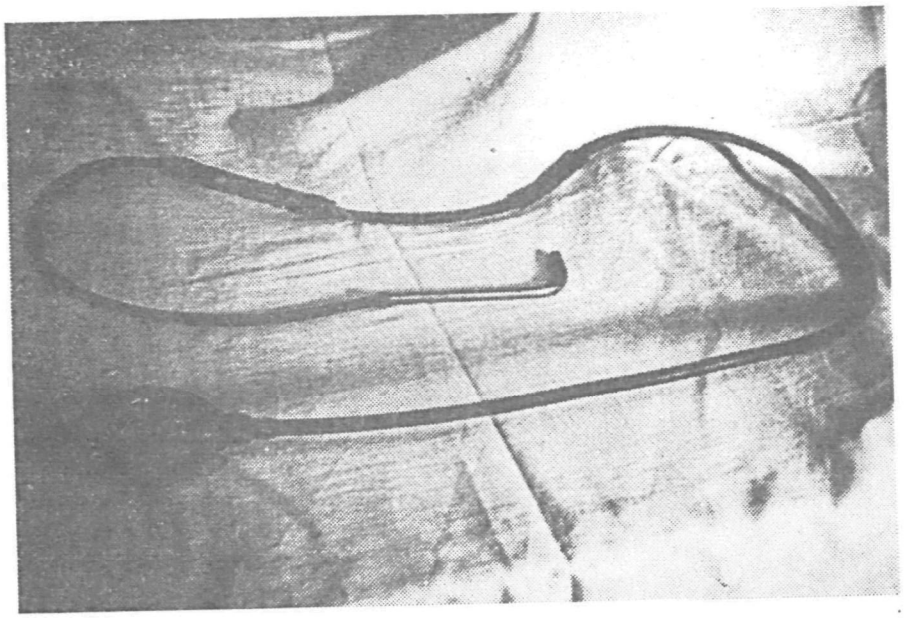
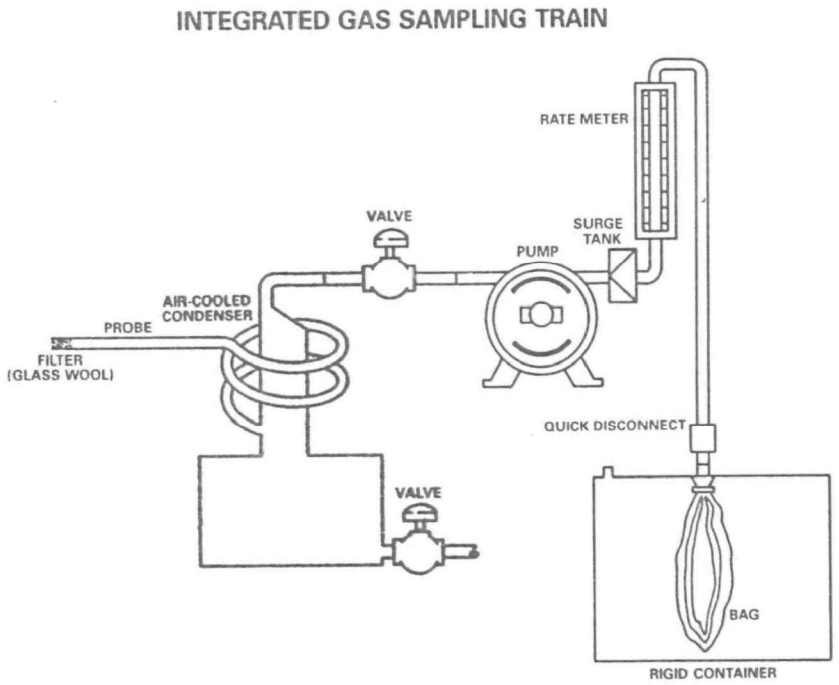


SLIDE 104-4



SLIDE 104-5

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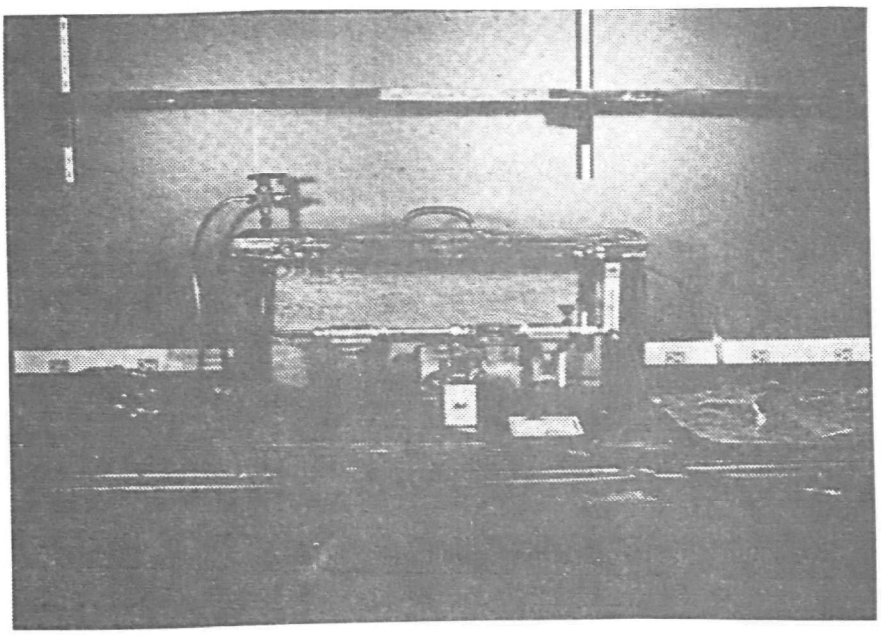
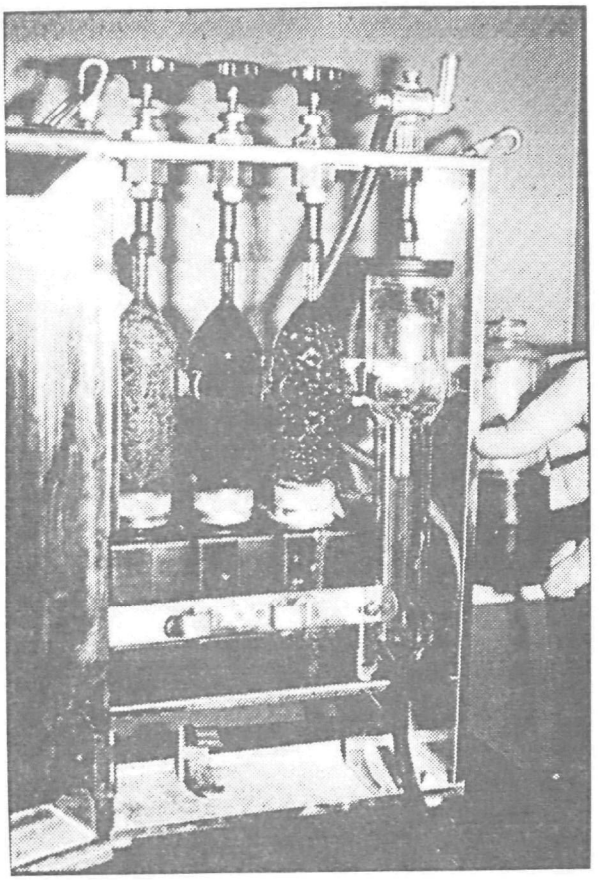


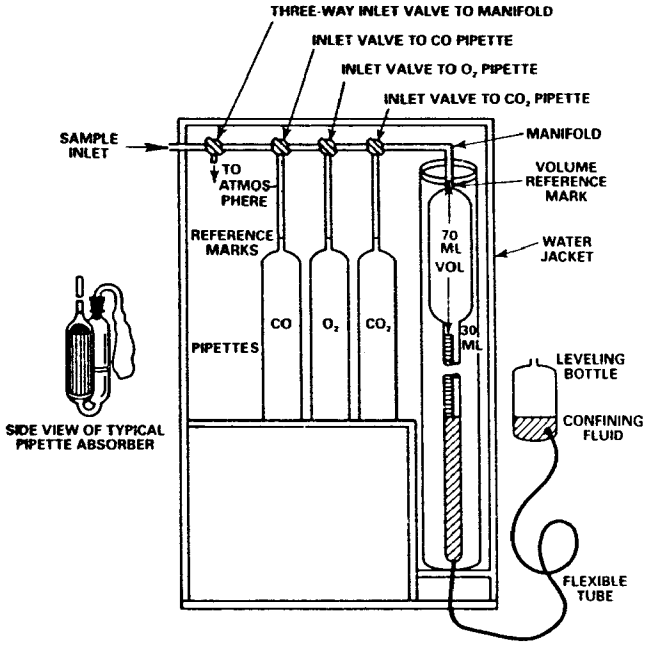
SLIDE 104-6



SLIDE 104-7

NOTES

ORSAT ANALYZER



SLIDE 104-8

ORSAT ANALYZER REAGENTS

GAS CONFINING SOLUTION

- A solution containing sodium sulfate, sulfuric acid and methyl orange

CARBON DIOXIDE ABSORBENT

- A solution of potassium or sodium hydroxide

OXYGEN ABSORBENT

- A solution of alkaline pyrogallic acid or chromous chloride

CARBON MONOXIDE ABSORBENT

- A solution of cuprous chloride or a sulfate solution

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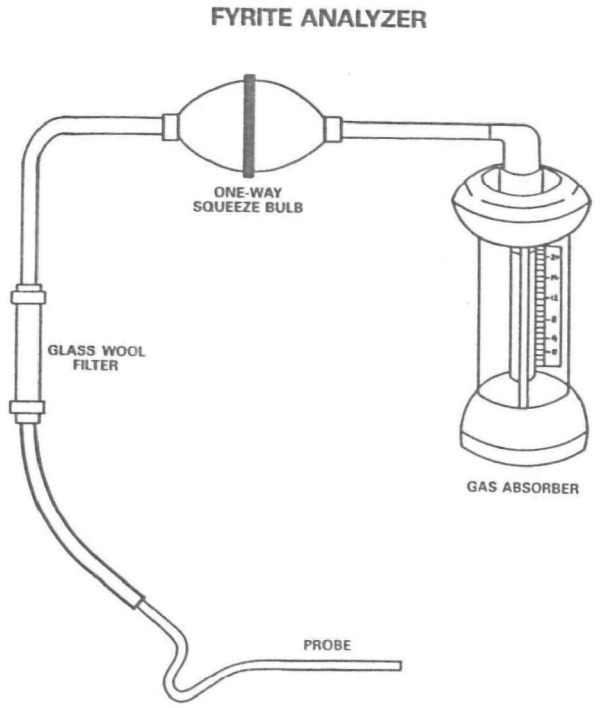
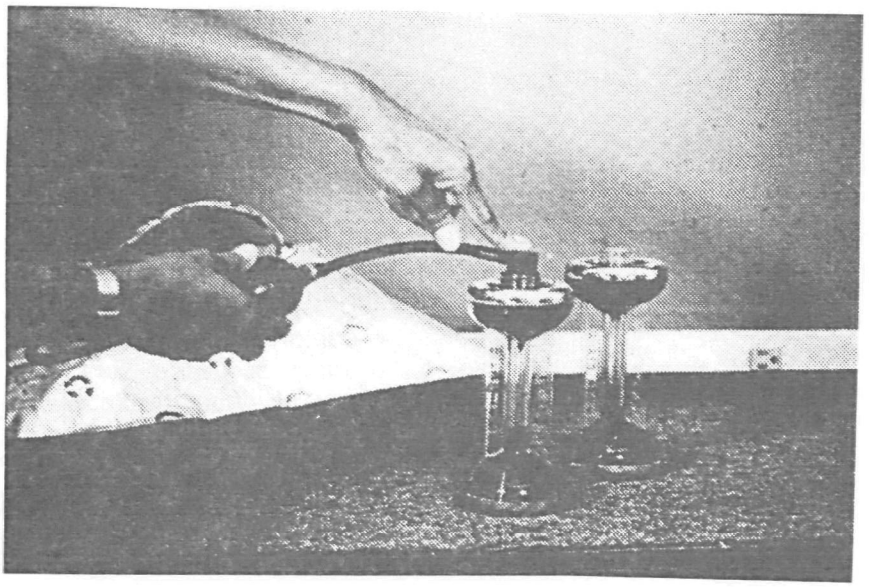
NOTES



SLIDE 104-10



SLIDE 104-11

NOTES

SLIDE 104-12

CALIBRATION OF ANALYZERS

AMBIENT AIR CHECK (O_2 reagent only)

The average of 3 replicates should be 20.8 ± 0.7%.

- a measured average value > 21.5% indicates poor operator technique
- a measured average value < 20.1% indicates a faulty analyzer and/or poor technique

CALIBRATION GAS CHECK

The average of 3 replicates should be ± 0.5% of the known concentration of each gas.

- high measured values indicate poor technique
- low values indicate faulty analyzer and/or poor technique

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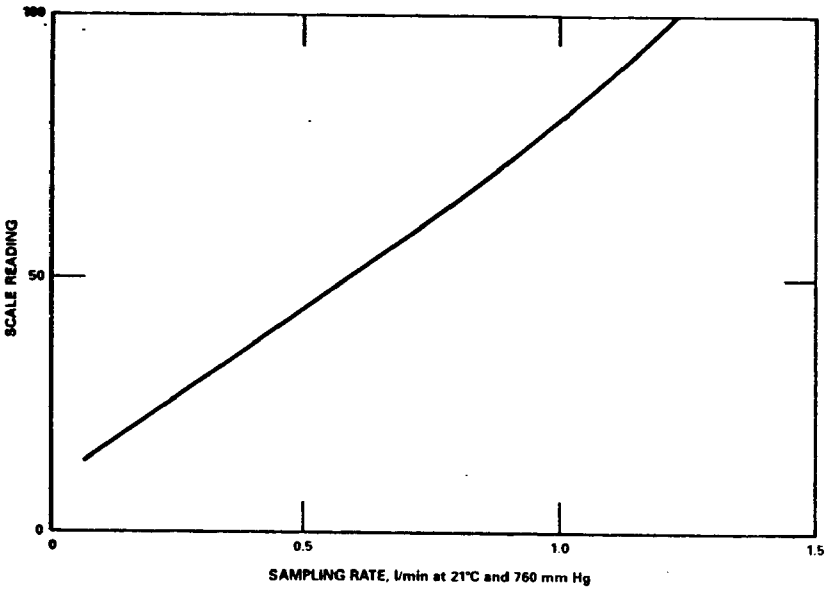
NOTES

ROTAMETER CALIBRATION CURVE



SLIDE 104-14

SAMPLING METHODS

Single-Point Grab Sampling

1. Sample point should be a centriod of the cross section or at a point 3.28 ft. from the walls of a large stack.
2. Place probe securely in stack and seal sampling port to prevent dilution of stack gas.
3. Purge sample line and attach to analyzer.
4. Aspirate sample into analyzer.

SLIDE 104-15

SAMPLING METHODS

Single-Point Integrated Sampling

1. Sample point and probe placement is same as for single-point grab sampling.
2. Leak-check the flexible bag.
3. Leak-check the sampling train.
4. Connect probe to train and purge the system.
5. Connect evacuated flexible bag and begin sampling.
6. Sample at constant rate; collect 30 to 90 liters of gas simultaneous with pollutant emission test.

SLIDE 104-16

NOTES

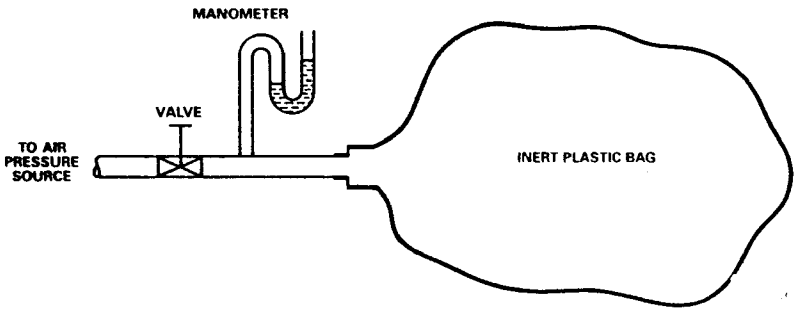
SAMPLING METHODS

Multi-Point Integrated Sampling

1. This procedure uses same sample train and equipment preparation as the single-point integrated sampling method.
2. Locate sampling points according to method 1.
3. Sample each point at the same rate and for the same time increment.
4. Collect 30 to 90 l. of gas simultaneous with pollutant emission test.

SLIDE 104-17

PLASTIC BAG LEAK-CHECK SYSTEM



SLIDE 104-18

INTEGRATED SAMPLE TRAIN LEAK-CHECK PROCEDURE

1. Attach vacuum gauge to condenser inlet.
2. Draw a vacuum of 10 in. Hg and plug line where bag attaches.
3. Turn off pump; vacuum reading should remain stable for 30 seconds.

SLIDE 104-19

NOTES

ORSAT ANALYZER LEAK-CHECK PROCEDURE

1. Bring liquid level in each pipette up to reference mark. Close pipette stopcock.
2. Bring liquid level of confining liquid onto graduated portion of the burette. Close manifold stopcock.
3. Record meniscus position.
4. Observe pipette and burette for 4 minutes.

The orsat analyzer passes leak-check if:

1. The liquid level in each pipette does not fall below bottom of capillary tubing.
2. The meniscus in the burette does not change by more than 0.2 ml.

SLIDE 104-20

DRY MOLECULAR WEIGHT DETERMINATION

1. Sample collection by any one of the three sampling methods.
2. Sample must be analyzed within 8 hrs. after collection.
3. Analysis may be conducted using an orsat or fyrite analyzer.
4. Repeat analysis until any three analyses differ from their mean by no more than 0.3 lb/lb-mole.
5. Average the three molecular weights and report results to nearest 0.1 lb/lb-mole.

SLIDE 104-21

DRY MOLECULAR WEIGHT EQUATION

$$M_d = 0.44(\%CO_2) + 0.32(\%0_2) + 0.28(\%N_2 + \%CO)$$

where: m_d = dry molecular weight
$\%CO_2$ = Percent CO_2 by volume (dry basis)
$\%O_2$ = Percent O_2 by volume (dry basis)
%CO = Percent CO by volume (dry basis)
$\%N_2$ = Percent N_2 by volume (dry basis)
0.44 = molecular weight of CO_2 divided by 100
0.32 = molecular weight of O_2 divided by 100
0.28 = molecular weight of N_2 divided by 100

SLIDE 104-22

NOTES

EMISSION RATE CORRECTION FACTOR OR EXCESS AIR DETERMINATION

1. Sampling method is specified in the standard.
2. Fyrite analyzer cannot be used.
3. Sample must be analyzed within 4 hours after collection.
4. Pre-test and post-test leak-check of the orsat analyzer are mandatory
5. Make repeated passes through each absorbing solution until two consecutive readings are the same.

SLIDE 104-23

(cont.)

6. Repeat analysis until results for any three analyses differ by no more than:

CO_2	O_2	CO
1. 0.3% by volume when CO^2 is > 4.0%.	1. 0.3% by volume when O_2 is < 15.0%.	1. 0.3% by volume
2. 0.2% by volume when CO_2 is ≤ 4.0%.	2. 0.2% by volume when O_2 is > 15.0%.	

7. Average the acceptable values and report results to the nearest 0.1%.

SLIDE 104-24

PERCENT EXCESS AIR EQUATION

$$\%EA = \left[\frac{\%O_2 - 0.5\%CO}{0.264\%N_2 - (\%O_2 - 0.5\%CO)}\right] 100$$

where: %EA = Percent excessive air
$\%O_2$ = Percent O_2 by volume (dry basis)
%CO = Percent CO by volume (dry basis)
$\%N_2$ = Percent N_2 by volume (dry basis)
0.264 = Ratio of O_2 to N_2 in air, V/V

SECTION E. METHOD 4

METHOD 4--DETERMINATION OF MOISTURE CONTENT IN STACK GASES

1. Principle and Applicability

1.1 Principle. A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

1.2 Applicability. This method is applicable for determining the moisture content of stack gas.

Two procedures are given. The first is a reference method, for accurate determinations of moisture content (such as are needed to calculate emission data). The second is an approximation method, which provides estimates of percent moisture to aid in setting isokinetic sampling rates prior to a pollutant emission measurement run. The approximation method described herein is only a suggested approach; alternative means for approximating the moisture content, e.g., drying tubes, wet bulb-dry bulb techniques, condensation techniques, stoichiometric calculations, previous experience, etc., are also acceptable.

The reference method is often conducted simultaneously with a pollutant emission measurement run; when it is, calculation of percent isokinetic, pollutant emission rate, etc., for the run shall be based upon the results of the reference method or its equivalent; these calculations shall not be based upon the results of the approximation method, unless the approximation method is shown, to the satisfaction of the Administrator, U. S. Environmental Protection Agency, to be capable of yielding results within 1 percent H_2O of the reference method.

Note: The reference method may yield questionable results when applied to saturated gas streams or to streams that contain water droplets. Therefore, when these conditions exist or are suspected, a second determination of the moisture content shall be made simultaneously with the reference method, as follows: Assume that the gas stream is saturated. Attach a temperature sensor [capable of measuring to $\pm$ 1° C (2° F)] to the reference method probe. Measure the stack gas temperature at each traverse point (see Section 2.2.1) during the reference method traverse; calculate the average stack gas temperature. Next, determine the moisture percentage, either by: (1) using a psychrometric chart and making appropriate corrections if stack pressure is different from that of the chart, or (2) using saturation vapor pressure tables. In cases where the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternate methods, subject to the approval of the Administrator, shall be used.

2. Reference Method

The procedure described in Method 5 for determining moisture content is acceptable as a reference method.

2.1 Apparatus. A schematic of the sampling train used in this reference method is shown in Figure 4-1. All components shall be maintained and calibrated according to the procedure outlined in Method 5.

2.1.1 Probe. The probe is constructed of stainless steel or glass tubing, sufficiently heated to prevent water condensation, and is equipped with a filter, either in-stack (e.g., a plug of glass

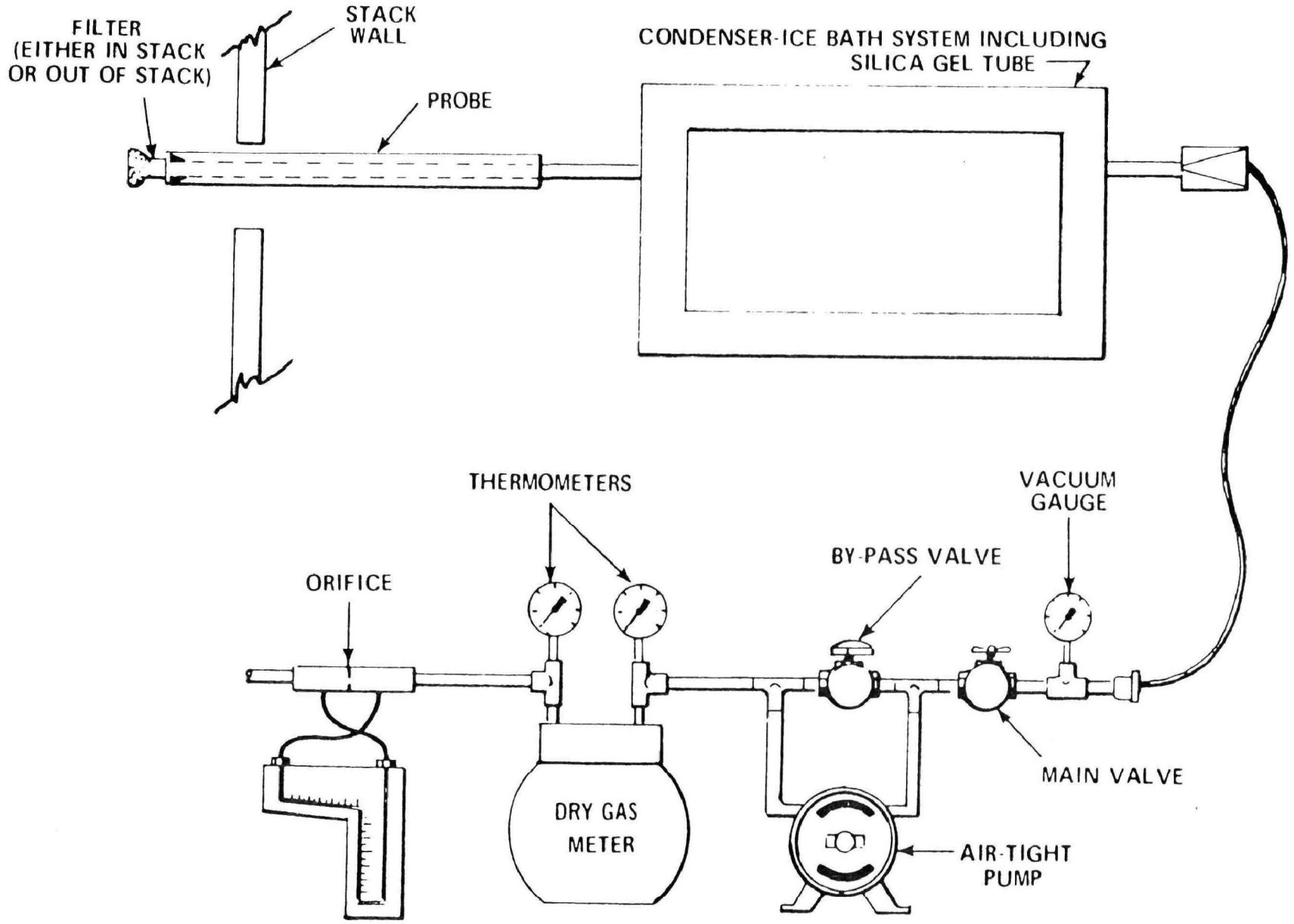


Figure 4-1. Moisture sampling train-reference method.

wool inserted into the end of the probe) or heated out-stack (e.g., as described in Method 5), to remove particulate matter.

When stack conditions permit, other metals or plastic tubing may be used for the probe, subject to the approval of the Administrator.

2.1.2 Condenser. The condenser consists of four impingers connected in series with ground glass, leak-free fittings or any similarly leak-free non-contaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 centimeter (1/2 inch) ID glass tube extending to about 1.3 cm (1/2 in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator.

The first two impingers shall contain known volumes of water, the third shall be empty, and the fourth shall contain a known weight of 6- to 16-mesh indicating type silica gel, or equivalent desiccant. If the silica gel has been previously used, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received. A thermometer, capable of measuring temperature to within 1°C (2°F), shall be placed at the outlet of the fourth impinger, for monitoring purposes.

Alternatively, any system may be used (subject to the approval of the Administrator) that cools the sample gas stream and allows

measurement of both the water that has been condensed and the moisture leaving the condenser, each to within 1 ml or 1 g. Acceptable means are to measure the condensed water, either gravimetrically or volumetrically, and to measure the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures, or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap, with exit gases kept below 20°C (68°F), and determining the weight gain.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump, to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

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

2.1.4 Metering System. This system includes a vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry gas meter capable of measuring volume to within 2 percent, and related equipment as shown in Figure 4-1. Other metering systems, capable of maintaining a constant sampling rate and determining sample gas volume, may be used, subject to the approval of the Administrator.

2.1.5 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg) may be used. In many cases, the barometric reading may be obtained from a nearby national weather service station, in which

case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.6 Graduated Cylinder and/or Balance. These items are used to measure condensed water and moisture caught in the silica gel to within 1 ml or 0.5 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. These balances are suitable for use here.

2.2 Procedure. The following procedure is written for a condenser system (such as the impinger system described in Section 2.1.2) incorporating volumetric analysis to measure the condensed moisture, and silica gel and gravimetric analysis to measure the moisture leaving the condenser.

2.2.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine points shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used in all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to the approval of the Administrator. Select a suitable probe and probe length such that all traverse points can be sampled. Consider sampling from opposite sides of the stack (four total sampling ports) for large stacks, to permit use of shorter probe lengths. Mark the probe with heat resistant tape or by some other method to

denote the proper distance into the stack or duct for each sampling point. Place known volumes of water in the first two impingers. Weigh and record the weight of the silica gel to the nearest 0.5 g, and transfer the silica gel to the fourth impinger; alternatively, the silica gel may first be transferred to the impinger, and the weight of the silica gel plus impinger recorded.

2.2.2 Select a total sampling time such that a minimum total gas volume of 0.60 scm (21 scf) will be collected, at a rate no greater than 0.021 m^3/min (0.75 cfm). When both moisture content and pollutant emission rate are to be determined, the moisture determination shall be simultaneous with, and for the same total length of time as, the pollutant emission rate run, unless otherwise specified in an applicable subpart of the standards.

2.2.3 Set up the sampling train as shown in Figure 4-1. Turn on the probe heater and (if applicable) the filter heating system to temperatures of about 120°C (248°F), to prevent water condensation ahead of the condenser; allow time for the temperatures to stabilize. Place crushed ice in the ice bath container. It is recommended, but not required, that a leak check be done, as follows: Disconnect the probe from the first impinger or (if applicable) from the filter holder. Plug the inlet to the first impinger (or filter holder) and pull a 380 mm (15 in.) Hg vacuum; a lower vacuum may be used, provided that it is not exceeded during the test. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m^3/min (0.02 cfm), whichever is less, is unacceptable. Following the leak check, reconnect the probe to the sampling train.

2.2.4 During the sampling run, maintain a sampling rate within 10 percent of constant rate, or as specified by the Administrator. For each run, record the data required on the example data sheet shown in Figure 4-2. Be sure to record the dry gas meter reading at the beginning and end of each sampling time increment and whenever sampling is halted. Take other appropriate readings at each sample point, at least once during each time increment.

2.2.5 To begin sampling, position the probe tip at the first traverse point. Immediately start the pump and adjust the flow to the desired rate. Traverse the cross section, sampling at each traverse point for an equal length of time. Add more ice and, if necessary, salt to maintain a temperature of less than 20°C (68°F) at the silica gel outlet.

2.2.6 After collecting the sample, disconnect the probe from the filter holder (or from the first impinger) and conduct a leak check (mandatory) as described in Section 2.2.3. Record the leak rate. If the leakage rate exceeds the allowable rate, the tester shall either reject the test results or shall correct the sample volume as in Section 6.3 of Method 5. Next, measure the volume of the moisture condensed to the nearest ml. Determine the increase in weight of the silica gel (or silica gel plus impinger) to the nearest 0.5 g. Record this information (see example data sheet, Figure 4-3) and calculate the moisture percentage, as described in 2.3 below.

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

PLANT _____

LOCATION _____

OPERATOR _____

DATE _____

RUN NO. _____

AMBIENT TEMPERATURE _____

BAROMETRIC PRESSURE _____

PROBE LENGTH m(ft) _____

SCHEMATIC OF STACK CROSS SECTION

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STACK TEMPERATURE °C (°F)	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER (ΔH), mm(in.) H_2O	METER READING GAS SAMPLE VOLUME m^3 (ft^3)	ΔV_m m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
						INLET (Tm_{in}), °C (°F)	OUTLET (Tm_{out}), °C (°F)	
TOTAL						Avg.	Avg.	
AVERAGE						Avg.		

Figure 4-2. Field moisture determination-reference method.

	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g
FINAL		
INITIAL		
DIFFERENCE		

Figure 4-3. Analytical data - reference method.

2.3.1 Nomenclature.

B_{ws} = Proportion of water vapor, by volume, in the gas stream.

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 (mm Hg)(m^3)/(g-mole)(°K) for metric units and 21.85 (in. Hg)(ft^3)/(lb-mole)(°R) for English units.

T_m = Absolute temperature at meter, °K (°R).

T_{std} = Standard absolute temperature, 293°K (528°R).

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

ΔV_m = Incremental dry gas volume measured by dry gas meter at each traverse point, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

$V_{wc(std)}$ = Volume of water vapor condensed corrected to standard conditions, scm (scf).

$V_{wsg(std)}$ = Volume of water vapor collected in silica gel corrected to standard conditions, scm (scf).

V_f = Final volume of condenser water, ml.

V_i = Initial volume, if any, of condenser water, ml.

W_f = Final weight of silica gel or silica gel plus impinger, g.

W_i = Initial weight of silica gel or silica gel plus impinger, g.

Y = Dry gas meter calibration factor.

ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).

2.3.2 Volume of water vapor condensed.

$$V_{wc(std)} = \frac{(V_f - V_i)\ \rho_w\ R\ T_{std}}{P_{std}\ M_w}$$

$$= K_1\ (V_f - V_i) \qquad \text{Equation 4-1}$$

where:

$K_1 = 0.001333\ m^3/ml$ for metric units

$= 0.04707\ ft^3/ml$ for English units

2.3.3 Volume of water vapor collected in silica gel.

$$V_{wsg(std)} = \frac{(W_f - W_i)\ R\ T_{std}}{P_{std}\ M_w}$$

$$= K_2\ (W_f - W_i) \qquad \text{Equation 4-2}$$

where:

$K_2 = 0.001335\ m^3/g$ for metric units

$= 0.04715\ ft^3/g$ for English units

2.3.4 Sample gas volume.

$$V_{m(std)} = V_m Y \frac{(P_m)\ (T_{std})}{(P_{std})\ (T_m)}$$

$$= K_3 Y \frac{V_m\ P_m}{T_m} \qquad \text{Equation 4-3}$$

where:

$K_3 = 0.3858\ °K/mm$ Hg for metric units

$= 17.64\ °R/in.$ Hg for English units

Note: If the post-test leak rate (Section 2.2.6) exceeds the allowable rate, correct the value of V_m in Equation 4-3, as described in Section 6.3 of Method 5.

2.3.5 Moisture Content.

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}} \qquad \text{Equation 4-4}$$

Note: In saturated or moisture droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one using a value based upon the saturated conditions (see Section 1.2), and another based upon the results of the impinger analysis. The lower of these two values of B_{ws} shall be considered correct.

2.3.6 Verification of constant sampling rate. For each time increment, determine the ΔV_m. Calculate the average. If the value for any time increment differs from the average by more than 10 percent, reject the results and repeat the run.

3. Approximation Method

The approximation method described below is presented only as a suggested method (see Section 1.2).

3.1 Apparatus.

3.1.1 Probe. Stainless steel or glass tubing, sufficiently heated to prevent water condensation and equipped with a filter (either in-stack or heated out-stack) to remove particulate matter. A plug of glass wool, inserted into the end of the probe, is a satisfactory filter.

3.1.2 Impingers. Two midget impingers, each with 30 ml capacity, or equivalent.

3.1.3 Ice Bath. Container and ice, to aid in condensing moisture in impingers.

3.1.4 Drying Tube. Tube packed with new or regenerated 6- to 16-mesh indicating-type silica gel (or equivalent desiccant), to dry the sample gas and to protect the meter and pump.

3.1.5 Valve. Needle valve, to regulate the sample gas flow rate.

3.1.6 Pump. Leak-free, diaphragm type, or equivalent, to pull the gas sample through the train.

3.1.7 Volume meter. Dry gas meter, sufficiently accurate to measure the sample volume within 2%, and calibrated over the range of flow rates and conditions actually encountered during sampling.

3.1.8 Rate Meter. Rotameter, to measure the flow range from 0. to 3 lpm (0. to 0.11 cfm).

3.1.9 Graduated Cylinder. 25 ml.

3.1.10 Barometer. Mercury, aneroid, or other barometer, as described in Section 2.1.5 above.

3.1.11 Vacuum Gauge. At least 760 mm Hg (30 in. Hg) gauge, to be used for the sampling leak check.

3.2 Procedure.

3.2.1 Place exactly 5 ml distilled water in each impinger. Leak check the sampling train as follows: Temporarily insert a vacuum gauge at or near the probe inlet; then, plug the probe inlet and pull a vacuum of at least 250 mm Hg (10 in. Hg). Note the time rate of change of the dry gas meter dial; alternatively, a rotameter (0-40 cc/min) may be temporarily attached to the dry gas meter outlet to determine the leakage rate. A leak rate not in excess of 2 percent of the average sampling rate is acceptable. Note: Carefully release the probe inlet plug before turning off the pump.

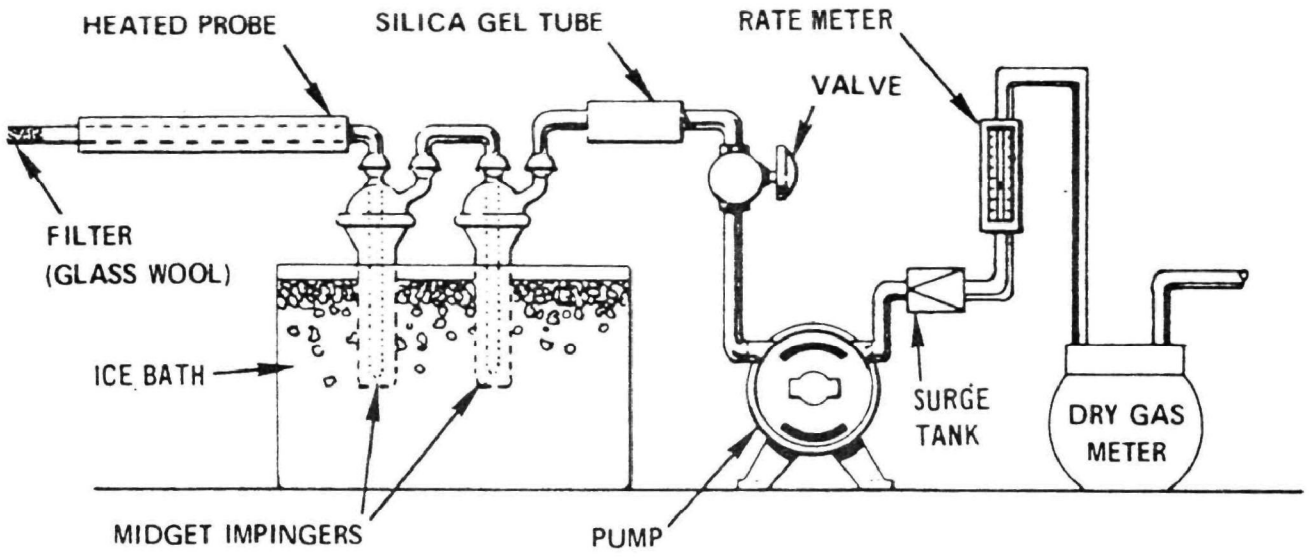


Figure 4-4. Moisture-sampling train - approximation method.

LOCATION _____ COMMENTS

TEST _____

DATE _____

OPERATOR _____

BAROMETRIC PRESSURE _____

CLOCK TIME	GAS VOLUME THROUGH METER, (Vm), m^3 (ft^3)	RATE METER SETTING m^3/min. (ft^3/min.)	METER TEMPERATURE, °C (°F)

Figure 4-5. Field moisture determination - approximation method.

3.2.2 Connect the probe, insert it into the stack, and sample at a constant rate of 2 lpm (0.071 cfm). Continue sampling until the dry gas meter registers about 30 liters (1.1 ft^3) or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4-5.

3.2.3 After collecting the sample, combine the contents of the two impingers and measure the volume to the nearest 0.5 ml.

3.3 Calculations. The calculation method presented is designed to estimate the moisture in the stack gas; therefore, other data, which are only necessary for accurate moisture determinations, are not collected. The following equations adequately estimate the moisture content, for the purpose of determining isokinetic sampling rate settings.

3.3.1 Nomenclature.

B_{wm} = Approximate proportion, by volume, of water vapor in the gas stream leaving the second impinger, 0.025.

B_{ws} = Water vapor in the gas stream, proportion by volume.

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole)

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter.

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 (mm Hg)(m^3)/(g-mole)(°K) for metric units and 21.85 (in. Hg)(ft^3)/lb-mole)(°R) for English units.

T_m = Absolute temperature at meter, °K (°R).

T_{std} = Standard absolute temperature, 293°K (528°R).

V_f = Final volume of impinger contents, ml.

V_i = Initial volume of impinger contents, ml.

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by dry gas meter, corrected to standard conditions, dscm (dscf).

$V_{wc(std)}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).

Y = Dry gas meter calibration factor.

ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).

3.3.2 Volume of water vapor collected.

$$V_{wc(std)} = \frac{(V_f - V_i)\ \rho_w\ R\ T_{std}}{P_{std}\ M_w}$$

$$= K_1\ (V_f - V_i) \qquad \text{Equation 4-5}$$

Where:

K_1 = 0.001333 m^3/ml for metric units.

= 0.04707 ft^3/ml for English units.

3.3.3 Gas Volume.

$$V_{m(std)} = V_m\ Y \left(\frac{P_m}{P_{std}}\right)\left(\frac{T_{std}}{T_m}\right) = K_2\ Y\ \frac{V_m\ P_m}{T_m}$$

Equation 4-6

where:

K_2 = 0.3858 °K/mm Hg for metric units.

= 17.64 °R/in. Hg for English units.

3.3.4 Approximate moisture content.

$$B_{ws} = \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + B_{wm} = \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + (0.025)$$

Equation 4-7

4. Calibration

4.1 For the reference method, calibrate equipment as specified in the following sections of Method 5: Section 5.3 (metering system); Section 5.5 (temperature gauges); and Section 5.7 (barometer). The recommended leak check of the metering system (Section 5.6 of Method 5) also applies to the reference method. For the approximation method, use the procedures outlined in Section 5.1.1 of Method 6 to calibrate the metering system, and the procedure of Method 5, Section 5.7 to calibrate the barometer.

5. Bibliography

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3. Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases. Western Precipitation Division of Joy Manufacturing Co. Los Angeles, Calif. Bulletin WP-50. 1968.

SLIDE 105-0

NOTES

METHOD — 4

Determination of Moisture
in Stack Gases

SLIDE 105-1

PRINCIPLE

A sample is extracted at a constant rate. Moisture is removed and determined volumetrically or gravimetrically.

APPLICABILITY

The method is applicable for determining moisture content of stack gas.

SLIDE 105-2

SUMMARY OF METHODS

REFERENCE METHOD

- used for accurate determination of moisture content
- usually conducted simultaneously with a pollutant measurement run
- results used to calculate isokinetics and pollutant emission rate

APPROMIXATION METHOD

- used to estimate percent moisture to aid in setting isokinetic sampling rate

SLIDE 105-3

NOTES

(cont.) SUMMARY OF METHODS

PARTIAL PRESSURE METHOD

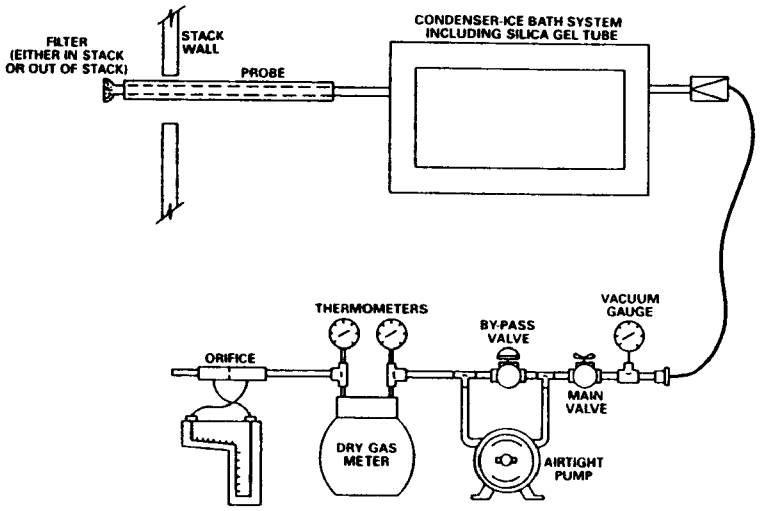
- used to determine moisture content in saturated gas streams and gas streams that contain water droplets.

WET BULB - DRY BULB METHOD

- a popular alternative approximation method for low temperature applications.

SLIDE 105-4

MOISTURE SAMPLING TRAIN (REFERENCE METHOD).



SLIDE 105-5

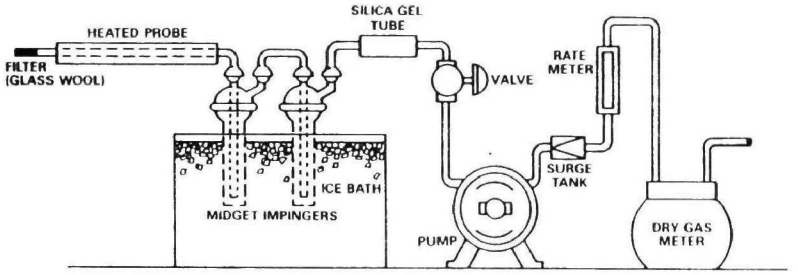
PROCEDURE

1. Determine traverse points using Method 1.
2. Select sampling time such that minimum gas volume of 21 SCF will be collected at rate no greater than 0.75 CFM.
3. Leak-check sampling train (optional).
4. Maintain sampling rate within 10% of constant rate.
5. After sampling, leak-check sampling train (mandatory).
6. Verify the constant sampling rate.

SLIDE 105-6

NOTES

MOISTURE SAMPLING TRAIN — APPROXIMATION METHOD



SLIDE 105-7

PROCEDURE

1. Place 5.0 ml of distilled water in each impinger.
2. Assemble and leak-check sampling train.
3. Sample at a constant rate of 0.07 CFM until a sample volume of 1.1 ft^3 is obtained.
4. Combine contents of impingers and measure volume to nearest 0.5 ml.

SLIDE 105-8

PARTIAL PRESSURE METHOD

1. Assume saturation.
2. Attach temperature sensor to reference method probe.
3. Measure stack gas temperature at each traverse point.
4. Calculate the average stack gas temperature.
5. Determine moisture fraction using saturation vapor pressure table.

SLIDE 105-9

NOTES

MOISTURE EQUATION - PARTIAL PRESSURE

$$B_{ws} = \frac{S.V.P.}{P}$$

Where: B_{ws} = proportion (by volume) of water vapor in a gas mixture
S.V.P. = saturated vapor pressure of water at average stack temperature
P = absolute pressure of the stack

SLIDE 105-10

EXAMPLE MOISTURE CALCULATION

DATA

Average stack temperature	140°F
Barometric pressure	29.2 in. Hg
Static pressure	+ 0.5 in. Hg
Saturated vapor pressure	5.88 in. Hg

CALCULATION

$$B_{ws} = \frac{5.88}{29.7}$$

$$B_{ws} = 0.1980$$

SLIDE 105-11

WET BULB - DRY BULB METHOD

1. Measure the wet bulb temperature.
2. Measure the dry bulb temperature.
3. Estimate moisture content using psychrometric chart.

MOISTURE EQUATION
(WET BULB - DRY BULB)

$$B_{ws} = \frac{V.P.}{P}$$

Where: $V.P. = S.V.P. - \left[(0.000367)\,(P)\,(t_d - t_w)\left(1+\frac{t_w - 32}{1571}\right)\right]$

S.V.P. = saturated water vapor pressure at the wet bulb temperature

P = absolute pressure in the stack

t_d = dry bulb temperature

t_w = wet bulb temperature

V.P. = water vapor pressure

SECTION F. METHOD 5

METHOD 5--DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of 120 ± 14°C (248 ± 25°F) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator, U. S. Environmental Protection Agency, for a particular application. The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources.

2. Apparatus

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 5-1. Complete construction details are given in APTD-0581 (Citation 2 in Section 7); commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure 5-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576 (Citation 3 in Section 7). Since correct usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

2.1.1 Probe Nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper shall be $\leq 30°$ and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing; other materials of construction may be used, subject to the approval of the Administrator.

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (1/8 to 1/2 in.)--or, larger if higher volume sampling trains are used--inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in Section 5.

2.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of 120 $\pm$ 14°C (248 $\pm$ 25°F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. (The tester may opt to operate the equipment at a temperature lower than that specified.) Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable.

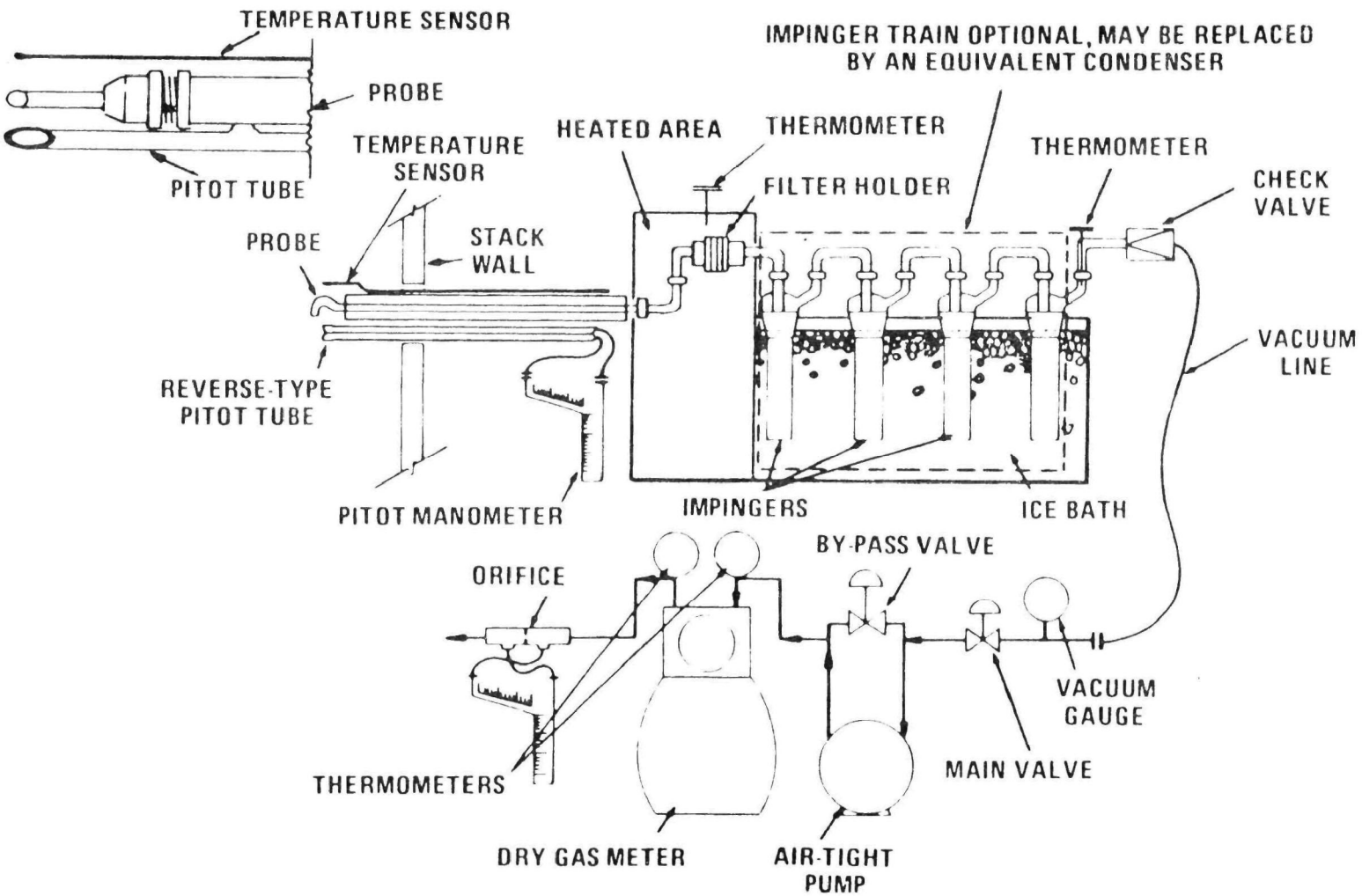


Figure 5 1. Particulate sampling train.

Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480°C (900°F); quartz liners shall be used for temperatures between 480 and 900°C (900 and 1650°F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate is 820°C (1508°F), and for quartz it is 1500°C (2732°F).

Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825,[1] or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

2.1.3 Pitot Tube. Type S, as described in Section 2.1 of Method 2, or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure 5-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of Method 2.

2.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used for velocity head (Δp) readings, and the other, for orifice differential pressure readings.

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[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel, Teflon, Viton) may be used, subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

2.1.6 Filter Heating System. Any heating system capable of maintaining a temperature around the filter holder during sampling of 120 ± 14°C (248 ± 25°F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. Alternatively, the tester may opt to operate the equipment at a temperature lower than that specified. A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

2.1.7 Condenser. The following system shall be used to determine the stack gas moisture content: Four impingers connected in series with leak-free ground glass fittings or any similar leak-free non-contaminating fittings. The first, third, and fourth impingers shall 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 (1/2 in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g.,

using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator. The first and second impingers shall contain known quantities of water (Section 4.1.3), the third shall be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A thermometer, capable of measuring temperature to within 1°C (2°F) shall be placed at the outlet of the fourth impinger for monitoring purposes.

Alternatively, any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g may be used, subject to the approval of the Administrator. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap with exit gases kept below 20°C (68°F) and determining the weight gain.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

Note: If a determination of the particulate matter collected in the impingers is desired in addition to moisture content, the impinger system described above shall be used, without modification. Individual States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

2.1.8 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

2.1.9 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.10 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attched just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2-7). As a second alternative, if a difference of not more than 1 percent in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

2.2 Sample Recovery. The following items are needed:

2.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Wash Bottles--Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

2.2.4 Petri Dishes. For filter samples, glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.

2.3 Analysis. For analysis, the following equipment is needed:

2.3.1 Glass Weighing Dishes.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure to within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 g.

2.3.5 Beakers. 250 ml.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

3. Reagents

3.1 Sampling. The reagents used in sampling are as follows:

3.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency ($\leq$0.05 percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D 2986-71. Test data from the supplier's quality control program are sufficient for this purpose.

In sources containing SO_2 or SO_3, the filter material must of a type that is unreactive to SO_2 or SO_3. Citation 10 in Section 7 may be used to select the appropriate filter.

3.1.2 Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 Water. When analysis of the material caught in the impingers is required, distilled water shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

3.1.4 Crushed Ice.

3.1.5 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

3.2 Sample Recovery. Acetone--reagent grade, ≤0.001 percent residue, in glass bottles--is required. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers; thus, acetone blanks shall be run prior to field use and only acetone with low blank values (≤0.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

3.3 Analysis. Two reagents are required for the analysis:

3.3.1 Acetone. Same as 3.2.

3.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

4. Procedure

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative,

label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and wejghing.

Desiccate the filters at 20 ± 5.6°C (68 ± 10°F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., ≤0.5 mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105°C (220°F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the particulate sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 min. (or some greater time interval as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors. The sampling time at each point shall be the same.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination

can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

When glass liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260°C (500°F) and an asbestos string gasket when temperatures are higher. See APTD-0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Set up the train as in Figure 5-1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

Place crushed ice around the impingers.

4.1.4 Leak-Check Procedures.

4.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum.

Note: A lower vacuum may be used, provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm Hg (15 in. Hg) vacuum (see Note immediately above). Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum; alternatively, the probe may be leak-checked with

the rest of the sampling train, in one step, at 380 mm Hg (15 in. Hg) vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m^3/min (0.02 cfm), whichever is less, are unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with bypass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable) and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and silica gel from being entrained backward into the third impinger.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4 percent of the average sampling rate

(whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

4.1.4.3 Post-test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

4.1.5 Particulate Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature around the filter of 120 $\pm$ 14°C (248 $\pm$ 25°F), or such other

temperature as specified by an applicable subpart of the standards or approved by the Administrator.

For each run, record the data required on a data sheet such as the one shown in Figure 5-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure 5-2 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

Clean the portholes prior to the test run to minimize the chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient is 0.85 ± 0.02, and the stack gas equivalent density (dry molecular weight) is equal to 29 ± 4. APTD-0576 details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Citation 7 in Section 7) are taken to compensate for the deviations.

PLANT _____

LOCATION _____

OPERATOR _____

DATE _____

RUN NO. _____

SAMPLE BOX NO. _____

METER BOX NO. _____

METER ΔH@ _____

C FACTOR _____

PITOT TUBE COEFFICIENT, C_p _____

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE _____

BAROMETRIC PRESSURE _____

ASSUMED MOISTURE, % _____

PROBE LENGTH, m (ft) _____

NOZZLE IDENTIFICATION NO. _____

AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) _____

PROBE HEATER SETTING _____

LEAK RATE, m^3/min. (cfm) _____

PROBE LINER MATERIAL _____

STATIC PRESSURE, mm Hg (in. Hg) _____

FILTER NO. _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	VACUUM mm Hg (in Hg)	STACK TEMPERATURE (T_S) °C (°F)	VELOCITY HEAD (ΔP_S), mm(in.)H_2O	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER mm H_2O (in. H_2O)	GAS SAMPLE VOLUME m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		FILTER HOLDER TEMPERATURE °C (°F)	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
							INLET °C (°F)	OUTLET °C (°F)		
TOTAL							Avg.	Avg.		
AVERAGE							Avg.			

Figure 5-2. Particulate field data.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent water from backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross-section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes; this minimizes the chance of extracting deposited material.

During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper level; add more ice and, if necessary, salt to maintain a temperature of less than 20°C (68°F) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak-check (see Section 4.1.4.2). The total particulate weight shall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

Note that when two or more trains are used, separate analyses of the front-half and (if applicable) impinger catches from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case, the front-half catches from the individual trains may be combined (as may the impinger catches) and one analysis of front-half catch and one analysis of impinger catch may be performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

At the end of the sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Method 2, Section 3.1; the lines must pass this leak-check, in order to validate the velocity head data.

4.1.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Calculations, Section 6) to determine whether the run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample Recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter holder.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the acetone used for cleanup as a blank. Take 200 ml of this acetone directly from the wash bottle being used and place it in a glass sample container labeled "acetone blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the petri dish any particulate matter and/or filter fibers which adhere to the filter holder gasket, by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow the Administrator's directions on analysis. Perform the acetone rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a Nylon bristle brush. Brush until the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone..

Brush and rinse the inside parts of the Swagelok fitting with acetone in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Let the acetone drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the acetone or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints have been wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone, also (if applicable). After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.

Container No. 3. Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water

or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for container No. 3 in Section 4.3.

Impinger Water. Treat the impingers as follows: Make a notation of any color or film in the liquid catch. Measure the liquid which is in the first three impingers to within ±1 ml by using a graduated cylinder or by weighing it to within ±0.5 g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see Note, Section 2.1.7).

If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

Whenever possible, containers should be shipped in such a way that they remain upright at all times.

4.3 Analysis. Record the data required on a sheet such as the one shown in Figure 5-3. Handle each sample container as follows:

Container No. 1. Leave the contents in the shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this Section, 4.3, the term "constant weight" means

Plant _____

Date _____

Run No. _____

Filter No. _____

Amount liquid lost during transport _____

Acetone blank volume, ml _____

Acetone wash volume, ml _____

Acetone blank concentration, mg/mg (equation 5-4) _____

Acetone wash blank, mg (equation 5-5) _____

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			
		Less acetone blank	
		Weight of particulate matter	

	VOLUME OF LIQUID WATER COLLECTED		
	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g	
FINAL			
INITIAL			
LIQUID COLLECTED			
TOTAL VOLUME COLLECTED		g*	ml

*CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER (1g/ml).

$$\frac{\text{INCREASE, g}}{\text{1 g/ml}} = \text{VOLUME WATER, ml}$$

Figure 5-3. Analytical data.

a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings.

Alternatively, the sample may be oven dried at 105°C (220°F) for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The tester may also opt to oven dry the sample at 105°C (220°F) for 2 to 3 hours, weigh the sample, and use this weight as a final weight.

Container No. 2. Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

Container No. 3. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

"Acetone Blank" Container. Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

Note: At the option of the tester, the contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

5. Calibration

Maintain a laboratory log of all calibrations.

5.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

5.2 Pitot Tube. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of Method 2.

5.3 Metering System. Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry gas meter dial readings to correspond to the wet test meter readings, calibration factors may be used to mathematically correct the gas meter dial readings to the proper values. Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm

pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.00057 m^3/min (0.02 cfm); at the end of the run, take the difference of the measured wet test meter and dry gas meter volumes; divide the difference by 10, to get the leak rate. The leak rate should not exceed 0.00057 m^3/min (0.02 cfm).

After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

Alternative procedures, e.g., using the orifice meter coefficients, may be used, subject to the approval of the Administrator.

Note: If the dry gas meter coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

5.4 Probe Heater Calibration. The probe heating system shall be calibrated before its initial use in the field according to the procedure outlined in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

5.5 Temperature Gauges. Use the procedure in Section 4.3 of Method 2 to calibrate in-stack temperature gauges. Dial thermometers,

such as are used for the dry gas meter and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

5.6 Leak Check of Metering System Shown in Figure 5-1. That portion of the sampling train from the pump to the orifice meter should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 5-4): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

5.7 Barometer. Calibrate against a mercury barometer.

6. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

6.1 Nomenclature.

A_n = Cross-sectional area of nozzle, m^2 (ft^2).

B_{ws} = Water vapor in the gas stream, proportion by volume.

C_a = Acetone blank residue concentration, mg/g.

c_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).

I = Percent of isokinetic sampling.

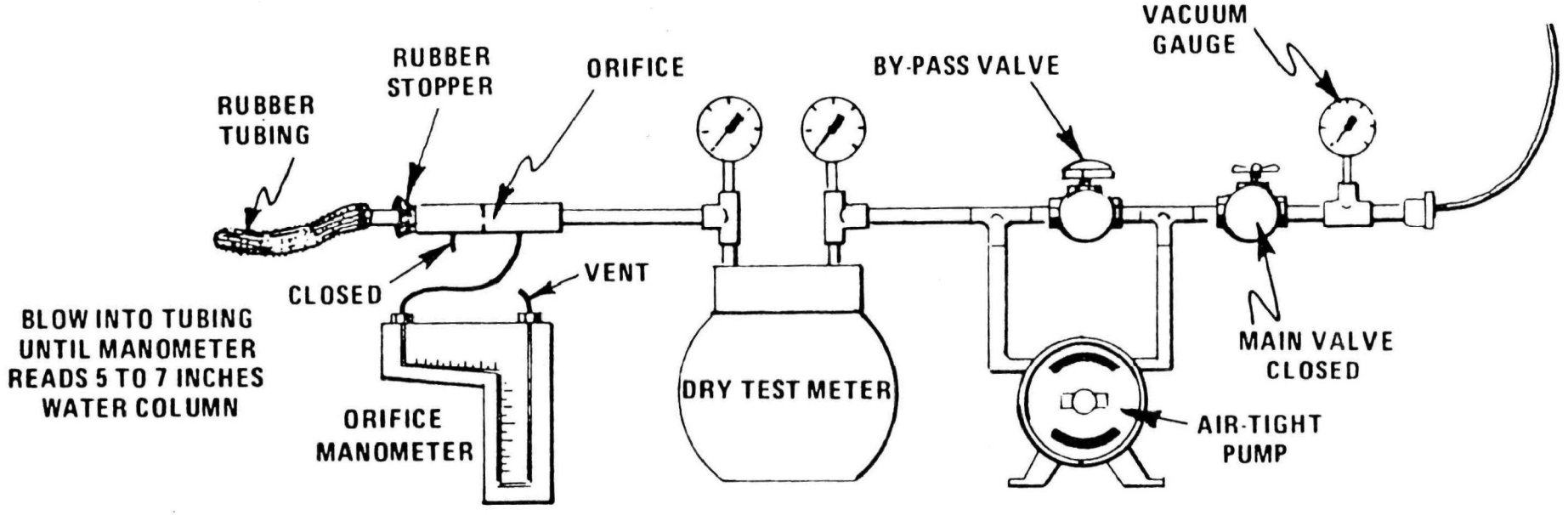

Figure 5-4. Leak check of meter box.

L_a = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m^3/min (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.

L_i = Individual leakage rate observed during the leak check conducted prior to the "i^{th}" component change (i = 1, 2, 3....n), m^3/min (cfm).

L_p = Leakage rate observed during the post-test leak check, m^3/min (cfm).

m_n = Total amount of particulate matter collected, mg.

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

m_a = Mass of residue of acetone after evaporation, mg.

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 mm Hg-m^3/°K-g-mole (21.85 in. Hg-ft^3/°R-lb-mole).

T_m = Absolute average dry gas meter temperature (see Figure 5-2), °K (°R).

T_s = Absolute average stack gas temperature (see Figure 5-2), °K (°R).

T_{std} = Standard absolute temperature, 293°K (528°R).

V_a = Volume of acetone blank, ml.

V_{aw} = Volume of acetone used in wash, ml.

V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.

V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).

v_s = Stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 5, m/sec (ft/sec).

W_a = Weight of residue in acetone wash, mg.

Y = Dry gas meter calibration factor.

ΔH = Average pressure differential across the orifice meter (see Figure 5-2), mm H_2O (in. H_2O).

ρ_a = Density of acetone, mg/ml (see label on bottle).

ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).

θ = Total sampling time, min.

θ_1 = Sampling time interval, from the beginning of a run until the first component change, min.

θ_i = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.

θ_p = Sampling time interval, from the final (n^{th}) component change until the end of the sampling run, min.

13.6 = Specific gravity of mercury.

60 = Sec/min.

100 = Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5-2).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20°C, 760 mm Hg or 68°F, 29.92 in. Hg) by using Equation 5-1.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right)\left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right] = K_1 V_m Y \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

Equation 5-1

where:

K_1 = 0.3858 °K/mm Hg for metric units

= 17.64 °R/in. Hg for English units

Note: Equation 5-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds L_a. If L_p or L_i exceeds L_a, Equation 5-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_m in Equation 5-1 with the expression:

$$[V_m - (L_p - L_a)\,\theta]$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_m in Equation 5-1 by the expression:

$$[V_m - (L_1 - L_a)\,\theta_1 - \sum_{i=2}^{n} (L_i - L_a)\,\theta_i - (L_p - L_a)\,\theta_p]$$

and substitute only for those leakage rates (L_i or L_p) which exceed L_a.

6.4 Volume of water vapor.

$$V_{w(std)} = V_{lc}\left(\frac{\rho_w}{M_w}\right)\left(\frac{RT_{std}}{P_{std}}\right) = K_2\,V_{lc} \qquad \text{Equation 5-2}$$

where:

K_2 = 0.001333 m^3/ml for metric units

= 0.04707 ft^3/ml for English units.

6.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \qquad \text{Equation 5-3}$$

Note: In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 5-3), and a second from the assumption of saturated conditions. The lower of the two values of B_{ws} shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note of Section 1.2 of Method 4. For the purposes of this method, the average stack gas temperature from Figure 5-2 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is $\pm$ 1°C (2°F).

6.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a \rho_a}$$ Equation 5-4

6.7 Acetone Wash Blank.

$$W_a = C_a V_{aw} \rho_a$$ Equation 5-5

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the acetone blank (see Figure 5-3). Note: Refer to Section 4.1.5 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$c_s = (0.001 \text{ g/mg}) \ (m_n/V_{m(std)})$$ Equation 5-6

6.10 Conversion Factors:

From	To	Multiply by
scf	m^3	0.02832
g/ft^3	gr/ft^3	15.43
g/ft^3	lb/ft^3	2.205×10^{-3}
g/ft^3	g/m^3	35.31

6.11 Isokinetic Variation.

6.11.1 Calculation From Raw Data.

$$I = \frac{100 \ T_s \ [K_3 \ V_{lc} + (V_m \ Y/T_m) \ (P_{bar} + \Delta H/13.6)]}{60 \ \theta \ v_s \ P_s \ A_n}$$

Equation 5-7

where:

K_3 = 0.003454 mm Hg-m^3/ml-°K for metric units

= 0.002669 in. Hg-ft^3/ml-°R for English units.

6.11.2 Calculation From Intermediate Values.

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} v_s \theta A_n P_s 60 (1-B_{ws})}$$

$$= K_4 \frac{T_s V_{m(std)}}{P_s v_s A_n \theta (1-B_{ws})} \qquad \text{Equation 5-8}$$

where:

K_4 = 4.320 for metric units

= 0.09450 for English units.

6.12 Acceptable Results. If 90 percent $\leq$ I $\leq$ 110 percent, the results are acceptable. If the results are low in comparison to the standard and I is beyond the acceptable range, or, if I is less than 90 percent, the Administrator may opt to accept the results. Use Citation 4 to make judgments. Otherwise, reject the results and repeat the test.

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SLIDE 106-0

NOTES

METHOD — 5

Determination of Particulate Emissions From Stationary Sources

SLIDE 106-1

PRINCIPLE

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of 248 ± 25°F.

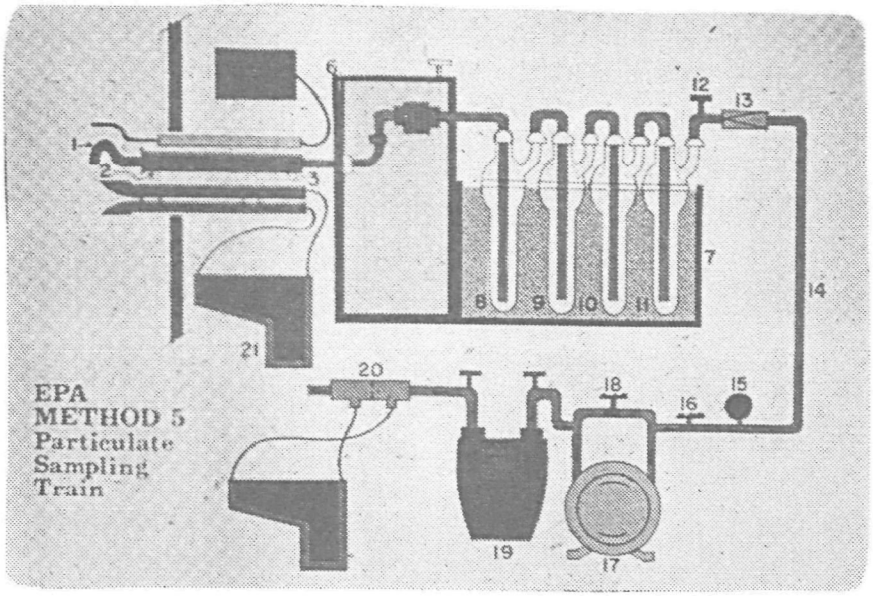
The particulate mass is determined gravimetrically after removal of uncombined water.

APPLICABILITY

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

SLIDE 106-2

NOTES



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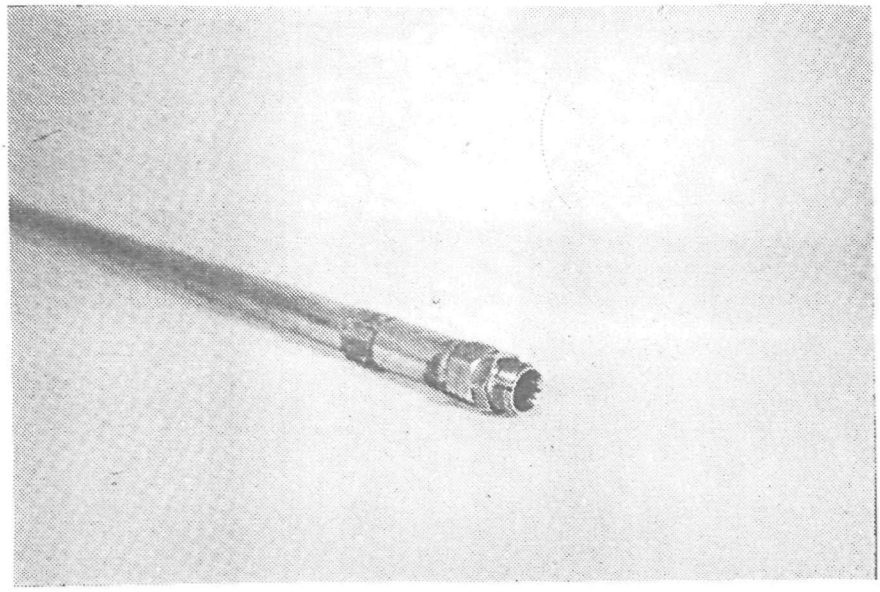

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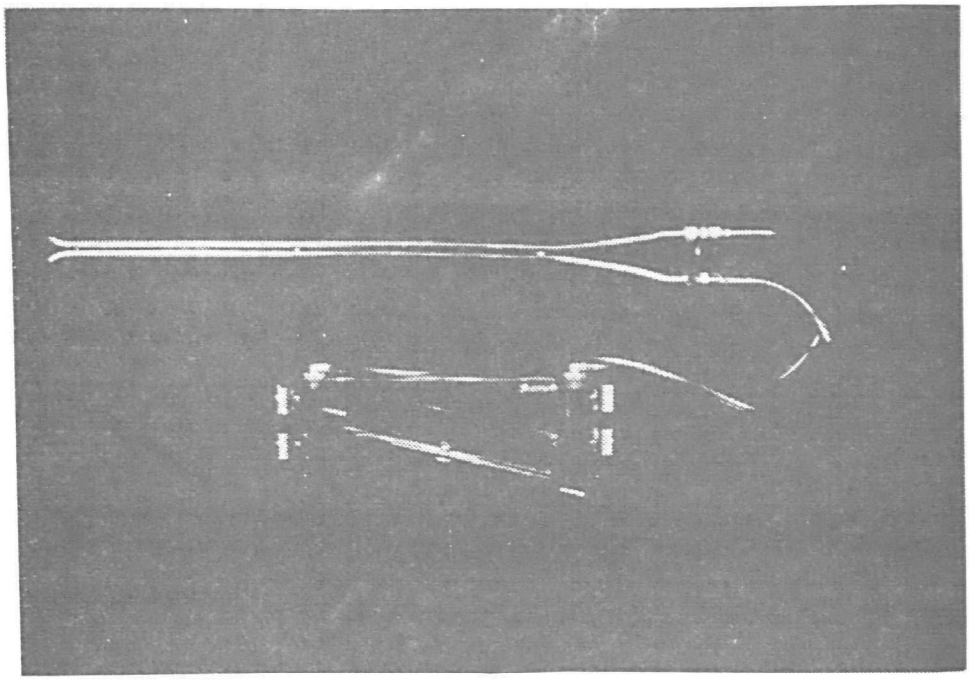

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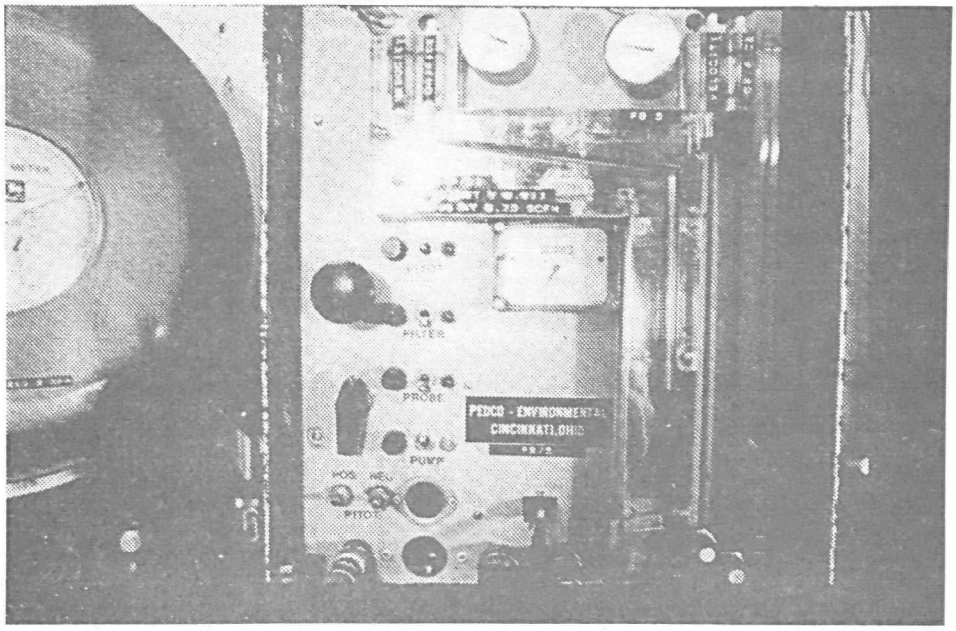

SLIDE 106-6

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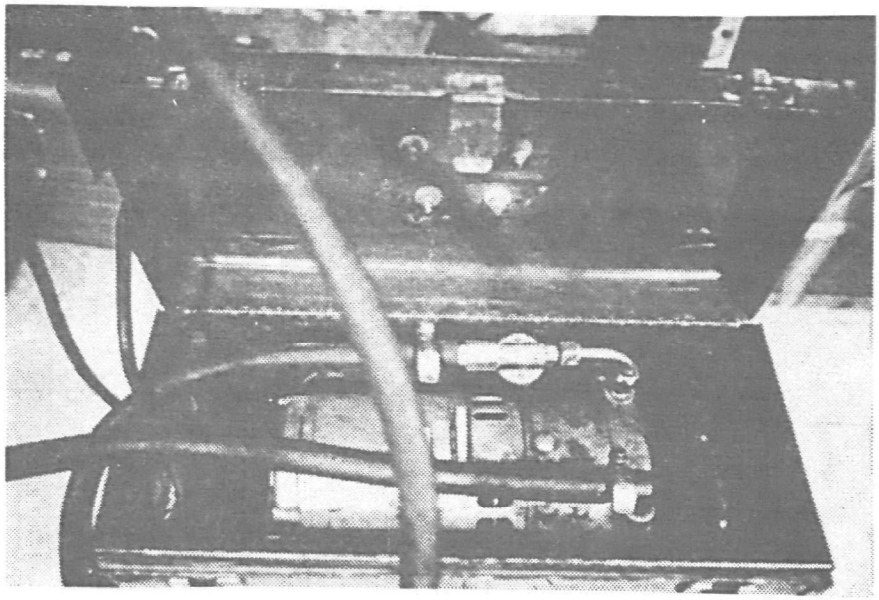

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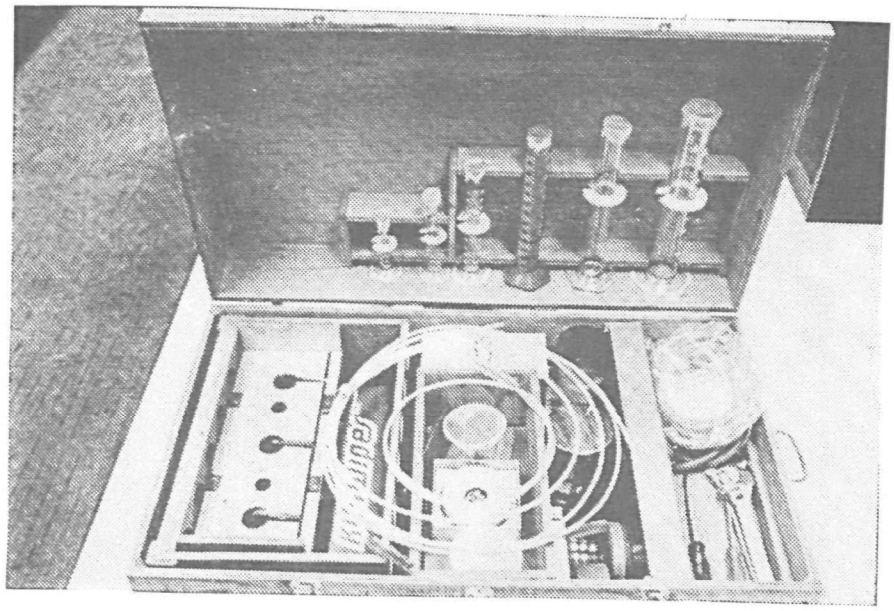

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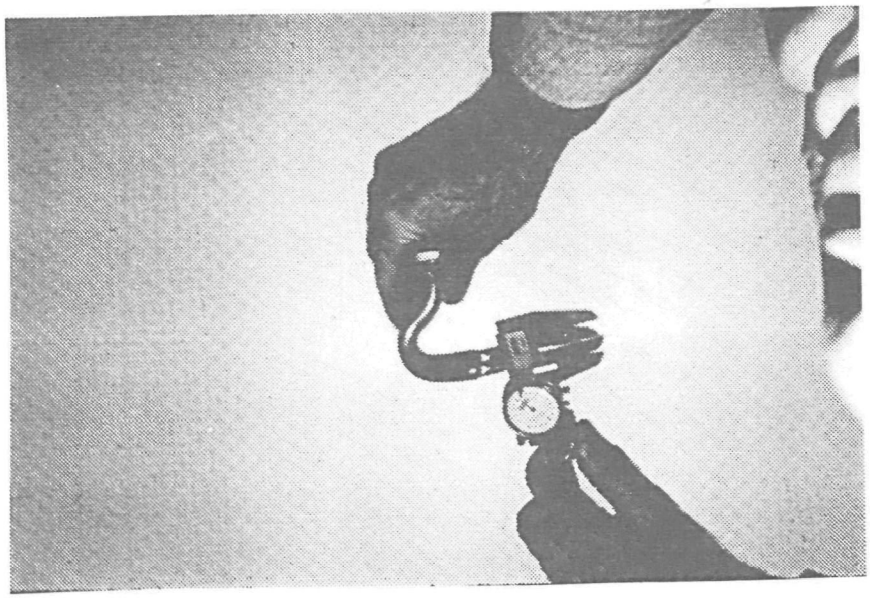
NOTES

COMPONENT CALIBRATION

PROBE NOZZLE

1. Measure three diameters of the nozzle.
2. Calculate the average measurement.
3. The difference between the high and low measurement shall not exceed 0.004 in.
4. Nozzle should be uniquely identified.

SLIDE 106-9



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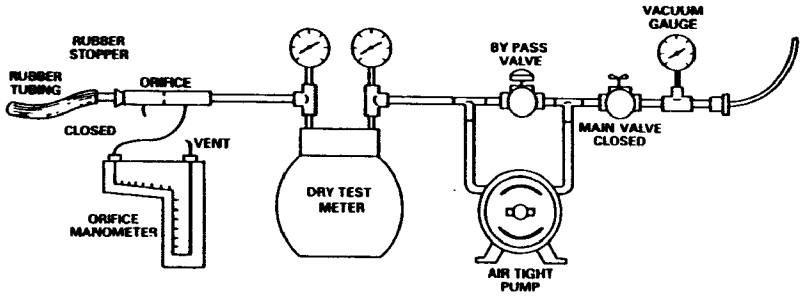
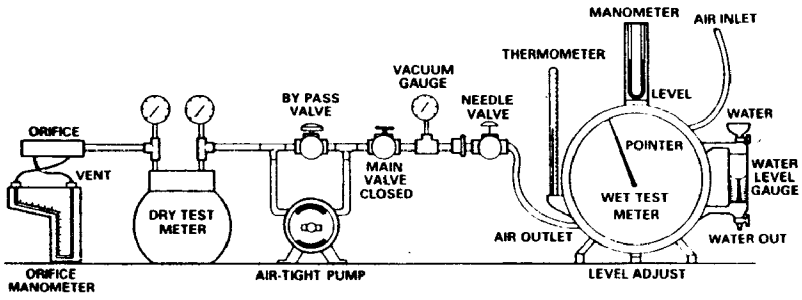
NOTES

METERING SYSTEM



SLIDE 106-11

SAMPLE METER SYSTEM CALIBRATION SETUP



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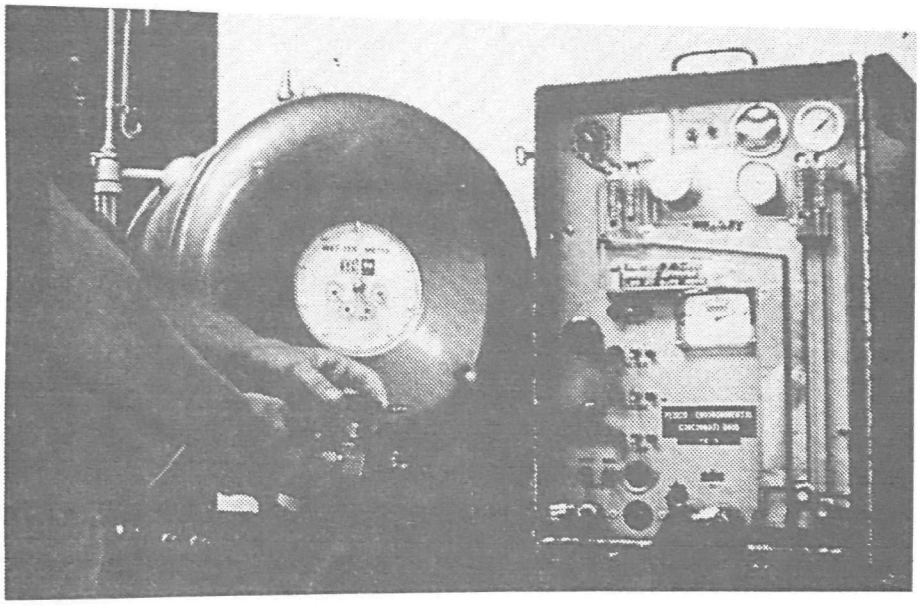


SLIDE 106- 13

TEMPERATURE GAUGE CALIBRATION

IMPINGER THERMOMETER

Calibrate with a mercury-in-glass thermometer which meets ASTM E-1 No. 63C or 63F specifications.

1. compare readings in ice bath
2. compare readings at room temperature
3. thermometer must agree within 2°F of the reference thermometer at both temperatures

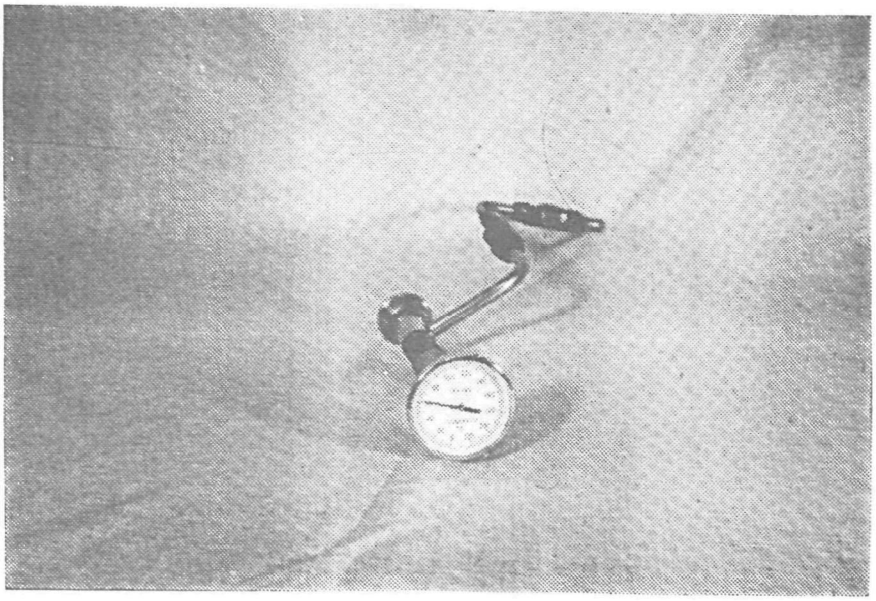
DRY GAS METER THERMOMETER

Calibrate with a mercury-in-glass thermometer which meets ASTM specifications.

1. compare readings in hot water bath 105° - 122°F
2. compare readings at room temperature
3. thermometers must agree within 5.4°F at both points or differential at both points within 5.4°F

SLIDE 106-14

NOTES



SLIDE 106-15

PROBE HEATER CALIBRATION

Calibrate probe heater if not constructed according to APTD - 0581 using procedure outlined in APTD - 0576

BALANCE CALIBRATION

ANALYTICAL BALANCE

- Calibrate using Class-S weights. (balance should agree within ± 2 mg)

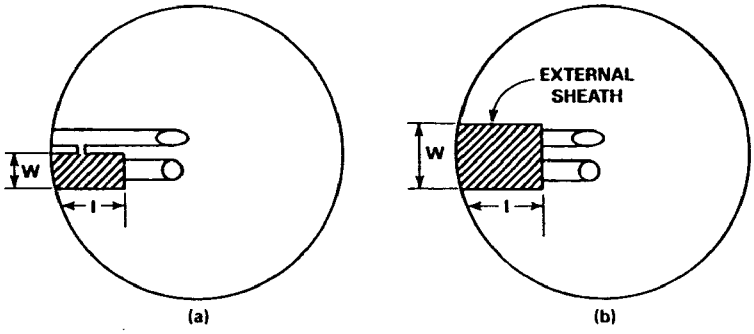
TRIP BALANCE

- Calibrate using Class-S weights (balance should agree within ± 0.5 g)

SLIDE 106-16

NOTES

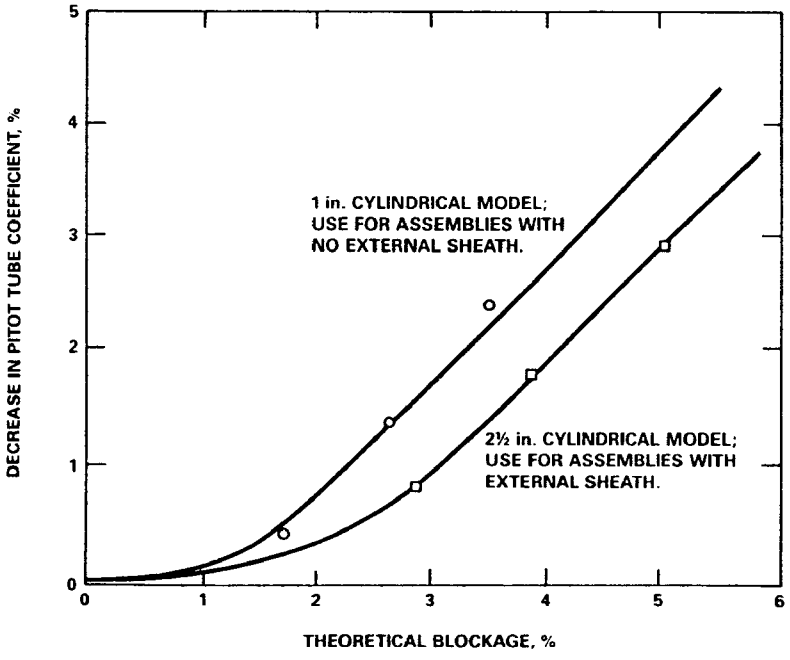
PROJECTED AREA MODELS FOR TYPICAL PITOBE ASSEMBLIES



$$\text{PERCENT THEORETICAL BLOCKAGE} = \left[\frac{l \times w}{\text{DUCT AREA}}\right] X\ 100$$

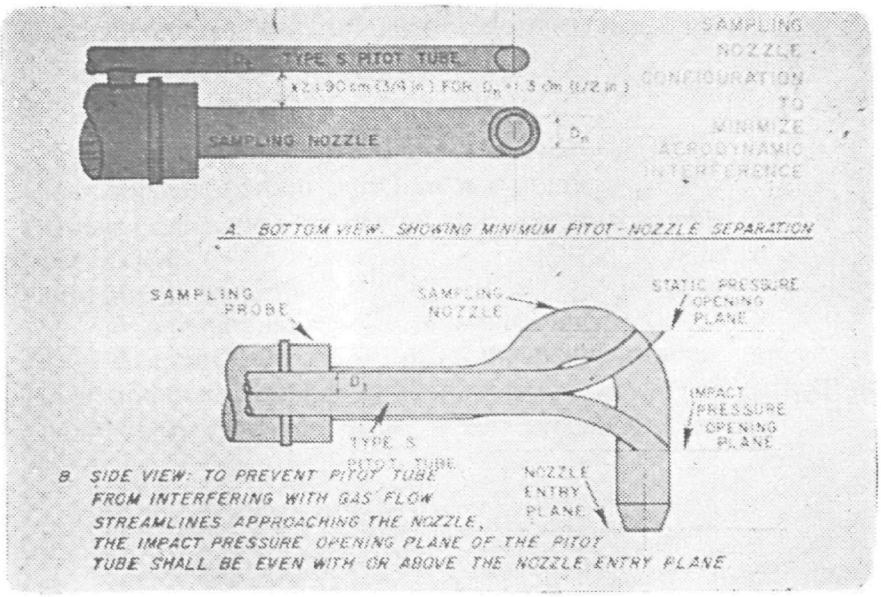
SLIDE 106-17

ADJUSTMENT OF TYPE "S" PITOT TUBE COEFFICIENTS TO ACCOUNT FOR BLOCKAGE



SLIDE 106-18

NOTES



SLIDE 106-19

ON SITE SAMPLING

1. Preliminary measurement and set up.
2. Collect stack parameters for setting the isokinetic sampling rate.
3. Set up nomograph and select proper nozzle size.
4. Prepare and assemble sampling train.
5. Leak-check sampling train.
6. Run the test.
7. Conduct post test leak-check.

SLIDE 106-20

NOTES

SAMPLE RECOVERY

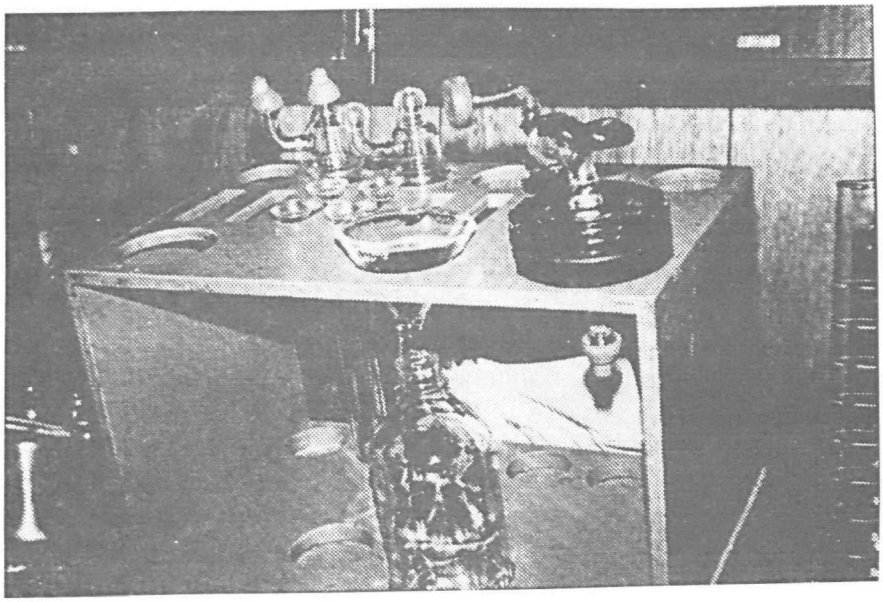
FILTER

1. Carefully remove filter from filter holder and place it in designated petri dish.
2. Seal and label petri dish.

PROBE AND CONNECTING GLASSWARE

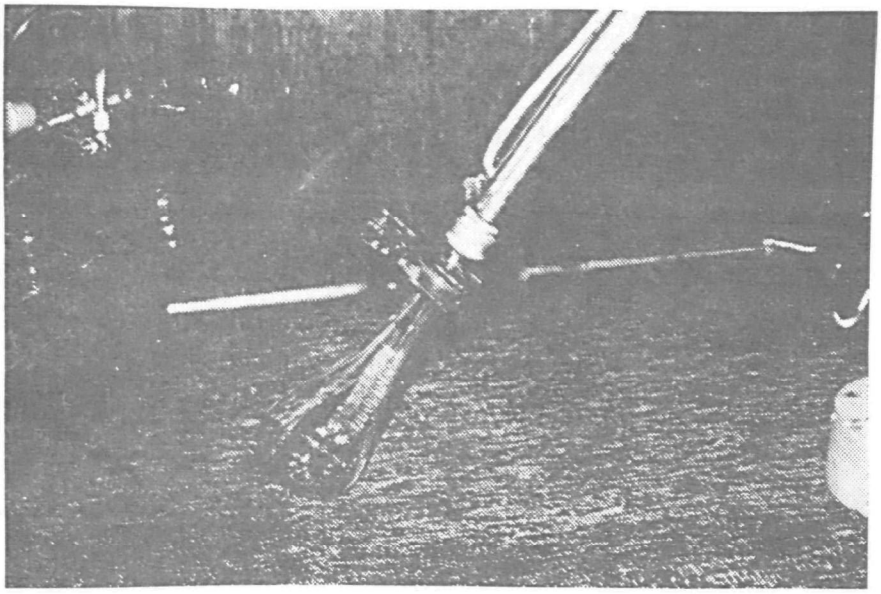
1. Clean outside of probe, pitot tube and nozzle.
2. Remove nozzle and rinse and brush inside surface until rinse is clear.
3. Rinse and brush probe.
   A. a minimum of 3 rinses for glass lined probes
   B. a minimum of 6 rinses for metal lined probes
4. Brush and rinse front half of filter holder.
5. Clean connecting glassware which precedes filter holder.

SLIDE 106-21



SLIDE 106-22

NOTES



SLIDE 106-23

PROBE AND CONNECTING GLASSWARE (cont.)

6. Securely seal sample bottle and mark liquid level.
7. Collect acetone blank sample.

CONDENSER

1. Determine liquid quantity in impingers to nearest 1 ml or 0.5 g.
2. Make note of any color or film in impinger water.

SILICA GEL

1. Note color of silica gel to determine if spent.
2. Determine weight gain to nearest 0.5 g.

SLIDE 106- 24

NOTES

ANALYSIS

FILTER

1. Desiccate filter for a minimum of 24 hrs.
2. Weigh to a constant weight and record results to nearest 0.1 mg.
3. Alternatively, filter may be oven dried at 220°F for 2 to 3 hrs., cooled in desiccator, and weighed to constant weight.
4. Analyze blank filter in same manner as sample filter.

SLIDE 106-25

(cont.) ANALYSIS

ACETONE RINSE

1. Confirm that no leakage occurred during shipment.
2. Measure contents of container to nearest 1 ml or 0.5 g.
3. Transfer to tared beaker; evaporate and desiccate for 24 hrs.
4. Weigh to constant weight; report data to nearest 0.1 mg.
5. Alternatively, acetone rinse may be evaporated at elevated temperature (below 133°F).
6. Analyze blank in same manner as the sample.

SECTION G. METHOD 6

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

1. Principle and Applicability

1.1. Principle. A gas sample is extracted from the sampling point in the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated. The sulfur dioxide fraction is measured by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfur dioxide emissions from stationary sources. The minimum detectable limit of the method has been determined to be 3.4 milligrams (mg) of SO_2/m^3 (2.12×10^{-7} lb/ft^3). Although no upper limit has been established, tests have shown that concentrations as high as 80,000 mg/m^3 of SO_2 can be collected efficiently in two midget impingers, each containing 15 milliliters of 3 percent hydrogen peroxide, at a rate of 1.0 lpm for 20 minutes. Based on theoretical calculations, the upper concentration limit in a 20-liter sample is about 93,300 mg/m^3.

Possible interferents are free ammonia, water-soluble cations, and fluorides. The cations and fluorides are removed by glass wool filters and an isopropanol bubbler, and hence do not affect the SO_2 analysis. When samples are being taken from a gas stream with high concentrations of very fine metallic fumes (such as in inlets to control devices), a high-efficiency glass fiber filter must be used in place of the glass wool plug (i.e., the one in the probe) to remove the cation interferents.

Free ammonia interferes by reacting with SO_2 to form particulate sulfite and by reacting with the indicator. If free ammonia is

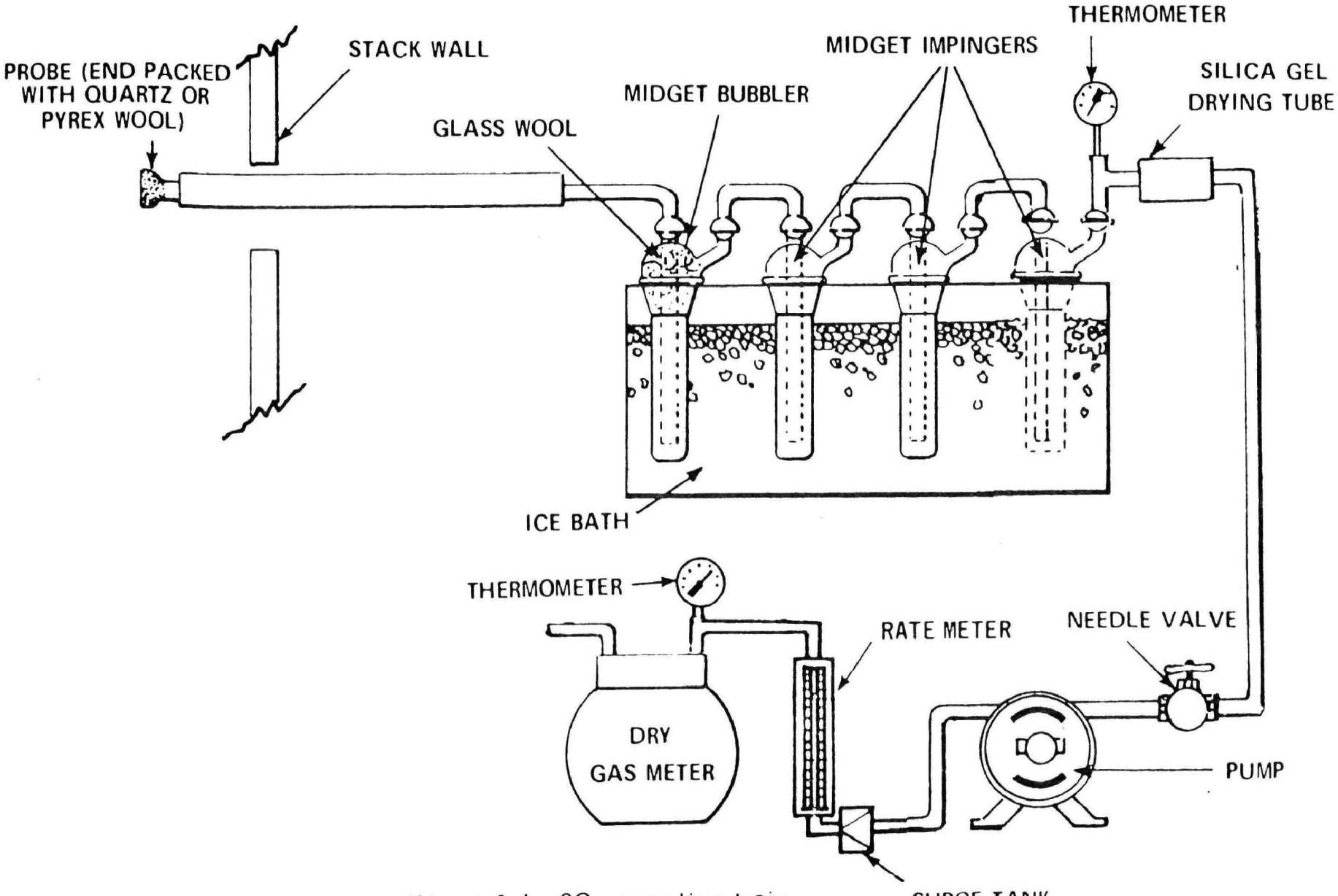


Figure 6-1. SO_2 sampling train.

present (this can be determined by knowledge of the process and noticing white particulate matter in the probe and isopropanol bubbler), alternative methods, subject to the approval of the Administrator, U. S. Environmental Protection Agency, are required.

2. Apparatus

2.1 Sampling. The sampling train is shown in Figure 6-1, and component parts are discussed below. The tester has the option of substituting sampling equipment described in Method 8 in place of the midget impinger equipment of Method 6. However, the Method 8 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 the flow rates and solution volumes defined in Method 8.

The tester also has the option of determining SO_2 simultaneously with particulate matter and moisture determinations by (1) replacing the water in a Method 5 impinger system with 3 percent peroxide solution, or (2) by replacing the Method 5 water impinger system with a Method 8 isopropanol-filter-peroxide system. The analysis for SO_2 must be consistent with the procedure in Method 8.

2.1.1 Probe. Borosilicate glass, or stainless steel (other materials of construction may be used, subject to the approval of the Administrator), approximately 6-mm inside diameter, with a heating system to prevent water condensation and a filter (either in-stack or heated out-stack) to remove particulate matter, including sulfuric acid mist. A plug of glass wool is a satisfactory filter.

2.1.2 Bubbler and Impingers. One midget bubbler, with medium-coarse glass frit and borosilicate or quartz glass wool

packed in top (see Figure 6-1) to prevent sulfuric acid mist carryover, and three 30-ml midget impingers. The bubbler and midget impingers must be connected in series with leak-free glass connectors. Silicone grease may be used, if necessary, to prevent leakage.

At the option of the tester, a midget impinger may be used in place of the midget bubbler.

Other collection absorbers and flow rates may be used, but are subject to the approval of the Administrator. Also, collection efficiency must be shown to be at least 99 percent for each test run and must be documented in the report. If the efficiency is found to be acceptable after a series of three tests, further documentation is not required. To conduct the efficiency test, an extra absorber must be added and analyzed separately. This extra absorber must not contain more than 1 percent of the total SO_2.

2.1.3 Glass Wool. Borosilicate or quartz.

2.1.4 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease may be used, if necessary.

2.1.5 Temperature Gauge. Dial thermometer, or equivalent, to measure temperature of gas leaving impinger train to within 1°C (2°F).

2.1.6 Drying Tube. Tube packed with 6- to 16-mesh indicating-type silica gel, or equivalent, to dry the gas sample and to protect the meter and pump. If the silica gel has been used previously, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to approval of the Administrator.

2.1.7 Valve. Needle valve, to regulate sample gas flow rate.

2.1.8 Pump. Leak-free diaphragm pump, or equivalent, to pull gas through the train. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.1.9 Rate Meter. Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of about 1000 cc/min.

2.1.10 Volume Meter. Dry gas meter, sufficiently accurate to measure the sample volume within 2 percent, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature gauge (dial thermometer, or equivalent) capable of measuring temperature to within 3°C (5.4°F).

2.1.11 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.12 Vacuum Gauge and Rotameter. At least 760 mm Hg (30 in. Hg) gauge and 0-40 cc/min rotameter, to be used for leak check of the sampling train.

2.2 Sample Recovery.

2.2.1 Wash Bottles. Polyethylene or glass, 500 ml, two.

2.2.2 Storage Bottles. Polyethylene, 100 ml, to store impinger samples (one per sample).

2.3 Analysis.

2.3.1 Pipettes. Volumetric type, 5-ml, 20-ml (one per sample), and 25-ml sizes.

2.3.2 Volumetric Flasks. 100-ml size (one per sample) and 1000-ml size.

2.3.3 Burettes. 5- and 50-ml sizes.

2.3.4 Erlenmeyer Flasks. 250 ml-size (one for each sample, blank, and standard).

2.3.5 Dropping Bottle. 125-ml size, to add indicator.

2.3.6 Graduated cylinder. 100-ml size.

2.3.7 Spectrophotometer. To measure absorbance at 352 nanometers.

3. Reagents

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

3.1 Sampling.

3.1.1 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst,

the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.1.2 Isopropanol, 80 percent. Mix 80 ml of isopropanol with 20 ml of deionized, distilled water. Check each lot of isopropanol for peroxide impurities as follows: shake 10 ml of isopropanol with 10 ml of freshly prepared 10 percent potassium iodide solution. Prepare a blank by similarly treating 10 ml of distilled water. After 1 minute, read the absorbance at 352 nanometers on a spectrophotometer (Note: Use a 1-cm path length). If absorbance exceeds 0.1, reject alcohol for use.

Peroxides may be removed from isopropanol by redistilling or by passage through a column of activated alumina; however, reagent grade isopropanol with suitably low peroxide levels may be obtained from commercial sources. Rejection of contaminated lots may, therefore, be a more efficient procedure.

3.1.3 Hydrogen Peroxide, 3 Percent. Dilute 30 percent hydrogen peroxide 1:9 (v/v) with deionized, distilled water (30 ml is needed per sample). Prepare fresh daily.

3.1.4 Potassium Iodide Solution, 10 Percent. Dissolve 10.0 grams KI in deionized, distilled water and dilute to 100 ml. Prepare when needed.

3.2 Sample Recovery.

3.2.1 Water. Deionized, distilled, as in 3.1.1.

3.2.2 Isopropanol, 80 Percent. Mix 80 ml of isopropanol with 20 ml of deionized, distilled water.

3.3 Analysis.

3.3.1 Water. Deionized, distilled, as in 3.1.1.

3.3.2 Isopropanol, 100 percent.

3.3.3 Thorin Indicator. 1-(o-arsonophenylazo)-2-naphthol-3, 6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 ml of deionized, distilled water.

3.3.4 Barium Perchlorate Solution, 0.0100 N. Dissolve 1.95 g of barium perchlorate trihydrate [$Ba(ClO_4)_2 \cdot 3H_2O$] in 200 ml distilled water and dilute to 1 liter with isopropanol. Alternatively, 1.22 g of [$BaCl_2 \cdot 2H_2O$] may be used instead of the perchlorate. Standardize as in Section 5.5.

3.3.5 Sulfuric Acid Standard, 0.0100 N. Purchase or standardize to $\pm$0.0002 N against 0.0100 N NaOH which has previously been standardized against potassium acid phthalate (primary standard grade).

4. Procedure.

4.1 Sampling.

4.1.1 Preparation of collection train. Measure 15 ml of 80 percent isopropanol into the midget bubbler and 15 ml of 3 percent hydrogen peroxide into each of the first two midget impingers. Leave the final midget impinger dry. Assemble the train as shown in Figure 6-1. Adjust probe heater to a temperature sufficient to prevent water condensation. Place crushed ice and water around the impingers.

4.1.2 Leak-check procedure. A leak check prior to the sampling run is optional; however, a leak check after the sampling run is mandatory. The leak-check procedure is as follows:

Temporarily attach a suitable (e.g., 0-40 cc/min) rotameter to the outlet of the dry gas meter and place a vacuum gauge at or near the probe inlet. Plug the probe inlet, pull a vacuum of at least 250 mm Hg (10 in. Hg), and note the flow rate as indicated by the rotameter. A leakage rate not in excess of 2 percent of the average sampling rate is acceptable. Note: Carefully release the probe inlet plug before turning off the pump.

It is suggested (not mandatory) that the pump be leak-checked separately, either prior to or after the sampling run. If done prior to the sampling run, the pump leak-check shall precede the leak check of the sampling train described immediately above; if done after the sampling run, the pump leak-check shall follow the train leak-check. To leak check the pump, proceed as follows: Disconnect the drying tube from the probe-impinger assembly. Place a vacuum gauge at the inlet to either the drying tube or the pump, pull a vacuum of 250 mm (10 in.) Hg, plug or pinch off the outlet of the flow meter and then turn off the pump. The vacuum should remain stable for at least 30 seconds.

Other leak-check procedures may be used, subject to the approval of the Administrator, U.S. Environmental Protection Agency. The procedure used in Method 5 is not suitable for diaphragm pumps.

4.1.3 Sample Collection. Record the initial dry gas meter reading and barometric pressure. To begin sampling, position the tip of the probe at the sampling point, connect the probe to the bubbler, and start the pump. Adjust the sample flow to a constant rate of approximately 1.0 liter/min as indicated by the rotameter. Maintain this constant rate (± 10 percent) during the entire sampling run. Take readings (dry gas meter, temperatures at dry gas meter and at impinger outlet and rate meter) at least every 5 minutes. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20°C (68°F) or less. At the conclusion of each run, turn off the pump, remove probe from the stack, and record the final readings. Conduct a leak check as in Section 4.1.2. (This leak check is mandatory.) If a leak is found, void the test run or use procedures acceptable to the Administrator to adjust the sample volume for leakage. Drain the ice bath, and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes at the sampling rate.

Clean ambient air can be provided by passing air through a charcoal filter or through an extra midget impinger with 15 ml of 3 percent H_2O_2. The tester may opt to simply use ambient air, without purification.

4.2 Sample Recovery. Disconnect the impingers after purging. Discard the contents of the midget bubbler. Pour the contents of the midget impingers into a leak-free polyethylene bottle for shipment. Rinse the three midget impingers and the connecting tubes with deionized, distilled water, and add the washings to the same storage container. Mark the fluid level. Seal and identify the sample container.

4.3 Sample Analysis. Note level of liquid in container, and confirm whether any sample was lost during shipment; note this on analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

Transfer the contents of the storage container to a 100-ml volumetric flask and dilute to exactly 100 ml with deionized, distilled water. Pipette a 20-ml aliquot of this solution into a 250-ml Erlenmeyer flask, add 80 ml of 100 percent isopropanol and two to four drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat and average the titration volumes. Run a blank with each series of samples. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is larger.

(Note: Protect the 0.0100 N barium perchlorate solution from evaporation at all times.)

5. Calibration

5.1 Metering System.

5.1.1 Initial Calibration. Before its initial use in the field, first leak check the metering system (drying tube, needle valve, pump, rotameter, and dry gas meter) as follows: place a vacuum gauge at the inlet to the drying tube and pull a vacuum of 250 mm (10 in.) Hg; plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum shall remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter end.

Next, calibrate the metering system (at the sampling flow rate specified by the method) as follows: connect an appropriately sized wet test meter (e.g., 1 liter per revolution) to the inlet of the drying tube. Make three independent calibration runs, using at least five revolutions of the dry gas meter per run. Calculate the calibration factor, Y (wet test meter calibration volume divided by the dry gas meter volume, both volumes adjusted to the same reference temperature and pressure), for each run, and average the results. If any Y value deviates by more than 2 percent from the average, the metering system is unacceptable for use. Otherwise, use the average as the calibration factor for subsequent test runs.

5.1.2 Post-Test Calibration Check. After each field test series, conduct a calibration check as in Section 5.1.1 above, except for the following variations: (a) the leak check is not to be conducted, (b) three, or more revolutions of the dry gas meter may be used, and (c) only two independent runs need be made. If the calibration factor does

not deviate by more than 5 percent from the initial calibration factor (determined in Section 5.1.1), then the dry gas meter volumes obtained during the test series are acceptable. If the calibration factor deviates by more than 5 percent, recalibrate the metering system as in Section 5.1.1, and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

5.2 Thermometers. Calibrate against mercury-in-glass thermometers.

5.3 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

5.4 Barometer. Calibrate against a mercury barometer.

5.5 Barium Perchlorate Solution. Standardize the barium perchlorate solution against 25 ml of standard sulfuric acid to which 100 ml of 100 percent isopropanol has been added.

6. Calculations

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

6.1 Nomenclature.

C_{SO_2} = Concentration of sulfur dioxide, dry basis corrected to standard conditions, mg/dscm (lb/dscf).

N = Normality of barium perchlorate titrant, milliequivalents/ml.

P_{bar} = Barometric pressure at the exit orifice of the dry gas meter, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

T_m = Average dry gas meter absolute temperature, °K (°R).

T_{std} = Standard absolute temperature, 293° K (528° R).

V_a = Volume of sample aliquot titrated, ml.

V_m = Dry gas volume as measured by the dry gas meter, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

V_{soln} = Total volume of solution in which the sulfur dioxide sample is contained, 100 ml.

V_t = Volume of barium perchlorate titrant used for the sample, ml (average of replicate titrations).

V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.

Y = Dry gas meter calibration factor.

32.03 = Equivalent weight of sulfur dioxide.

6.2 Dry sample gas volume, corrected to standard conditions.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right) \left(\frac{P_{bar}}{P_{std}}\right) = K_1 Y \frac{V_m P_{bar}}{T_m}$$

Equation 6-1

where:

K_1 = 0.3858 °K/mm Hg for metric units.

= 17.64 °R/in. Hg for English units.

6.3 Sulfur dioxide concentration.

$$C_{SO_2} = K_2 \frac{(V_t - V_{tb})N\left(\frac{V_{soln}}{V_a}\right)}{V_{m(std)}}$$

Equation 6-2

where:

K_2 = 32.03 mg/meq. for metric units.

= 7.061×10^{-5} lb/meq. for English units.

7. Bibliography

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SLIDE 107-0

NOTES

METHOD — 6

Determination of
Sulfur Dioxide Emissions
From Stationary Sources

SLIDE 107-1

PRINCIPLE

A gas sample is extracted from the stack. Sulfuric acid mist and sulfur dioxide are separated.

Sulfur dioxide is measured by the barium-thorin titration method.

APPLICABILITY

This method is applicable for determination of sulfur dioxide emissions from stationary sources.

- minimum detectable limit is 3.4 mg of SO_2 per m^3
- theoretical calculations indicate the upper concentration limit in a 20 liter sample is $\approx$ 93,300 mg per m^3.

SLIDE 107-2

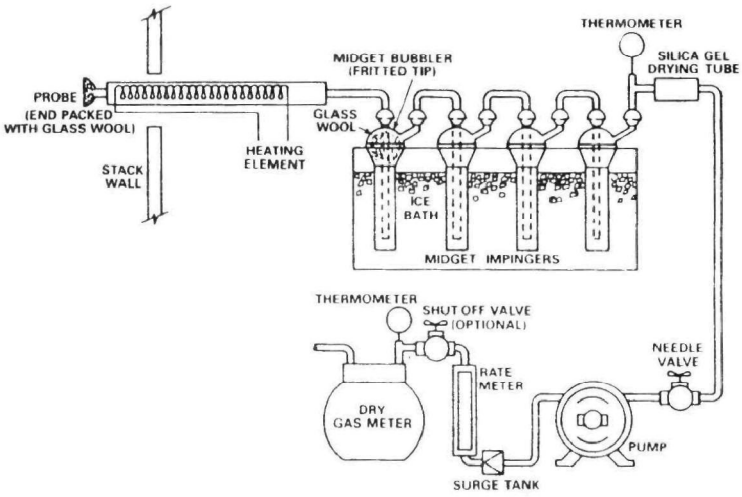
METHOD 6 INTERFERENCES

1. CATIONS — Removed by glass wool filter and isopropanol bubbler.
2. FLUORIDES — Removed by glass wool filter and isopropanol bubbler.
3. FINE METALLIC FUMES — Removed by high efficiency glass fiber filter.
4. AMMONIA — Use alternative methods (Subject to Approval of the Administrator).

SLIDE 107-3

NOTES

SO_2 SAMPLING TRAIN



SLIDE 107-4

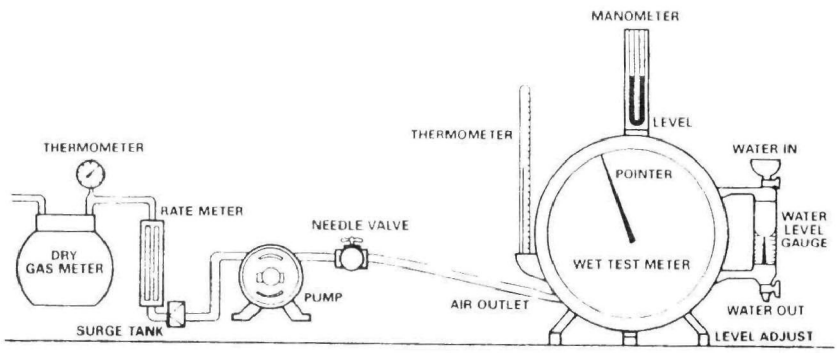
REAGENTS

Reagents must conform to specifications established by American Chemical Society or use best available grade.

- Check each lot of isopropanol for peroxide impurities.
- Prepare 3% hydorgen peroxide fresh each day.
- Standardize barium perchlorate solution against standard sulfuric acid to which 100 ml of 100% isopropanol has been added.
- Standardize sulfuric acid 0.0100 N against 0.0100 N sodium hydroxide.

SLIDE 107-5

SAMPLE METER SYSTEM CALIBRATION SETUP



SLIDE 107-6

NOTES

THERMOMETER CALIBRATION

- Impinger thermometer should agree to within 2°F of the standard at both points.
- Dry gas meter thermometer should agree to within 5.4°F of the standard at both points.

ROTAMETER

- calibration not required
- maintain according to manufacturer's instructions

BAROMETER

- calibrate according to procedures in Method 2

SLIDE 107-7

ON SITE SAMPLING

1. Prepare collection train.
2. Conduct pretest leak-check (optional).
3. Run the test.
4. Conduct post test leak-check (mandatory).
5. Purge sample train for 15 minutes.

SLIDE 107-8

SAMPLE RECOVERY

1. Disconnect impingers and discard contents of the bubbler.
2. Recover contents of the impingers into a leak-free polyethylene bottle.
3. Rinse impingers and connecting tubes with distilled water; add washings to sample bottle.
4. Mark liquid level, seal, and identify the container.

SLIDE 107-9

NOTES

SAMPLE ANALYSIS

1. Check liquid level of the sample.
2. Transfer sample to a 100 ml volumetric flask and dilute to 100 ml with deionized distilled water.
3. Pipette a 20 ml aliquot into a 250 ml erlenmeyer flask.
4. Add 80 ml of 100% isopropanol and two to four drops of thorin indicator.
5. Titrate to a pink endpoint using 0.0100 N barium perchlorate.
6. Repeat and average titration volumes.
7. Run a blank with each series of samples.

SECTION H. METHOD 7

METHOD 7--DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxide, are measured colorimetrically using the phenoldisulfonic acid (PDS) procedure.

1.2 Applicability. This method is applicable to the measurement of nitrogen oxides emitted from stationary sources. The range of the method has been determined to be 2 to 400 milligrams NO_x (as NO_2) per dry standard cubic meter, without having to dilute the sample.

2. Apparatus

2.1 Sampling (see Figure 7-1). Other grab sampling systems or equipment, capable of measuring sample volume to within ±2.0 percent and collecting a sufficient sample volume to allow analytical reproducibility to within ±5 percent, will be considered acceptable alternatives, subject to approval of the Administrator, U. S. Environmental Protection Agency. The following equipment is used in sampling:

2.1.1 Probe. Borosilicate glass tubing, sufficiently heated to prevent water condensation and equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Stainless steel or Teflon[1] tubing may also be used for the probe. Heating is not necessary if the probe remains dry during the purging period.

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[1]Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

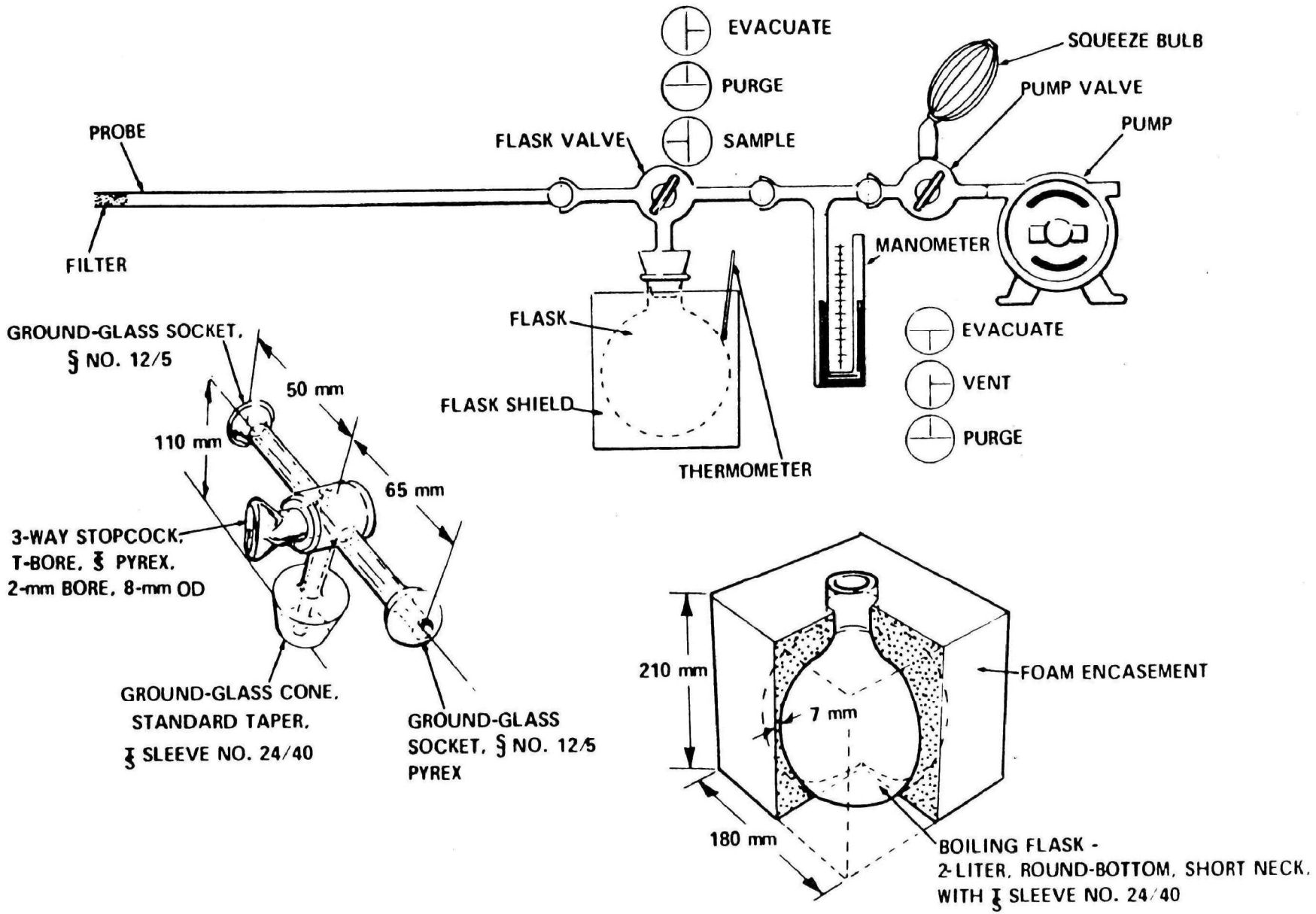

Figure 7-1. Sampling train, flask valve, and flask.

2.1.2 Collection Flask. Two-liter borosilicate, round bottom flask, with short neck and 24/40 standard taper opening, protected against implosion or breakage.

2.1.3 Flask Valve. T-bore stopcock connected to a 24/40 standard taper joint.

2.1.4 Temperature Gauge. Dial-type thermometer, or other temperature gauge, capable of measuring 1°C (2°F) intervals from -5 to 50°C (25 to 125°F).

2.1.5 Vacuum Line. Tubing capable of withstanding a vacuum of 75 mm Hg (3 in. Hg) absolute pressure, with "T" connection and T-bore stopcock.

2.1.6 Vacuum Gauge. U-tube manometer, 1 meter (36 in.), with 1-mm (0.1-in.) divisions, or other gauge capable of measuring pressure to within $\pm$2.5 mm Hg (0.10 in. Hg).

2.1.7 Pump. Capable of evacuating the collection flask to a pressure equal to or less than 75 mm Hg (3 in. Hg) absolute.

2.1.8 Squeeze Bulb. One-way.

2.1.9 Volumetric Pipette. 25 ml.

2.1.10 Stopcock and Ground Joint Grease. A high-vacuum, high-temperature chlorofluorocarbon grease is required. Halocarbon 25-5S has been found to be effective.

2.1.11 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value

(which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase, or vice versa for elevation decrease.

2.2 Sample Recovery. The following equipment is required for sample recovery:

2.2.1 Graduated Cylinder. 50 ml with 1-ml divisions.

2.2.2 Storage Containers. Leak-free polyethylene bottles.

2.2.3 Wash Bottle. Polyethylene or glass.

2.2.4 Glass Stirring Rod.

2.2.5 Test Paper for Indicating pH. To cover the pH range of 7 to 14.

2.3 Analysis. For the analysis, the following equipment is needed:

2.3.1 Volumetric Pipettes. Two 1 ml, two 2 ml, one 3 ml, one 4 ml, two 10 ml, and one 25 ml for each sample and standard.

2.3.2 Porcelain Evaporating Dishes. 175- to 250-ml capacity with lip for pouring, one for each sample and each standard. The Coors No. 45006 (shallow-form, 195 ml) has been found to be satisfactory. Alternatively, polymethyl pentene beakers (Nalge No. 1203, 150 ml), or glass beakers (150 ml) may be used. When glass beakers are used, etching of the beakers may cause solid matter to be present in the analytical step; the solids should be removed by filtration (see Section 4.3).

2.3.3 Steam Bath. Low-temperature ovens or thermostatically controlled hot plates kept below 70°C (160°F) are acceptable alternatives.

2.3.4 Dropping Pipette or Dropper. Three required.

2.3.5 Polyethylene Policeman. One for each sample and each standard.

2.3.6 Graduated Cylinder. 100 ml with 1-ml divisions.

2.3.7 Volumetric Flasks. 50 ml (one for each sample and each standard), 100 ml (one for each sample and each standard, and one for the working standard KNO_3 solution), and 1000 ml (one).

2.3.8 Spectrophotometer. To measure absorbance at 410 nm.

2.3.9 Graduated Pipette. 10 ml with 0.1-ml divisions.

2.3.10 Test Paper for Indicating pH. To cover the pH range of 7 to 14.

2.3.11 Analytical Balance. To measure to within 0.1 mg.

3. Reagents

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

3.1 Sampling. To prepare the absorbing solution, cautiously add 2.8 ml concentrated H_2SO_4 to 1 liter of deionized, distilled water. Mix well and add 6 ml of 3 percent hydrogen peroxide, freshly prepared from 30 percent hydrogen peroxide solution. The absorbing solution should be used within 1 week of its preparation. Do not expose to extreme heat or direct sunlight.

3.2 Sample Recovery. Two reagents are required for sample recovery:

3.2.1 Sodium Hydroxide (1 N). Dissolve 40 g NaOH in deionized, distilled water and dilute to 1 liter.

3.2.2 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the $KMNO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.3 Analysis. For the analysis, the following reagents are required:

3.3.1 Fuming Sulfuric Acid. 15 to 18 percent by weight free sulfur trioxide. HANDLE WITH CAUTION.

3.3.2 Phenol. White solid.

3.3.3 Sulfuric Acid. Concentrated, 95 percent minimum assay. HANDLE WITH CAUTION.

3.3.4 Potassium Nitrate. Dried at 105 to 110°C (220 to 230°F) for a minimum of 2 hours just prior to preparation of standard solution.

3.3.5 Standard KNO_3 Solution. Dissolve exactly 2.198 g of dried potassium nitrate (KNO_3) in deionized, distilled water and dilute to 1 liter with deionized, distilled water in a 1000-ml volumetric flask.

3.3.6 Working Standard KNO_3 Solution. Dilute 10 ml of the standard solution to 100 ml with deionized distilled water. One milliliter of the working standard solution is equivalent to 100 µg nitrogen dioxide (NO_2).

3.3.7 Water. Deionized, distilled as in Section 3.2.2.

3.3.8 Phenoldisulfonic Acid Solution. Dissolve 25 g of pure white phenol in 150 ml concentrated sulfuric acid on a steam bath. Cool, add 75 ml fuming sulfuric acid, and heat at 100°C (212°F) for 2 hours. Store in a dark, stoppered bottle.

4. Procedures

4.1 Sampling.

4.1.1 Pipette 25 ml of absorbing solution into a sample flask, retaining a sufficient quantity for use in preparing the calibration standards. Insert the flask valve stopper into the flask with the valve in the "purge" position. Assemble the sampling train as shown in Figure 7-1 and place the probe at the sampling point. Make sure that all fittings are tight and leak-free, and that all ground glass joints have been properly greased with a high-vacuum, high-temperature chlorofluorocarbon-based stopcock grease. Turn the flask valve and the pump valve to their "evacuate" positions. Evacuate the flask to 75 mm Hg (3 in. Hg) absolute pressure, or less. Evacuation to a pressure approaching the vapor pressure of water at the existing temperature is desirable. Turn the pump valve to its "vent" position and turn off the pump. Check for leakage by observing the manometer for any pressure fluctuation. (Any variation greater than 10 mm Hg (0.4 in. Hg) over a period of 1 minute is not acceptable, and the flask is not to be used until the leakage problem is corrected. Pressure in the flask is not to exceed 75 mm Hg (3 in. Hg) absolute at the time sampling is commenced.) Record the volume of the flask and valve (V_f), the flask temperature (T_i), and the barometric pressure. Turn the flask valve counterclockwise to its "purge" position

and do the same with the pump valve. Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and the flask valve area, heat the probe and purge until the condensation disappears. Next, turn the pump valve to its "vent" position. Turn the flask valve clockwise to its "evacuate" position and record the difference in the mercury levels in the manometer. The absolute internal pressure in the flask (P_i) is equal to the barometric pressure less the manometer reading. Immediately turn the flask valve to the "sample" position and permit the gas to enter the flask until pressures in the flask and sample line (i.e., duct, stack) are equal. This will usually require about 15 seconds; a longer period indicates a "plug" in the probe, which must be corrected before sampling is continued. After collecting the sample, turn the flask valve to its "purge" position and disconnect the flask from the sampling train. Shake the flask for at least 5 minutes.

4.1.2 If the gas being sampled contains insufficient oxygen for the conversion of NO to NO_2 (e.g., an applicable subpart of the standard may require taking a sample of a calibration gas mixture of NO in N_2), then oxygen shall be introduced into the flask to permit this conversion. Oxygen may be introduced into the flask by one of three methods: (1) Before evacuating the sampling flask, flush with pure cylinder oxygen, then evacuate flask to 75 mm Hg (3 in. Hg) absolute pressure or less; or (2) inject oxygen into the flask after sampling; or (3) terminate sampling with a minimum of 50 mm Hg (2 in. Hg) vacuum remaining in the flask, record this final pressure, and then vent the flask to the atmosphere until the flask pressure is almost equal to atmospheric pressure.

4.2 Sample Recovery. Let the flask set for a minimum of 16 hours and then shake the contents for 2 minutes. Connect the flask to a mercury filled U-tube manometer. Open the valve from the flask to the manometer and record the flask temperature (T_f), the barometric pressure, and the difference between the mercury levels in the manometer. The absolute internal pressure in the flask (P_f) is the barometric pressure less the manometer reading. Transfer the contents of the flask to a leak-free polyethylene bottle. Rinse the flask twice with 5-ml portions of deionized, distilled water and add the rinse water to the bottle. Adjust the pH to between 9 and 12 by adding sodium hydroxide (1 N), dropwise (about 25 to 35 drops). Check the pH by dipping a stirring rod into the solution and then touching the rod to the pH test paper. Remove as little material as possible during this step. Mark the height of the liquid level so that the container can be checked for leakage after transport. Label the container to clearly identify its contents. Seal the container for shipping.

4.3 Analysis. Note the level of the liquid in container and confirm whether or not any sample was lost during shipment; note this on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Immediately prior to analysis, transfer the contents of the shipping container to a 50-ml volumetric flask, and rinse the container twice with 5-ml portions of deionized, distilled water. Add the rinse water to the flask and dilute to the mark with deionized, distilled water; mix thoroughly. Pipette

a 25-ml aliquot into the porcelain evaporating dish. Return any unused portion of the sample to the polyethylene storage bottle. Evaporate the 25-ml aliquot to dryness on a steam bath and allow to cool. Add 2 ml phenoldisulfonic acid solution to the dried residue and triturate thoroughly with a polyethylene policeman. Make sure the solution contacts all the residue. Add 1 ml deionized, distilled water and four drops of concentrated sulfuric acid. Heat the solution on a steam bath for 3 minutes with occasional stirring. Allow the solution to cool, add 20 ml deionized, distilled water, mix well by stirring, and add concentrated ammonium hydroxide, dropwise, with constant stirring, until the pH is 10 (as determined by pH paper). If the sample contains solids, these must be removed by filtration (centrifugation is an acceptable alternative, subject to the approval of the Administrator), as follows: filter through Whatman No. 41 filter paper into a 100-ml volumetric flask; rinse the evaporating dish with three 5-ml portions of deionized, distilled water; filter these three rinses. Wash the filter with at least three 15-ml portions of deionized, distilled water. Add the filter washings to the contents of the volumetric flask and dilute to the mark with deionized, distilled water. If solids are absent, the solution can be transferred directly to the 100-ml volumetric flask and diluted to the mark with deionized, distilled water. Mix the contents of the flask thoroughly, and measure the absorbance at the optimum wavelength used for the standards (Section 5.2.1), using the blank solution as a zero reference. Dilute the sample and the blank with equal volumes of deionized, distilled water if the absorbance exceeds A_4, the absorbance of the 400 µg NO_2 standard (see Section 5.2.2).

5. Calibration

5.1 Flask Volume. The volume of the collection flask-flask valve combination must be known prior to sampling. Assemble the flask and flask valve and fill with water, to the stopcock. Measure the volume of water to ±10 ml. Record this volume on the flask.

5.2 Spectrophotometer Calibration.

5.2.1 Optimum Wavelength Determination. Calibrate the wavelength scale of the spectrophotometer every 6 months. The calibration may be accomplished by using an energy source with an intense line emission such as a mercury lamp, or by using a series of glass filters spanning the measuring range of the spectrophotometer. Calibration materials are available commercially and from the National Bureau of Standards. Specific details on the use of such materials should be supplied by the vendor; general information about calibration techniques can be obtained from general reference books on analytical chemistry. The wavelength scale of the spectrophotometer must read correctly within ±5 nm at all calibration points; otherwise, the spectrophotometer shall be repaired and recalibrated. Once the wavelength scale of the spectrophotometer is in proper calibration, use 410 nm as the optimum wavelength for the measurement of the absorbance of the standards and samples.

Alternatively, a scanning procedure may be employed to determine the proper measuring wavelength. If the instrument is a double-beam spectrophotometer, scan the spectrum between 400 and 415 nm using a 200 µg NO_2 standard solution in the sample cell and a blank solution in the reference cell. If a peak does not occur, the spectrophotometer is probably malfunctioning and should be repaired. When a peak is obtained within the 400 to 415 nm range, the wavelength at which this peak occurs shall be the optimum wavelength for the measurement of absorbance of both the standards and the samples. For a single-beam spectrophotometer, follow the scanning procedure described above, except that the blank and standard solutions shall be scanned separately. The optimum wavelength shall be the wavelength at which the maximum difference in absorbance between the standard and the blank occurs.

5.2.2 Determination of Spectrophotometer Calibration Factor K_c. Add 0.0 ml, 2.0 ml, 4.0 ml, 6.0 ml, and 8.0 ml of the KNO_3 working standard solution (1 ml = 100 µg NO_2) to a series of five 50-ml volumetric flasks. To each flask, add 25 ml of absorbing solution, 10 ml deionized, distilled water, and sodium hydroxide (1N) dropwise until the pH is between 9 and 12 (about 25 to 35 drops each). Dilute to the mark with deionized, distilled water. Mix thoroughly and pipette a 25-ml aliquot of each solution into a separate porcelain evaporating dish. Beginning with the evaporation step, follow the analysis procedure of Section 4.3, until the solution has been transferred to the 100-ml volumetric flask and diluted to the mark. Measure the absorbance of each solution, at the optimum wavelength, as determined in Section 5.2.1. This calibration procedure must be repeated

on each day that samples are analyzed. Calculate the spectrophotometer calibration factor as follows:

$$K_c = 100 \frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2} \qquad \text{Equation 7-1}$$

where:

K_c = Calibration factor

A_1 = Absorbance of the 100-μg NO_2 standard

A_2 = Absorbance of the 200-μg NO_2 standard

A_3 = Absorbance of the 300-μg NO_2 standard

A_4 = Absorbance of the 400-μg NO_2 standard

5.3 Barometer. Calibrate against a mercury barometer.

5.4 Temperature Gauge. Calibrate dial thermometers against mercury-in-glass thermometers.

5.5 Vacuum Gauge. Calibrate mechanical gauges, if used, against a mercury manometer such as that specified in 2.1.6.

5.6 Analytical Balance. Calibrate against standard weights.

6. Calculations

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

6.1 Nomenclature.

A = Absorbance of sample.

C = Concentration of NO_x as NO_2, dry basis, corrected to standard conditions, mg/dscm (lb/dscf).

F = Dilution factor (i.e., 25/5, 25/10, etc., required only if sample dilution was needed to reduce the absorbance into the range of calibration).

K_c = Spectrophotometer calibration factor.

m = Mass of NO_x as NO_2 in gas sample, µg.

P_f = Final absolute pressure of flask, mm Hg (in. Hg).

P_i = Initial absolute pressure of flask, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

T_f = Final absolute temperature of flask, °K (°R).

T_i = Initial absolute temperature of flask, °K (°R).

T_{std} = Standard absolute temperature, 293°K (528°R).

V_{sc} = Sample volume at standard conditions (dry basis), ml.

V_f = Volume of flask and valve, ml.

V_a = Volume of absorbing solution, 25 ml.

2 = 50/25, the aliquot factor. (If other than a 25-ml aliquot was used for analysis, the corresponding factor must be substituted).

6.2 Sample volume, dry basis, corrected to standard conditions.

$$V_{sc} = \frac{T_{std}}{P_{std}} (V_f - V_a) \left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right] = K_1 (V_f - 25\ ml) \left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right]$$

Equation 7-2

where:

$$K_1 = 0.3858 \frac{°K}{mm\ Hg} \text{ for metric units}$$

$$= 17.64 \frac{°R}{in.\ Hg} \text{ for English units}$$

6.3 Total μg NO_2 per sample.

$$m = 2K_c AF \qquad \text{Equation 7-3}$$

Note: If other than a 25-ml aliquot is used for analysis, the factor 2 must be replaced by a corresponding factor.

6.4 Sample concentration, dry basis, corrected to standard conditions.

$$C = K_2 \frac{m}{V_{sc}} \qquad \text{Equation 7-4}$$

where:

$$K_2 = 10^3 \frac{mg/m^3}{\mu g/ml} \text{ for metric units}$$

$$= 6.243 \times 10^{-5} \frac{lb/scf}{\mu g/ml} \text{ for English units}$$

7. Bibliography

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5. Hamil, H. F. and D. E. Camann. Collaborative Study of Method for the Determination of Nitrogen Oxide Emissions from Stationary Sources (Fossil Fuel-Fired Steam Generators). Southwest Research Institute report for Environmental Protection Agency. Research Triangle Park, N. C. October 5, 1973.

6. Hamil, H. F. and R. E. Thomas. Collaborative Study of Method for the Determination of Nitrogen Oxide Emissions from Stationary Sources (Nitric Acid Plants). Southwest Research Institute report for Environmental Protection Agency. Research Triangle Park, N. C. May 8, 1974.

SLIDE 108-0

NOTES

METHOD — 7

Determination of
Nitrogen Oxide Emissions
From Stationary Sources

SLIDE 108-1

PRINCIPLE

A GRAB sample is collected in an evacuated flask.

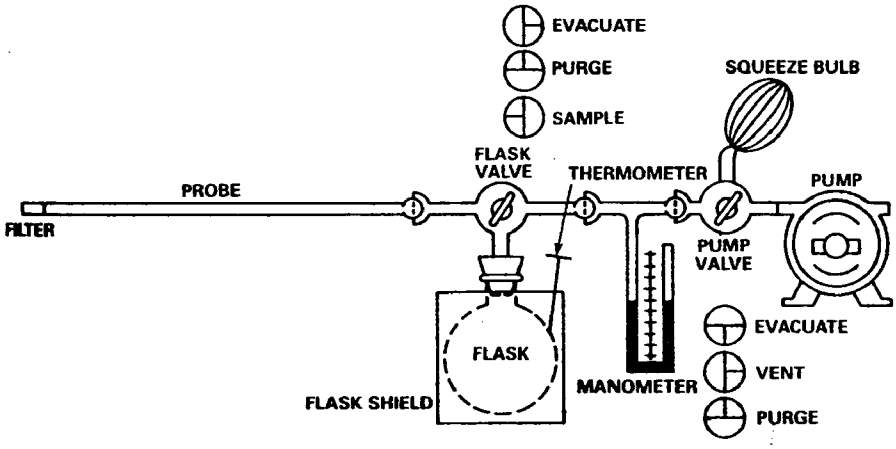
Nitrogen oxides (NO_x), except nitrous oxide, are measured colorimetrically.

APPLICABILITY

This method is applicable for the measurement of NO_x emissions from stationary sources.

SLIDE 108-2

EVACUATED FLASK SAMPLING TRAIN



SLIDE 108-3

NOTES

SPECIAL ANALYTICAL APPARATUS

- **Porcelain Evaporating Dishes**
- **Spectrophotometer**

SLIDE 108-4

SPECIAL REAGENTS

ABSORBING SOLUTION

The absorbing solution consists of concentrated H_2SO_4, deionized distilled water, and 3% hydrogen peroxide.

STANDARD KNO_3 SOLUTION

The standard KNO_3 solution consists of 2.198g of potassium nitrate dissolved in 1 liter of deionized distilled water.

WORKING STANDARD KNO_3 SOLUTION

The working standard KNO_3 solution consists of 10 ml of standard solution diluted to 100 ml with deionized distilled water.

SLIDE 108-5

COLLECTION FLASK CALIBRATION

1. Assemble clean flasks and valves.
2. Fill flask with room temperature water.
3. Measure flask volume to $\pm$ 10 ml.
4. Number each flask and record volume on flask or foam case.
5. Only one calibration is required if flasks and valves are not switched.

SLIDE 108-6

NOTES

SPECTROPHOTOMETER CALIBRATION

OPTIMUM WAVELENGTH DETERMINATION

Calibrate against a standard with a certified wavelength of 410 nm every 6 months.

Alternatively, for variable wavelength spectrophotometers scan the spectrum between 500 and 415 nm using a 200 μg NO_2 standard solution.

When a peak is obtained, the wavelength at which this peak occurs shall be the optimum wavelength.

SLIDE 108-7

SPECTROPHOTOMETER CALIBRATION

CALIBRATION FACTOR DETERMINATION

- Prepare calibration solutions containing 0.0, 1.0, 2.0, 3.0 and 4.0 ml of the KNO_3 working standard solution.
- Measure absorbance of each solution at the optimum wavelength.
- Determine calibration factor each day that samples are analyzed.

SLIDE 108-8

CALIBRATION FACTOR CALCULATION

$$K_c = 100 \frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2}$$

SLIDE 108-9

NOTES

ONSITE SAMPLING

1. Pipette 24 ml of absorbing solution into a sample flask.
2. Place probe at sampling point and connect to train.
3. Evacuate flask to 3 in. Hg absolute pressure or less.
4. Turn of pump and check for leaks.
5. Record volume of flask and valve, flask temperature, and barometric pressure.
6. Purge the probe and vacuum tubing.

SLIDE 108-10

(cont.) ONSITE SAMPLING

7. Measure absolute flask pressure.
8. Turn flask valve to "sample" permitting stack gas to enter flask.
9. Turn flask to "purge" and disconnect flask from train.
10. Shake flask for at least 5 minutes.

SLIDE 108-11

ADDING SUPPLEMENTAL OXYGEN

1. Flush cyliner with pure oxygen prior to evacuation.
2. Inject oxygen into flask after sampling.
3. Terminate sampling with minimum vacuum of 2 in. Hg and vent flask to the atmosphere.

SLIDE 108-12

NOTES

SAMPLE RECOVERY

1. Let flask set for minimum of 16 hours.
2. Shake flask for 2 min.
3. Measure absolute pressure in flask.
4. Record flask temperature and barometric pressure.
5. Transfer content of flask to leak-free polyethylene bottle.
6. Adjust PH to between 9 and 12.
7. Mark liquid level, seal, and identify container.

SLIDE 108-13

ANALYSIS

1. Check liquid level of sample container.
2. Transfer sample to a 50 ml volumetric flask; dilute to volume and mix thoroughly.
3. Pipette a 25 ml aliquot into the porcelain evaporation dish and evaporate on a steam bath.
4. Add 2 ml phenoldisulfonic acid solution to dried residue and triturate.
5. Add 1 ml deionized distilled water, four drops of concentrated sulfuric acid, and heat on a steam bath for 3 min.

SLIDE 108-14

(cont.) ANALYSIS

6. Allow solution to cool; add 20 ml deionized distilled water; add concentrated ammonium hydroxide until PH is 10.
7. Remove solids if necessary.
8. Transfer solution to a 100 ml volumetric flask and dilute to mark with distilled water.
9. Mix contents of flask thoroughly and measure the absorbance using the blank solution as a zero reference.
10. Dilute the sample and blank with equal volumes of distilled water if absorbance exceeds A_4.

SECTION I. METHOD 8

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

1. Principle and Applicability

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

1.2 Applicability. This method is applicable for the determination of sulfuric acid mist (including sulfur trioxide, and in the absence of other particulate matter) and sulfur dioxide emissions from stationary sources. Collaborative tests have shown that the minimum detectable limits of the method are 0.05 milligrams/cubic meter (0.03 x 10^{-7} pounds/cubic foot) for sulfur trioxide and 1.2 mg/m^3(0.74 x 10^{-7} lb/ft^3) for sulfur dioxide. No upper limits have been established. Based on theoretical calculations for 200 milliliters of 3 percent hydrogen peroxide solution, the upper concentration limit for sulfur dioxide in a 1.0 m^3 (35.3 ft^3) gas sample is about 12,500 mg/m^3 (7.7 x 10^{-4} lb/ft^3). The upper limit can be extended by increasing the quantity of peroxide solution in the impingers.

Possible interfering agents of this method are fluorides, free ammonia, and dimethyl aniline. If any of these interfering agents are present (this can be determined by knowledge of the process), alternative methods, subject to the approval of the Administrator, U. S. Environmental Protection Agency, are required.

Filterable particulate matter may be determined along with SO_3 and SO_2 (subject to the approval of the Administrator) by inserting a heated glass fiber filter between the probe and isopropanol impinger (see Section 2.1 of Method 6). If this option is chosen, particulate analysis is gravimetric only; H_2SO_4 acid mist is not determined separately.

2. Apparatus

2.1 Sampling. A schematic of the sampling train used in this method is shown in Figure 8-1; it is similar to the Method 5 train except that the filter position is different and the filter holder does not have to be heated. Commercial models of this train are available. For those who desire to build their own, however, complete construction details are described in APTD-0581. Changes from the APTD-0581 document and allowable modifications to Figure 8-1 are discussed in the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. Further details and guidelines on operation and maintenance are given in Method 5 and should be read and followed whenever they are applicable.

2.1.1 Probe Nozzle. Same as Method 5, Section 2.1.1.

2.1.2 Probe Liner. Borosilicate or quartz glass, with a heating system to prevent visible condensation during sampling. Do not use metal probe liners.

2.1.3 Pitot Tube. Same as Method 5, Section 2.1.3.

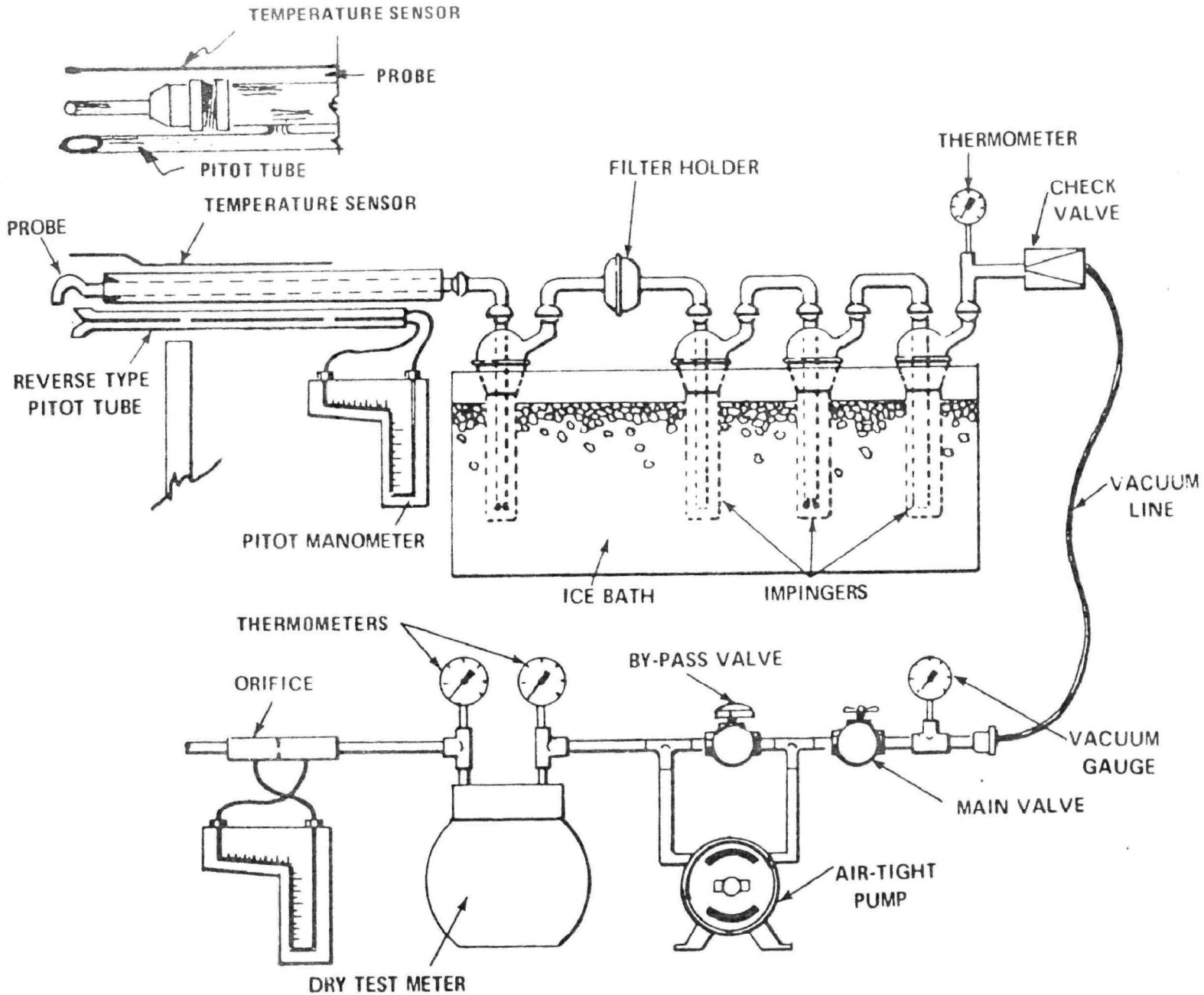


Figure 8-1. Sulfuric acid mist sampling train.

2.1.4 Differential Pressure Gauge. Same as Method 5, Section 2.1.4.

2.1.5 Filter Holder. 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 the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The filter holder shall be placed between the first and second impingers. Note: Do not heat the filter holder.

2.1.6 Impingers--Four, as shown in Figure 8-1. The first and third shall be of the Greenburg-Smith design with standard tips. The second and fourth shall be of the Greenburg-Smith design, modified by replacing the insert with an approximately 13 millimeter (0.5 in.) ID glass tube, having an unconstricted tip located 13 mm (0.5 in.) from the bottom of the flask. Similar collection systems, which have been approved by the Administrator, may be used.

2.1.7 Metering System. Same as Method 5, Section 2.1.8.

2.1.8 Barometer. Same as Method 5, Section 2.1.9.

2.1.9 Gas Density Determination Equipment. Same as Method 5, Section 2.1.10.

2.1.10 Temperature Gauge. Thermometer, or equivalent, to measure the temperature of the gas leaving the impinger train to within 1°C (2°F).

2.2 Sample Recovery.

2.2.1 Wash Bottles. Polyethylene or glass, 500 ml. (two).

2.2.2 Graduated Cylinders. 250 ml, 1 liter. (Volumetric flasks may also be used.)

2.2.3 Storage Bottles. Leak-free polyethylene bottles, 1000 ml size (two for each sampling run).

2.2.4 Trip Balance. 500-gram capacity, to measure to ±0.5 g (necessary only if a moisture content analysis is to be done).

2.3 Analysis.

2.3.1 Pipettes. Volumetric 25 ml, 100 ml.

2.3.2 Burette. 50 ml.

2.3.3 Erlenmeyer Flask. 250 ml. (one for each sample blank and standard).

2.3.4 Graduated Cylinder. 100 ml.

2.3.5 Trip Balance. 500 g capacity, to measure to ±0.5 g.

2.3.6 Dropping Bottle. To add indicator solution, 125-ml size.

3. Reagents

Unless otherwise indicated, all reagents are to conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Otherwise, use the best available grade.

3.1 Sampling.

3.1.1 Filters. Same as Method 5, Section 3.1.1.

3.1.2 Silica Gel. Same as Method 5, Section 3.1.2.

3.1.3 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.1.4 Isopropanol, 80 Percent. Mix 800 ml of isopropanol with 200 ml of deionized, distilled water.

Note: Experience has shown that only A.C.S. grade isopropanol is satisfactory. Tests have shown that isopropanol obtained from commercial sources occasionally has peroxide impurities that will cause erroneously high sulfuric acid mist measurement. Use the following test for detecting peroxides in each lot of isopropanol: Shake 10 ml of the isopropanol with 10 ml of freshly prepared 10 percent potassium iodide solution. Prepare a blank by similarly treating 10 ml of distilled water. After 1 minute, read the absorbance on a spectrophotometer at 352 nanometers (Note: Use a 1 cm path length). If the absorbance exceeds 0.1, the isopropanol shall not be used. Peroxides may be removed from isopropanol by redistilling, or by passage through a column of activated alumina. However, reagent-grade isopropanol with suitably low peroxide levels is readily available from commercial sources; therefore, rejection of contaminated lots may be more efficient than following the peroxide removal procedure.

3.1.5 Hydrogen Peroxide, 3 Percent. Dilute 100 ml of 30 percent hydrogen peroxide to 1 liter with deionized, distilled water. Prepare fresh daily.

3.1.6 Crushed Ice.

3.2 Sample Recovery.

3.2.1 Water. Same as 3.1.3.

3.2.2 Isopropanol, 80 Percent. Same as 3.1.4.

3.3 Analysis.

3.3.1 Water. Same as 3.1.3.

3.3.2 Isopropanol, 100 Percent.

3.3.3 Thorin Indicator. 1-(o-arsonophenylazo)-2-naphthol-3, 6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 ml of deionized, distilled water.

3.3.4 Barium Perchlorate (0.0100 Normal). Dissolve 1.95 g of barium perchlorate trihydrate ($Ba(ClO_4)_2 \cdot 3H_2O$) in 200 ml deionized, distilled water, and dilute to 1 liter with isopropanol; 1.22 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) may be used instead of the barium perchlorate. Standardize with sulfuric acid as in Section 5.2. This solution must be protected against evaporation at all times.

3.3.5 Sulfuric Acid Standard (0.0100 N). Purchase or standardize to $\pm$0.0002 N against 0.0100 N NaOH that has previously been standardized against primary standard potassium acid phthalate.

4. Procedure

4.1 Sampling.

4.1.1 Pretest Preparation. Follow the procedure outlined in Method 5, Section 4.1.1; filters should be inspected, but need not be desiccated, weighed, or identified. If the effluent gas can be considered dry, i.e., moisture free, the silica gel need not be weighed.

4.1.2 Preliminary Determinations. Follow the procedure outlined in Method 5, Section 4.1.2.

4.1.3 Preparation of Collection Train. Follow the procedure outlined in Method 5, Section 4.1.3 (except for the second paragraph and other obviously inapplicable parts) and use Figure 8-1 instead of Figure 5-1. Replace the second paragraph with: Place 100 ml of

80 percent isopropanol in the first impinger, 100 ml of 3 percent hydrogen peroxide in both the second and third impingers; retain a portion of each reagent for use as a blank solution. Place about 200 g of silica gel in the fourth impinger.

Note: If moisture content is to be determined by impinger analysis, weigh each of the first three impingers (plus absorbing solution) to the nearest 0.5 g and record these weights. The weight of the silica gel (or silica gel plus container) must also be determined to the nearest 0.5 g and recorded.

4.1.4 Pretest Leak-Check Procedure. Follow the basic procedure outlined in Method 5, Section 4.1.4.1, noting that the probe heater shall be adjusted to the minimum temperature required to prevent condensation, and also that verbage such as, ". . . plugging the inlet to the filter holder . . . ," shall be replaced by, ". . . plugging the inlet to the first impinger . . . ." The pretest leak-check is optional.

4.1.5 Train Operation. Follow the basic procedures outlined in Method 5, Section 4.1.5, in conjunction with the following special instructions. Data shall be recorded on a sheet similar to the one in Figure 8-2. The sampling rate shall not exceed 0.030 m^3/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 it does occur, adjust the probe heater setting upward to the minimum temperature required to prevent condensation. If component changes become necessary during a run, a leak-check shall

PLANT _____

LOCATION _____

OPERATOR _____

DATE _____

RUN NO. _____

SAMPLE BOX NO. _____

METER BOX NO. _____

METER ΔH@ _____

C FACTOR _____

PITOT TUBE COEFFICIENT, Cp _____

SCHEMATIC OF STACK CROSS SECTION

STATIC PRESSURE, mm Hg (in.Hg) _____

AMBIENT TEMPERATURE _____

BAROMETRIC PRESSURE _____

ASSUMED MOISTURE, % _____

PROBE LENGTH, m (ft) _____

NOZZLE IDENTIFICATION NO. _____

AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) _____

PROBE HEATER SETTING _____

LEAK RATE, m^3/min, (cfm) _____

PROBE LINER MATERIAL _____

FILTER NO.: _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	VACUUM mm Hg (in.Hg)	STACK TEMPERATURE (T_S), °C (°F)	VELOCITY HEAD (ΔP_S), mm H_2O (in. H_2O)	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER, mm H_2O (in. H_2O)	GAS SAMPLE VOLUME, m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
							INLET, °C (°F)	OUTLET, °C (°F)	
TOTAL							Avg	Avg	
AVERAGE							Avg		

Figure 8-2. Field data.

be done immediately before each change, according to the procedure outlined in Section 4.1.4.2 of Method 5 (with appropriate modifications, as mentioned in Section 4.1.4 of this method); record all leak rates. If the leakage rate(s) exceed the specified rate, the tester shall either void the run or shall plan to correct the sample volume as outlined in Section 6.3 of Method 5. Immediately after component changes, leak-checks are optional. If these leak-checks are done, the procedure outlined in Section 4.1.4.1 of Method 5 (with appropriate modifications) shall be used.

After turning off the pump and recording the final readings at the conclusion of each run, remove the probe from the stack. Conduct a post-test (mandatory) leak-check as in Section 4.1.4.3 of Method 5 (with appropriate modifications) and record the leak rate. If the post-test leakage rate exceeds the specified acceptable rate, the tester shall either correct the sample volume, as outlined in Section 6.3 of Method 5, or shall void the run.

Drain the ice bath and, with the probe disconnected, purge the remaining part of the train, by drawing clean ambient air through the system for 15 minutes at the average flow rate used for sampling.

Note: Clean ambient air can be provided by passing air through a charcoal filter. At the option of the tester, ambient air (without cleaning) may be used.

4.1.6 Calculation of Percent Isokinetic. Follow the procedure outlined in Method 5, Section 4.1.6.

4.2 Sample Recovery.

4.2.1 Container No. 1. If a moisture content analysis is to be done, weigh the first impinger plus contents to the nearest 0.5 g and record this weight.

Transfer the contents of the first impinger to a 250-ml graduated cylinder. Rinse the probe, first impinger, all connecting glassware before the filter, and the front half of the filter holder with 80 percent isopropanol. Add the rinse solution to the cylinder. Dilute to 250 ml with 80 percent isopropanol. Add the filter to the solution, mix, and transfer to the storage container. Protect the solution against evaporation. Mark the level of liquid on the container and identify the sample container.

4.2.2 Container No. 2. If a moisture content analysis is to be done, weigh the second and third impingers (plus contents) to the nearest 0.5 g and record these weights. Also, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

Transfer the solutions from the second and third impingers to a 1000-ml graduated cylinder. Rinse all connecting glassware (including back half of filter holder) between the filter and silica gel impinger with deionized, distilled water, and add this rinse water to the cylinder. Dilute to a volume of 1000 ml with deionized, distilled water. Transfer the solution to a storage container. Mark the level of liquid on the container. Seal and identify the sample container.

4.3 Analysis.

Note the level of liquid in containers 1 and 2, and confirm whether or not any sample was lost during shipment; note this on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

4.3.1 Container No. 1. Shake the container holding the isopropanol solution and the filter. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample. Pipette a 100-ml aliquot of this solution into a 250-ml Erlenmeyer flask, add 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat the titration with a second aliquot of sample and average the titration values. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is greater.

4.3.2 Container No. 2. Thoroughly mix the solution in the container holding the contents of the second and third impingers. Pipette a 10-ml aliquot of sample into a 250-ml Erlenmeyer flask. Add 40 ml of isopropanol, 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat the titration with a second aliquot of sample and average the titration values. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is greater.

4.3.3 Blanks. Prepare blanks by adding 2 to 4 drops of thorin indicator to 100 ml of 80 percent isopropanol. Titrate the blanks in the same manner as the samples.

5. Calibration

5.1 Calibrate equipment using the procedures specified in the following sections of Method 5: Section 5.3 (metering system); Section 5.5 (temperature gauges); Section 5.7 (barometer). Note that the recommended leak-check of the metering system, described in Section 5.6 of Method 5, also applies to this method.

5.2 Standardize the barium perchlorate solution with 25 ml of standard sulfuric acid, to which 100 ml of 100 percent isopropanol has been added.

6. Calculations

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

6.1 Nomenclature.

A_n = Cross-sectional area of nozzle, m^2 (ft^2).

B_{ws} = Water vapor in the gas stream, proportion by volume.

$C_{H_2SO_4}$ = Sulfuric acid (including SO_3) concentration, g/dscm (lb/dscf).

C_{SO_2} = Sulfur dioxide concentration, g/dscm (lb/dscf).

I = Percent of isokinetic sampling.

N = Normality of barium perchlorate titrant, g equivalents/liter.

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

T_m = Average absolute dry gas meter temperature (see Figure 8-2), °K (°R).

T_s = Average absolute stack gas temperature (see Figure 8-2), °K (°R).

T_{std} = Standard absolute temperature, 293°K (528°R).

V_a = Volume of sample aliquot titrated, 100 ml for H_2SO_4 and 10 ml for SO_2.

V_{lc} = Total volume of liquid collected in impingers and silica gel, ml.

V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf).

v_s = Average stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 8, m/sec (ft/sec).

V_{soln} = Total volume of solution in which the sulfuric acid or sulfur dioxide sample is contained, 250 ml or 1000 ml, respectively.

V_t = Volume of barium perchlorate titrant used for the sample, ml.

V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.

Y = Dry gas meter calibration factor.

ΔH = Average pressure drop across orifice meter, mm (in.) H_2O.

θ = Total sampling time, min.

13.6 = Specific gravity of mercury.

60 = sec/min.

100 = Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 8-2).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20°C and 760 mm Hg or 68°F and 29.92 in. Hg) by using Equation 8-1.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right) \frac{P_{bar} + \left(\frac{\Delta H}{13.6}\right)}{P_{std}} = K_1 V_m Y \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

Equation 8-1

where:

K_1 = 0.3858 °K/mm Hg for metric units.

= 17.64 °R/in. Hg for English units.

Note: If the leak rate observed during any mandatory leak-checks exceeds the specified acceptable rate, the tester shall either correct the value of V_m in Equation 8-1 (as described in Section 6.3 of Method 5), or shall invalidate the test run.

6.4 Volume of Water Vapor and Moisture Content. Calculate the volume of water vapor using Equation 5-2 of Method 5; the weight of water collected in the impingers and silica gel can be directly converted to milliliters (the specific gravity of water is 1 g/ml). Calculate the moisture content of the stack gas, using Equation 5-3 of Method 5. The "Note" in Section 6.5 of Method 5 also applies to this method. Note that if the effluent gas stream can be considered dry, the volume of water vapor and moisture content need not be calculated.

6.5 Sulfuric acid mist (including SO_3) concentration.

$$C_{H_2SO_4} = K_2 \frac{N (V_t - V_{tb}) \left(\frac{V_{soln}}{V_a}\right)}{V_{m(std)}}$$

Equation 8-2

where:

K_2 = 0.04904 g/milliequivalent for metric units.

= 1.081×10^{-4} lb/meq for English units.

6.6 Sulfur dioxide concentration.

$$C_{SO_2} = K_3 \frac{N\,(V_t - V_{tb})\left(\frac{V_{soln}}{V_a}\right)}{V_{m(std)}}$$

Equation 8-3

where:

K_3 = 0.03203 g/meq for metric units.

= 7.061×10^{-5} lb/meq for English units.

6.7 Isokinetic Variation.

6.7.1 Calculation from raw data.

$$I = \frac{100\,T_s\,[K_4 V_{lc} + (V_m Y/T_m)\,(P_{bar} + \Delta H/13.6)]}{60\,\theta\,v_s\,P_s\,A_n}$$

Equation 8-4

where:

K_4 = 0.003464 mm Hg-m^3/ml-°K for metric units.

= 0.002676 in. Hg-ft^3/ml-°R for English units.

6.7.2 Calculation from intermediate values.

$$I = \frac{T_s\,V_{m(std)}\,P_{std}\,100}{T_{std}\,v_s\,\theta\,A_n\,P_s\,60\,(1-B_{ws})}$$

$$= K_5 \frac{T_s\,V_{m(std)}}{P_s\,v_s\,A_n\,\theta\,(1-B_{ws})}$$

Equation 8-5

where:

K_5 = 4.320 for metric units.

= 0.09450 for English units.

6.8 Acceptable Results. If 90 percent $\leq$ I $\leq$ 110 percent, the results are acceptable. If the results are low in comparison to the standards and I is beyond the acceptable range, the Administrator may opt to accept the results. Use Citation 4 in the Bibliography of Method 5 to make judgments. Otherwise, reject the results and repeat the test.

7. Bibliography

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3. Martin, Robert M. Construction Details of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N. C. Air Pollution Control Office Publication No. APTD-0581. April, 1971.

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7. Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis. pp. 40-42. American Society for Testing and Materials. Philadelphia, Pa. 1974.

SLIDE 109-0

NOTES

METHOD — 8

Determination of Sulfuric Acid Mist
and Sulfur Dioxide Emissions
from Stationary Sources

SLIDE 109-1

PRINCIPLE

A gas sample is extracted isokinetically from the stack.

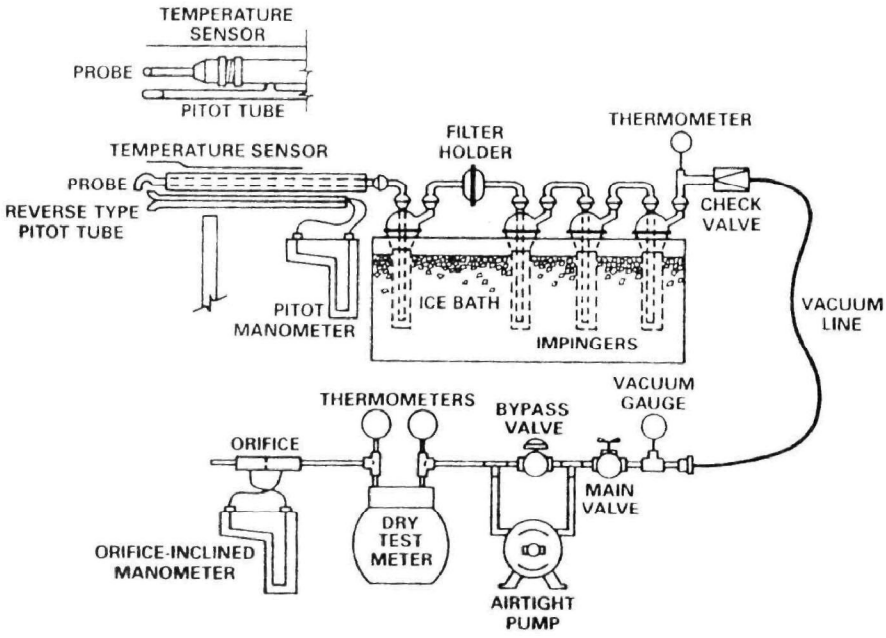
Sulfuric acid mist and sulfur dioxide are separated and measured using the barium-thorin titration method.

APPLICABILITY

This method is applicable for the determination of sulfuric acid mist emissions from stationary sources.

SLIDE 109-2

METHOD 8 SAMPLING TRAIN



SLIDE 109-3

NOTES

REAGENTS

Isopropanol which contains peroxide impurities will cause erroneously high sulfuric acid mist results.

Test each lot of isopropanol and reject if absorbance exceeds 0.1.

SLIDE 109-4

CALIBRATION

All components are calibrated using procedures outlined in Method 5.

SLIDE 109-5

ON SITE SAMPLING

1. Preliminary measurement and set up.
2. Collect stack parameters for setting isokinetic sampling rate.
3. Set up nomograph and select proper nozzle size.

SLIDE 109-6

NOTES

MAXIMUM ΔH CALCULATION

$$\text{MAXIMUM } \Delta H \leq \frac{1.09\, P_m\, M\, \Delta H\, @}{T_M}$$

SLIDE 109-7

(cont.) ON SITE SAMPLING

4. Prepare and assemble sampling train.
5. Leak-check sampling train.
6. Run test.
7. Conduct post test leak-check.
8. Purge sampling train.

SLIDE 109-8

SAMPLE RECOVERY

1. Weigh impingers to nearest 0.5 g for moisture determination.
2. Transfer contents of first impinger to a 250 ml cylinder; add filter and dilute to mark with 80% isopropanol. Transfer to container No. 1.
3. Transfer contents from second and third impingers to a 1000 ml graduated cylinder; dilute to volume with distilled water. Tansfer to container No. 2.

SLIDE 109-9

NOTES

ANALYSIS

1. Confirm that no leakage occured during transport of samples.
2. Shake container No. 1 and allow filter fragments to settle.
3. Pipette a 100 ml aliquot into a 250 ml flask.
4. Add thorin indicator and titrate to a pink end point.
5. Repeat titration. Replicate titrant volumes should be within 1% or 0.2 ml.

SLIDE 109-10

(cont.) ANALYSIS

6. Thoroughly mix solution in container No. 2.
7. Pipette a 10 ml aliquot into a 250 ml flask.
8. Add 40 ml of isopropanol and two to four drops of thorin indicator.
9. Titrate to a pink end point.
10. Repeat titration. Replicate titrant volumes should be within 1% or 0.2 ml.
11. Prepare and titrate blanks in the same manner as samples.

SECTION J. HIGHLIGHTS OF EPA METHODS 1-5

This section is under development and will be included in the manual as soon as it is available.

SECTION K. SUMMARY OF EQUATIONS

Source Sampling Calculations

This section presents the equations used for source sampling calculations. These equations are divided into two parts—equipment calibration, and source test calculations. Gaseous source test equations are included to aid the source sampler performing both particulate and gaseous emissions tests. The purpose of the section is to give the reader a quick reference to necessary mathematical expressions used in source testing experiments.

EQUIPMENT CALIBRATION EQUATIONS

Stausscheibe (Type S) Pitot Tube Calibration

Calibration Coefficient (C_p)

(Eq. 6-1)
$$C_{p(s)} = C_{p(std)}\sqrt{\frac{\Delta p_{std}}{\Delta p_s}}$$

Deviation from Average C_p (Leg A or B of Type S tube)

(Eq. 6-2)
$$Deviation = C_{p(std)} - C_p$$

Average deviation from the mean δ (Leg A or B)

(Eq. 6-3)
$$\delta = \sum_{1}^{3} \frac{|C_{p(s)} - \bar{C}_{p(A \text{ or } B)}|}{3}$$

Sampling Probe Calibration Developed by Experiment and Graphed for Each Probe Length

Test Meter Calibration Using Spirometer

Spirometer volume (temperature and pressure correction not necessary for ambient conditions)

(Eq. 6-4) $[Spirometer\ displacement\ (cm)] \times [liters/cm] = liters\ volume$

Convert liters to cubic feet (ft^3)

Test Meter Correction Factor

(Eq. 6-5)
$$\frac{Spirometer\ Standard\ ft^3}{Test\ meter\ ft^3} = Test\ meter\ correction\ factor$$

Correct Volume

(Eq. 6-6) $[Test\ meter\ volume] \times [Test\ meter\ correction\ factor] = correct\ volume$

Orifice Meter Calibration Using Test Meter

Test meter volumetric flowrate (Q_m) in cubic feet per minute

(Eq. 6-7) $Q_m = [Test\ meter\ (V_f) - Test\ Meter\ V_i] \times [Test\ meter\ correction\ factor]$

Proportionality Factor (K_m)

(Eq. 6-8) $$K_m = Q_m \sqrt{\frac{P_m\ M_m}{T_m\ \Delta H}}$$

Orifice meter $\Delta H_@$ *Flow Rate*

(Eq. 6-9) 1. *English units* $$\Delta H_@ = \frac{0.9244}{K_m{}^2}$$

where $\Delta H_@ = 0.75$ *cfm at* 68°F *and* 29.92 *in. Hg*

(Eq. 6-9) 2. *Metric units* $$\Delta H_@ = \frac{0.3306}{K_m{}^2}$$

where $\Delta H_@ = 0.021\ m^3/min$ *at* 760 *mm Hg and* 20°C

Sampling Meter Console Calibration

Ratio of the accuracy of Console Gas Meter Calibration Test Meter (γ).
Tolerance 1 ± 0.02

(Eq. 6-10) $$\gamma = \frac{V_T\ T_m\ P_b}{V_m\ T_t\ \left(P_b + \frac{\Delta H}{13.6}\right)}$$

Meter Console Orifice Meter Calibration ($\Delta H_@$)

(Eq. 6-11) 1. $$\Delta H_@ = \frac{K\ \Delta H}{P_b\ T_m} \left[\frac{T_T \theta}{V_T}\right]^2$$

where $K = 0.0317$ *English units*
$= 0.0012$ *metric units*

(Eq. 6-12) 2. $$\Delta H_@ = \frac{0.9244}{\bar{K}_m{}^2}$$

Source Sampling Nomograph Calibration

Isokinetic ΔH Equation

(Eq. 6-13) Isokinetic $$\Delta H = \left[846.72\ D_n^4\ \Delta H@\ C_p^2\ (1 - B_{ws})^2\ \frac{M_d\ T_m\ P_s}{M_s\ T_s\ P_m} \right] \Delta p$$

Sampling Nozzle Equation

(Eq. 6-14) $$D_n = \sqrt{\frac{0.0358\ Q_m\ P_m}{T_m\ C_p (1 - B_{ws})}\ \sqrt{\frac{T_s\ M_s}{P_s\ (\overline{\Delta p})}}}$$

Adjusted C-Factor (C_p)

(Eq. 6-15) $$C\text{-}factor\ adjusted = C_{factor}\ \left[\frac{C_p}{0.85} \right]^2$$

Adjusted C-Factor ($M_d \neq 29$)

(Eq. 6-15) $$C\text{-}factor\ adjusted = C_{factor}\ \frac{1 - B_{ws} + 18\ B_{ws}/29}{1 - B_{ws} + 18 B_{ws}/M_d}$$

SOURCE SAMPLING CALCULATIONS

Method 1—Site Selection

Equal Area Equation (circular ducts)

(Eq. 6-16) $$P = 50 \left[1 - \sqrt{\frac{2j - 1}{2n}} \right]$$

Equivalent Diameter for a Rectangular Duct

(Eq. 6-17) $$D_E = \frac{2(length)\ (width)}{length + width}$$

Method 2—Gas Velocity and Volumetric Flow Rate

Average Stack Gas Velocity

(Eq. 6-18) $$\bar{v}_s = K_p C_p \sqrt{\frac{T_s}{P_s M_s}} \left(\sqrt{\Delta p} \right)_{ave}$$

Average Dry Stack Gas Volumetric Flow Rate at Standard Conditions ($\bar{Q}_s$)

(Eq. 6-19) $$\bar{Q}_s = 3600\ (1 - B_{ws}) \bar{v}_s A_s \left[\frac{T_{std}}{P_{std}} \right] \frac{P_s}{T_s}$$

Method 3—Orsat Analysis

Stack Gas Dry Molecular Weight

(Eq. 6-20) $M_d = \Sigma M_x B_x = 0.44(\%CO_2) + 0.32(\%0_2) + 0.28(\%N_2 + \%CO)$

Stack Gas Wet Molecular Weight

(Eq. 6-21) $$M_s = M_d(1 - B_{ws}) + 18\ B_{ws}$$

Percent Excess Air (%EA)

(Eq. 6-22) $$\%EA = \frac{(\%0_2) - 0.05(\%CO)}{0.264\ (\%N_2) - (\%0_2) + 0.5(\%CO)} \times 100$$

Method 4—Reference Moisture Content of a Stack Gas

Volume Water Vapor Condensed at Standard Conditions (V_{wc})

(Eq. 6-23) $$V_{wc} = \frac{(ml\ H_2O)\varrho_w\ R\ T_{std}}{P_{std}\ M_w} = K_1\ (V_f - V_i)$$

where $K_1 = 0.001333\ m^3/ml$ *for metric units*
$= 0.04707\ ft.^3/ml$ *for English units*

Silica Gel

(Eq. 6-24) $$K_2 = (W_f - W_i) = V_{w_{SG}}$$

where $K_2 = 0.001335\ m^3/gm$ *for metric units*
$= 0.04715\ ft.^3/gm$ *for English units*

Gas Volume at Standard Conditions

(Eq. 6-25) $$V_{m(std)} = V_m Y_m \left(\frac{T_{std}}{P_{std}}\right) \left(\frac{P_b + \frac{\Delta H}{13.6}}{T_m}\right)$$

Moisture Content

(Eq. 6-26) $$B_{ws} = \frac{V_{wc} + V_{w_{SG}}}{V_{wc} + V_{w_{SG}} + V_{m(std)}}$$

Method 5—Particulate Emissions Testing

Dry Gas Volume Metered at Standard Conditions

Leak Rate Adjustment

(Eq. 6-27) $$V_m = [V_m - (L_1 - L_a)\theta - \sum_{i=2}^{N} (L_i - L_a)\theta_i - (L_p - L_a)\theta_p]$$

Standard Dry Volume at Sampling Meter

(Eq. 6-28) $$V_{m(std)} = V_m Y_m \left(\frac{T_{std}}{P_{std}}\right) \left(\frac{P_b + \frac{\Delta H}{13.6}}{T_m}\right)$$

Isokinetic Variation

Raw Data

(Eq. 6-29) $$\%I = \frac{100\ T_s\ [K_3 V_{lc} + (V_m / T_m)\ (P_b + \Delta H/13.6)]}{60\ \theta_s\ v_s P_s A_n}$$

where $$K_3 = 0.003454 \frac{mm\ Hg\ m^3}{ml\ °K}$$

$$= 0.002669 \frac{in.\ Hg\ ft^3}{ml\ °R}$$

Note: This equation includes a correction for the pressure differential across the dry gas meter measured by the orifice meter—average sampling run ΔH readings.

Intermediate Data

(Eq. 6-30) $$\%I = 100 \frac{T_s\ V_{m(std)}\ P_{std}}{T_{std}\ \bar{v}_s\ \theta\ A_n\ P_s\ 60(1-B_{ws})} = K_4\ \frac{T_s\ V_{m(std)}}{P_s\ \bar{v}_s\ A_n \theta (1 - B_{ws})}$$

where $K_4 = 4.320$ *for metric units*
0.09450 *for English units*

Method 8—Sulfuric Acid Mist and Sulfur Dioxide Emissions Testing

Dry volume metered at standard conditions (see equations in previous sections of this outline)

Sulfur Dioxide concentration

(Eq. 6-31) $$c_{so2} = K_3 \frac{N(V_t - V_{tb})\left(\frac{V_{solution}}{V_{aliquot}}\right)}{V_{m(std)}}$$

where $K_3 = 0.03203$ *g/meq for metric units*
$= 7.061 \times 10^{-5}$ *lb/meq for English units*

Sulfuric acid mist (including sulfur trioxide) concentration

(Eq. 6-32) $$c_{H_2SO_4} = K_2 \frac{N(V_t - V_{tb})\left(\frac{V_{solution}}{V_{aliquot}}\right)}{V_{m(std)}}$$

where $K_2 = 0.04904$ *g/meq for metric units*

$= 1.08 \times 10^{-4}$ *lb/meq for English units*

Isokinetic Variation

Raw Data

(Eq. 6-33) $$\%I = 100 \frac{T_s \left[K_4 V_{lc} + (V_m/Tm)(P_b + \Delta H/13.6)\right]}{60\theta A_n v_s P_s}$$

where $K_4 = 0.003464$ *mm Hg* $- m^3/ml - °K$

$= 0.002676$ *in. Hg* $- ft^3/ml - °R$

Concentration Correction Equations

Concentration Correction to 12% CO_2

(Eq. 6-34) $$c_{s12} = c_s \left[\frac{12}{\%CO_2}\right]$$

Concentration Correction to 50% Excess Air Concentration

(Eq. 6-35) $$c_{s50} = \left[\frac{100 + \%EA}{150}\right]$$

Correction to 50% Excess Air Using Raw Orsat Data

(Eq. 6-36) $$c_{s50} = \frac{c_s}{1 - \left[\frac{(1.5)(\%O_2) - (0.133)(\%N_2) - 0.75(\%CO)}{21}\right]}$$

F-Factor Equations

F_c Factor

(Eq. 6-37) $$E = F_c c_s \left(\frac{100}{\%CO_2}\right)$$

Used when measuring c_s and CO_2 on a wet or dry basis.

F_d Factor

When measuring O_{2d} and c_s on a dry basis

(Eq. 6-38) $$E = F_d c_{sd} \left[\frac{20.9}{20.9 - \%O_{2d}}\right]$$

When measuring O_{2d} and c_s on a wet basis

(Eq. 6-39) $$E = F_d c_{ws} \left[\frac{20.9}{20.9(1 - B_{ws}) - \frac{\%O_{2w}}{1 - B_{ws}}}\right]$$

F_w Factor

- When measuring c_s and O_2 on a wet basis
- B_{wa} = moisture content of ambient air
- Cannot be used after a wet scrubber

(Eq. 6-40) $$E = F_w \, c_{ws} \left[\frac{20.9}{20.9(1 - B_{wa}) - \%O_{2w}} \right]$$

F_0 Factor

1. Miscellaneous factor for checking Orsat data

(Eq. 6-41) $$F_o = \frac{20.9}{100} \frac{F_d}{F_c} = \frac{20.9 - O_{2d}}{\%CO_{2d}} \qquad \left(\begin{array}{c} O_2 \text{ and } CO_2 \text{ measured} \\ \text{on dry basis} \end{array} \right)$$

Opacity Equations

%Opacity

(Eq. 6-42) $$\%\,Opacity = 100 - \%\,Transmittance$$

Optical Density

(Eq. 6-43) $$Optical\ Density = \log_{10} \left[\frac{1}{1 - Opacity} \right]$$

(Eq. 6-44) $$Optical\ Density = \log_{10} \left[\frac{1}{Transmittance} \right]$$

Transmittance

(Eq. 6-45) $$Transmittance = e^{-naql}$$

Plume Opacity Correction

(Eq. 6-46) $$\log(1 - O_1) = (L_1 / L_2) \log(1 - O_2)$$

Nomenclature

A_n	—	sampling nozzle cross-sectional area
A_s	—	stack cross-sectional area
a	—	mean particle projected area
B_{wm}	—	percent moisture present in gas at meter
B_{ws}	—	percent moisture present in stack gas
C_p	—	pitot tube calibration coefficient
$C_{p(std)}$	—	standard pitot-static tube calibration coefficient
c_s	—	particulate concentration in stack gas mass/volume
c_{ws}	—	particulate concentration on a wet basis mass/wet volume
c_{s12}	—	particulate concentration corrected to 12% CO_2
c_{s50}	—	particulate concentration corrected to 50% excess air
D_E	—	equivalent diameter
D_H	—	hydraulic diameter
D_n	—	source sampling nozzle diameter
E	—	emission rate mass/heat Btu input
e	—	base of natural logarithms (ln10 = 2.302585)
%EA	—	percent excess air
F_c	—	F-factor using c_s and CO_2 on wet or dry basis
F_d	—	F-factor using c_s and O_2 on a dry basis
F_w	—	F-factor using c_{ws} and O_2 on a wet basis
F_o	—	miscellaneous F-factor for checking orsat data
$\Delta H@$	—	pressure drop across orifice meter for 0.75 CFM flow rate at standard conditions
ΔH	—	pressure drop across orifice meter
j	—	equal area centroid
K_p	—	pitot tube equation dimensional constant

$$\text{Metric Units} = 34.97 \text{ m/sec.} \left[\frac{\text{g/g mole (mm Hg)}}{(^\circ\text{K})(\text{mm } H_2O)}\right]^{1/2}$$

$$\text{English Units} = 85.49 \text{ ft./sec.} \left[\frac{\text{lb/lb mole(in. Hg)}}{(^\circ\text{R})(\text{in. } H_2O)}\right]^{1/2}$$

L	—	length of duct cross-section at sampling site
l'	—	path length
L_1	—	plume exit diameter
L_2	—	stack diameter
m	—	mass
M_d	—	dry stack gas molecular weight
M_s	—	wet stack gas molecular weight
n	—	number of particles
N_{Re}	—	Reynolds number
O_1	—	plume opacity at exit
O_2	—	in stack plume opacity
P_{atm}	—	atmospheric pressure
P_b	—	barometric pressure ($P_b = P_{atm}$)
P_m	—	absolute pressure at the meter
pmr	—	Pollutant mass rate
P_s	—	absolute pressure in the stack
P_{std}	—	standard absolute pressure Metric Units = 760 mm Hg English Units = 29.92 in. Hg
Δp	—	gas velocity pressure
$\Delta p_{(std)}$	—	standard velocity pressure read by the standard pitot tube
Δp_{test}	—	gas velocity pressure read by the type "S" pitot tube
q	—	particle extinction coefficient
Q_s	—	stack gas volumetric flow rate corrected to standard conditions
R	—	Gas law constant, $21.83 \frac{(\text{in. Hg})(\text{ft.}^3)}{(\text{lb} - \text{mole})(^\circ\text{R})}$
t	—	temperature (°Fahrenheit or °Celsius)
T_m	—	absolute temperature at the meter Metric Units = °C + 273 = °K English Units = °F + 460 = °R
T_s	—	absolute temperature of stack gas
T_{std}		standard absolute temperature Metric Units = °20°C + 273 = 293°K English Units = 68°F + 460 = 528°R
V_m	—	volume metered at actual conditions
$V_{m_{std}}$	—	volume metered corrected to standard conditions
v.p.	—	water vapor pressure
v_s	—	stack gas velocity
Volume H_2O	—	Metric units = 0.00134 m^3/ml × ml H_2O English units = 0.0472 $ft.^3$/ml × ml H_2O
W	—	width of the duct cross-section at the sampling site
θ	—	time in minutes

SOURCE SAMPLING CALCULATION SHEET

Company _____ Address _____

Test Team _____ Address _____

Test Date _____ Evaluation Date _____

Observer _____ Evaluator _____

(1) WATER VAPOR VOLUME: ($V_{w\text{-}std}$)

$$V_{w\text{-}std} = 0.0471(V_l)$$

	Run_____	Run_____	Run_____	
V_l =				ml

	Run_____	Run_____	Run_____	
$V_{w\text{-}std}$ =				scf

(2) DRY GAS VOLUME: ($V_{m\text{-}std}$)

$$V_{m\text{-}std} = 17.65Y \frac{(V_m)}{(T_m)} \left(P_{bar} + \frac{\Delta H}{13.6}\right)$$

	Run_____	Run_____	Run_____	
V_m =				cf
T_m =				^oR
P_{bar} =				"Hg.
ΔH =				"H_2O
Y =				

	Run_____	Run_____	Run_____	
$V_{m\text{-}std}$ =				scf, dry

(3) MOISTURE CONTENT: (B_w)

$$B_w = \frac{(V_{w\text{-}std})}{(V_{m\text{-}std}) + (V_{w\text{-}std})} \times 100$$

	Run_____	Run_____	Run_____	
$V_{w\text{-}std}$ =				scf
$V_{m\text{-}std}$ =				scf

	Run_____	Run_____	Run_____	
B_w =				%

(4) GAS ANALYSIS: (M_d)

	Run_____	Run_____	Run_____	
CO_2 =				%
O_2 =				%
CO =				%
N_2 =				%

	Run_____	Run_____	Run_____	
$\%CO_2 \times 0.44$ =				
$\%O_2 \times 0.32$ =				
$\%CO \times 0.28$ =				
$\%N_2 \times 0.28$ =				
M_d =				#/#-mole, dry

(5) GAS MOLECULAR WEIGHT: (M_s)

$$M_s = (M_d)(1 - \frac{B_w}{100}) + 18(\frac{B_w}{100})$$

	Run_____	Run_____	Run_____	
M_s =				#/#-mole, wet

(6) ABSOLUTE STACK PRESSURE (P_s)

$$P_s = P_{bar} + \frac{P_{stat}}{13.6}$$

P_s =	Run_____	Run_____	Run_____	"Hg.

(7) STACK VELOCITY: (V_s)

$$V_{s\text{-}avg} = 85.48(C_p)(\sqrt{\Delta p})_{avg}\sqrt{\frac{T_{s\text{-}avg}}{(P_s)(M_s)}}$$

		Run_____	Run _____	Run_____	
C_p	=				
$(\sqrt{\Delta p})avg$	=				
$T_{s\text{-}avg}$	=				^oR
P_s	=				"Hg.
M_s	=				#/#-mole

$V_{s\text{-}avg}$ =	Run_____	Run _____	Run _____	fps

(8) ISOKINETIC VARIATION: (I)

$$I = \frac{1.667(T_{s\text{-}avg})[0.00267(V_l) + \left(\frac{V_m}{T_m}\right)(Y)(P_{bar} + \frac{H}{13.6})]}{(V_{s\text{-}avg})(P_s)(\Theta)(A_n)}$$

		Run_____	Run_____	Run_____	
$T_{s\text{-}avg}$	=				^oR
V_l	=				ml
V_m	=				cf
$v_{s\text{-}avg}$	=				fps
P_s	=				"Hg.
Θ	=				min.
A_n	=				sq. ft.
Y	=				

	Run_____	Run_____	Run_____	
I =				%

(9) PARTICULATE CONCENTRATION: (c)

		Run_____	Run_____	Run_____	
A	M_n =				mg.
B	$V_{m\text{-}std}$ =				scf
R = A / B	=				

		Run_____	Run_____	Run_____	
c = 35,310(R)	=				micrograms/cubic meter, normal
= 0.0154(R)	=				grains/std. cubic foot
= 2.205×10^{-6}(R)	=				pounds/std. cubic foot

(10) VOLUMETRIC FLOW RATE (Actual):(Q)

For circular ducts

$Q = 47.1\ (V_{s\text{-}avg})(D_s)^2$

For rectangular ducts

$Q = (L)(W)(V_{s\text{-}avg}) \times 60$

		Run_____	Run_____	Run_____	
$V_{s\text{-}avg}$	=				fps
D_s	=				ft.
L	=				ft.
W	=				ft.

	Run_____	Run _____	Run_____	
$\underline{Q}$ =				acfm

(11) VOLUMETRIC FLOW RATE (Standard Conditions): (Q_{std})

$$Q_{std} = \frac{17.65(1 - \frac{B_w}{100})(Q)(P_s)}{T_{s\text{-}avg}}$$

		Run_____	Run_____	Run_____	
B_w	=				%
Q	=				acfm
P_s	=				"Hg.
$T_{s\text{-}avg}$	=				^oR

	Run_____	Run_____	Run_____	
$\underline{Q_{std}}$ =				scfm, dry

(12) POLLUTANT MASS RATE: PMR

$$PMR_c = 1.323 \times 10^{-4}(R)(Q_{std})$$

	Run_____	Run_____	Run_____	
R =				
Q_{std} =				scfm

	Run_____	Run_____	Run_____	
$\underline{PMR_c}$ =				lbs/hr

For circular ducts

$$PMR_a = \frac{1.323 \times 10^{-4}(M_n)}{\theta}\left(\frac{D_s}{D_n}\right)^2$$

For rectangular ducts

$$PMR_a = \frac{1.323 \times 10^{-4}(M_n)(L)(W)}{\theta \quad A_n}$$

	Run_____	Run_____	Run_____	
M_n =				mg.
θ =				min.
D_s =				ft.
D_n =				ft.
L =				ft.
W =				ft.
A_n =				sq. ft.

	Run_____	Run_____	Run_____	
$\underline{PMR_a}$ =				lbs/hr

(13) ISOKINETIC CHECK:

$I = PMR_a/PMR_c \times 100 =$

Run___	Run___	Run___

(14) "F" FACTOR CALCULATION

$$E = 2.205 \times 10^{-6}(R)(F)\left(\frac{20.9}{20.9-\%O_2}\right)$$

	Run___	Run___	Run___	
R =				
F =				
O_2 =				%

	Run___	Run___	Run___	
E =				lb/MM Btu

SLIDE 151-0

NOTES

SUMMARY OF EQUATIONS

EPA REFERENCE METHODS 1 - 5

SLIDE 151-1

ABSOLUTE STACK PRESSURE (P_s)

$$P_s = P_{bar} + \frac{P_{stat}}{13.6}$$

SLIDE 151-2

STACK VELOCITY (V_s)

$$V_{s\text{-}avg} = 85.49\,(C_p)(\sqrt{\Delta P})_{avg}\sqrt{\frac{T_{s\text{-}avg}}{(P_s)\,(M_s)}}$$

SLIDE 151-3

NOTES

DRY MOLECULAR WEIGHT (M_d)

$$M_d = 0.44\,(\%\,CO_2) + 0.32\,(\%\,O_2) + 0.28\,(\%\,N_2 + \%\,CO)$$

SLIDE 151-4

MOLECULAR WEIGHT OF STACK GAS (M_s)

$$M_s = M_d\,(1 - B_{ws}) + 18\,(B_{ws})$$

SLIDE 151-5

PERCENT EXCESS AIR (EA)

$$\%\,EA = \left[\frac{\%\,O_2 - 0.5\%\,CO}{0.264\%\,N_2 - (\%\,O_2 - 0.5\%\,CO)}\right]$$

NOTES

SLIDE 151-6

VOLUME OF WATER VAPOR CONDENSED ($V_{w(std)}$)

$$V_{W(std)} = \frac{(V_f - V_i)\, P_w\, R\, T_{std}}{P_{std}\, M_w}$$
$$= K_1 (V_f - V_c)$$

SLIDE 151-7

MOISTURE CONTENT (B_{ws})

$$B_{ws} = \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}}$$

SLIDE 151-8

SAMPLE GAS VOLUME ($V_{m\,std}$)

$$V_{m\,std} = V_m\, Y \left(\frac{T_{std}}{T_m}\right) \left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right]$$
$$= K_1\, V_m\, Y\, \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

SLIDE 151-9

NOTES

LEAK RATE CORRECTION CASE NO. 1

$$V_m - \left[(L_p - L_a)\,\theta \right]$$

SLIDE 151-10

LEAK RATE CORRECTION CASE NO. 2

$$V_m - (L_1 - L_a)\theta_1 - \sum_{i=2}^{n} (L_i - L_a)\theta_2 - (L_p - L_a)\theta_p$$

SLIDE 151-11

STACK GAS FLOW RATE (Q_s)

$$Q_s = A_s \times V_s \times 60$$

SLIDE 151-12

NOTES

STACK GAS FLOW RATE AT STANDARD CONDITIONS ($Q_{s(std)}$)

$$Q_{s(std)} = Q_s \frac{P_s}{P_{std}} \frac{T_{std}}{T_s} (1 - B_{ws})$$

SLIDE 151-13

ISOKINETIC VARIATION (I) FROM RAW DATA

$$I = \frac{T_s [K_3 V_{ic} + (V_m/T_m)(P_{bar} + \Delta H/13.6)]}{60 \theta V_s P_s A_N} 100$$

SLIDE 151-14

ISOKINETIC VARIATION (I) FROM INTERMEDIATE VALUES

$$I = \frac{T_s V_{m(std)} P_{std}}{T_{std} V_s \Theta A_n P_s 60 (1\text{-}Bws)} 100$$

SLIDE 151-15

NOTES

PARTICULATE CONCENTRATION(c_s)

GRAINS PER SCF

$$C_s = 0.154 \frac{Mn}{V_{m(std)}}$$

lbs per hour

$$C_s = 2.2 \times 10^{-6} \frac{Mn}{Vm(std)} \times Q_{s(std)} \times 60$$

SLIDE 151-16

ACETONE BLANK CONCENTRATION (C_a)

$$C_a = \frac{M_a}{V_a P_a}$$

SLIDE 151-17

ACETONE WASH BANK (WA)

$$W_a = C_a V_{aw} P_a$$

SECTION L. MISALIGNMENT OF PITOT TUBE

Pitot Tube Errors Due to Misalignment and Nonstreamlined Flow *

D. JAMES GROVE and WALTER S. SMITH

Although the pitot tube is the tool most commonly used for measuring velocities in stacks and ducts, it is also the tool most commonly misused. In order to use the tube properly, the stack sampler must first gain a better understanding of the errors which occur when it is misused. This paper will be limited to a discussion of the errors caused by misalignment of the pitot tube with respect to the flowing gases, and errors caused by the non-streamlined flow of the gases. Although the errors discussed here are common to all pitot tubes, data will be presented only for "S" type pitot tubes, since they are the type most commonly used. The two basic types of pitot tube misalignment are shown in Figure 1. From experimental data taken in a 12" diameter demonstration duct, with velocities on the order of 20 feet per second, the magitude of the error in the velocity measurement is given in Figures 2 and 3. The error is plotted as a

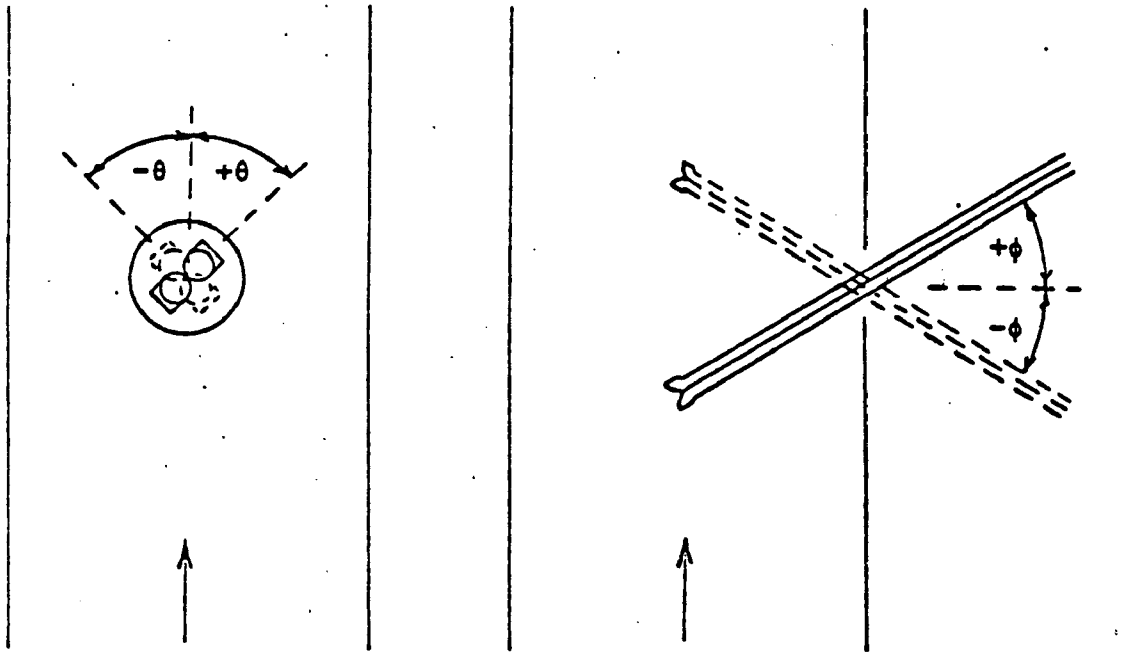


Figure 1. Types of pitot tube misalignment.

function of the angle (θ or ϕ) of misalignment, and it is the error in the velocity, not the velocity pressure (Δp). To get the approximate error in the Δp, the errors from the graph would have to be multiplied by two.

From Figure 2 we can draw several conclusions. Contrary to popular opinion, the alignment which gives the highest reading does not indicate the direction of flow. The direction of flow is indicated by the inflection point, or by a 90° rotation from the alignment which yields a zero velocity pressure. Also important is that misalignment of up to 50° will result in only a 5% error in velocity.

Figure 3 offers one immediate conclusion, that the error is not symmetrical on either side of the correct alignment. When the pitot tube is pointed into the flow, the velocities measured are generally too high, and when the pitot tube is pointed away from the flow, the velocities measured are too low. These errors are also of a much larger magnitude than those encountered from case A.

If a pitot tube is aligned so that neither θ nor ϕ are zero (case A and B simultaneously), the resulting errors would be approximately the sum of the two individual errors.

* Taken from Stack Sampling News, Vol. 1, Number 5, Nov. 1973.

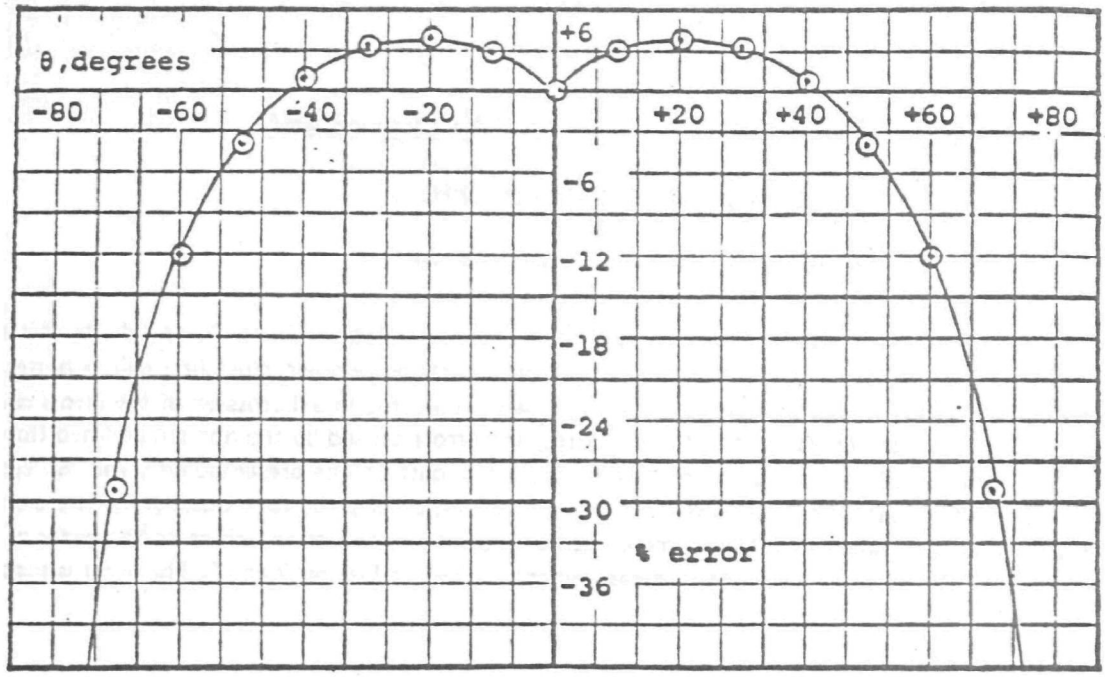


Figure 2. Velocity errors from case A Misalignment.

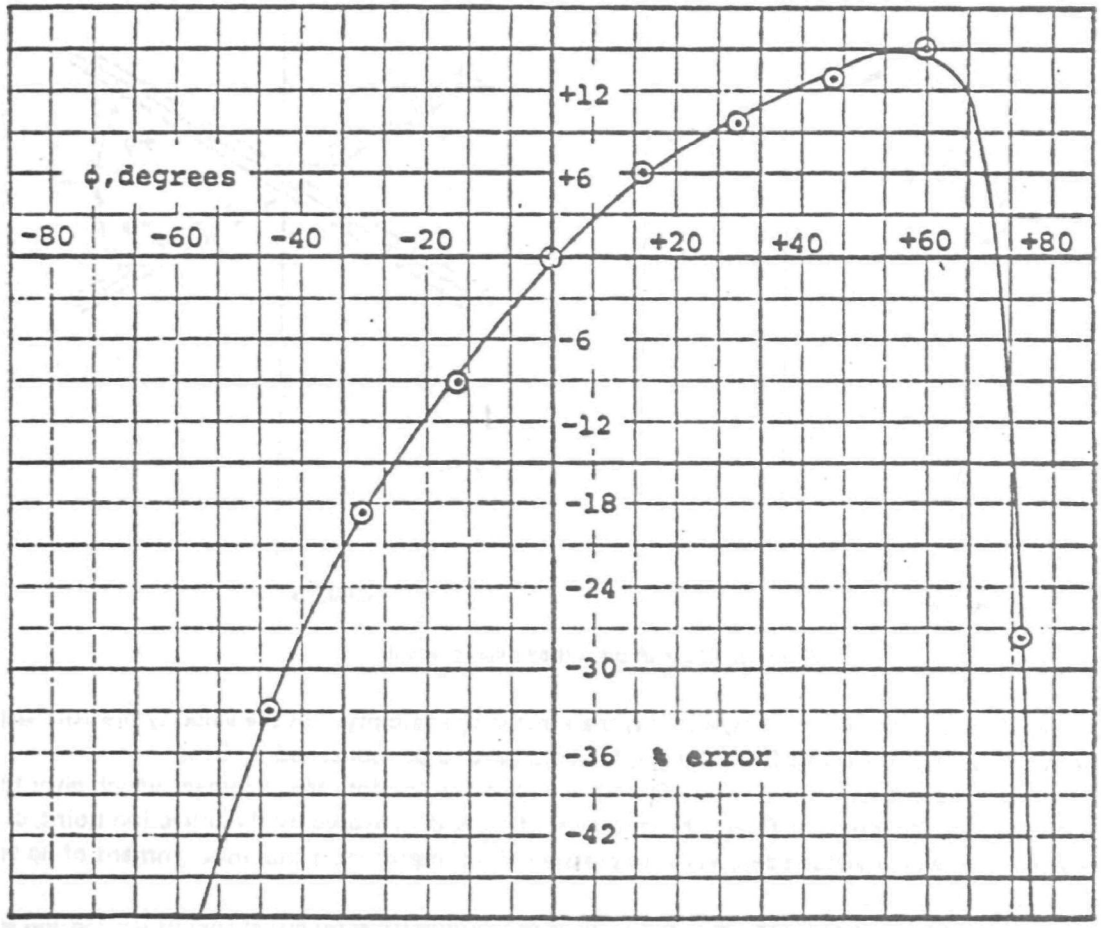


Figure 3. Velocity errors from case B misalignment.

Now that the errors due to misalignment are apparent, velocity measurements in non-streamlined flow should be considered. The primary use of the pitot tube by the stack sampler is measuring the velocity at several points on the stack cross-section which represent the average face velocity of the gases. The normal procedure is to divide the stack cross-section into equal areas, and make velocity measurements at the centroid of each of these areas. In order to obtain an average face velocity, the sampler sums the velocities at each point and divides by the number of points (equal areas). Note that in this procedure, the sampler assumes that the gases are flowing perpendicular to each of these equal areas. If this is not the case, the sampler should determine the upward (parallel to the stack walls) vector of the velocity at each area.

It should be considered what errors result when the pitot tube is placed properly with respect to the stack, but the gases are traveling at an angle to the stack wall (an an angle ϕ or θ with respect to the pitot tube). The curves in Figures 2 and 3 show that when the pitot tube is not aligned properly with the flow, the velocity reading is neither the velocity vector in the direction of flow, nor the velocity vector perpendicular to the tube. The curves do give us the error between the velocity readings and the velocity vector in the direction of flow for a given value of ϕ or θ. Also the velocity vector parallel to the stack walls will be cos ϕ or cos θ times the velocity vector in the direction of flow. Consequently, the error between the velocity readings and the velocity vector parallel to the stack walls will be a combination of error in Figures 2 or 3 and the cosine of the angle between the flow and the pitot tube. This combined error is shown in Figure 4 for case A and Figure 5 for case B. Keep in mind that these are errors in the velocity, not the Δp.

The most important conclusion to draw from these curves (Figures 4 and 5) is that if the pitot tube is aligned properly with respect to the stack, regardless of the direction of flow, the resultant velocity readings will be either close to correct or too high. There will never be a large error on the low side.

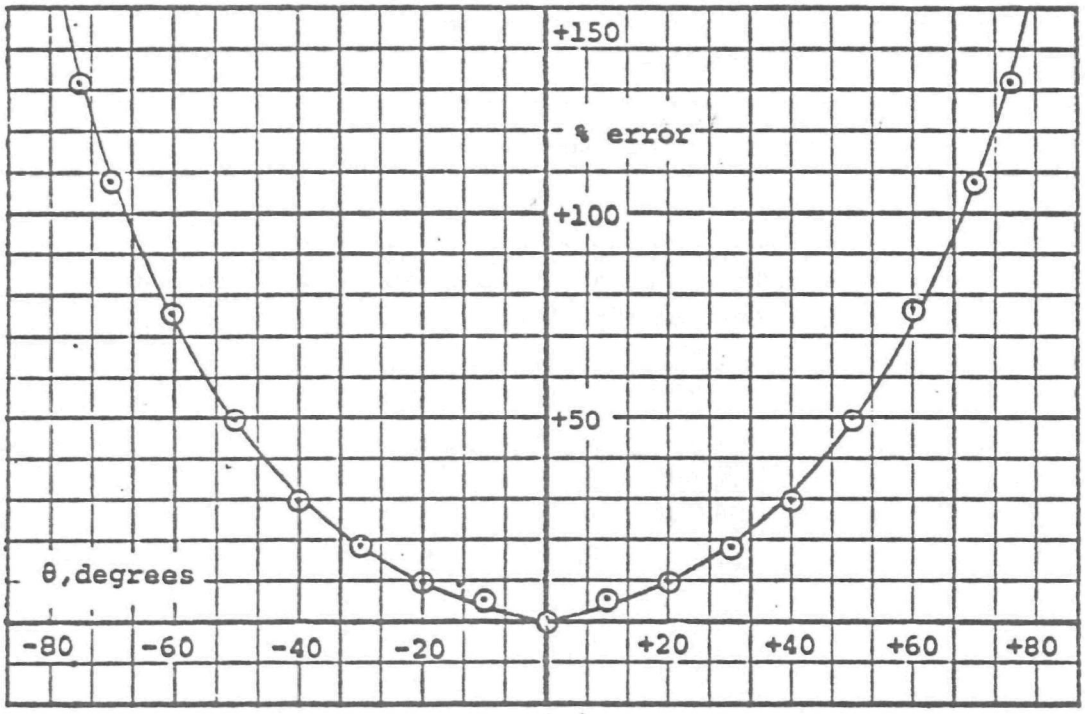


Figure 4. Velocity errors from case A flow misalignment.

There are two predominant cases of non-streamlined flow which occur in stacks and ducts that should be discussed in more detail. The first of these, which is by far the most common case, is after a bend or an elbow in the duct. The resulting flow is shown in Figure 6.

In attempting to measure the upward vector of the velocity, the error (with the pitot tube properly aligned with respect to the stack) will depend on which of the three ports shown (X, Y, and Z) are used. Port Y will give errors

corresponding to Figure 4. Port X will give errors corresponding to the left half of Figure 5, while Port Z corresponds to the right half of Figure 5. Port X is the only port which gives velocity readings at all close to the upward velocity vector, regardless of the angle.

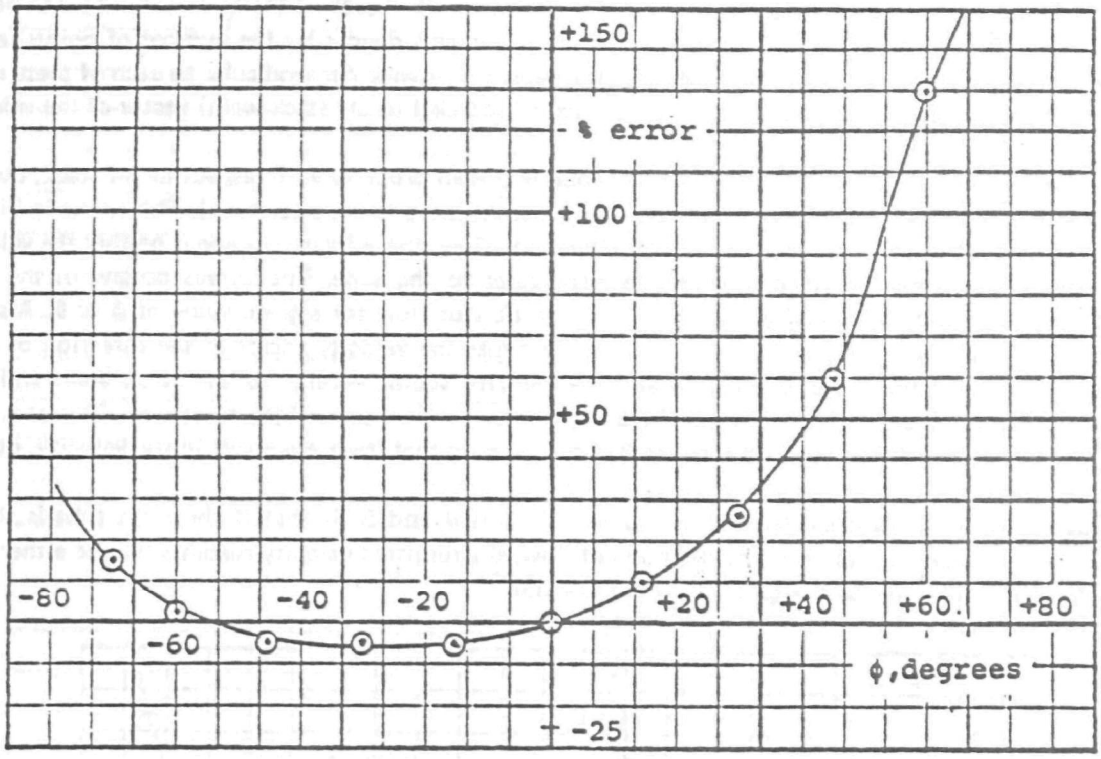


Figure 5. Velocity errors from case B flow misalignment.

The second important case of non-streamlined flow is called tangential or cyclonic flow, which normally occurs after a cyclone or a cyclonic scrubber. The resulting flow is shown in Figure 7. There are only two velocity vectors, axially and tangentially (move radially), so that regardless of which port is used (X', Y', or Z'), the error always corresponds to that in Figure 4. The larger the tangential vector, the larger the error.

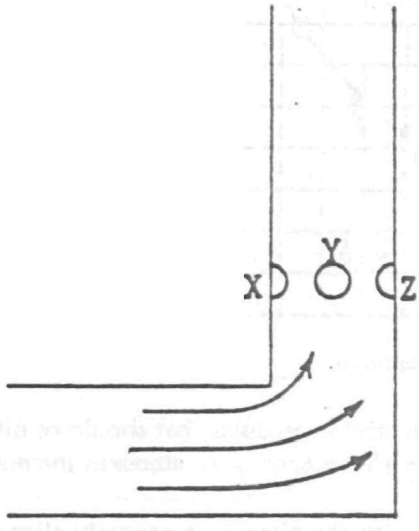


Figure 6. Flow after a bend or an elbow.

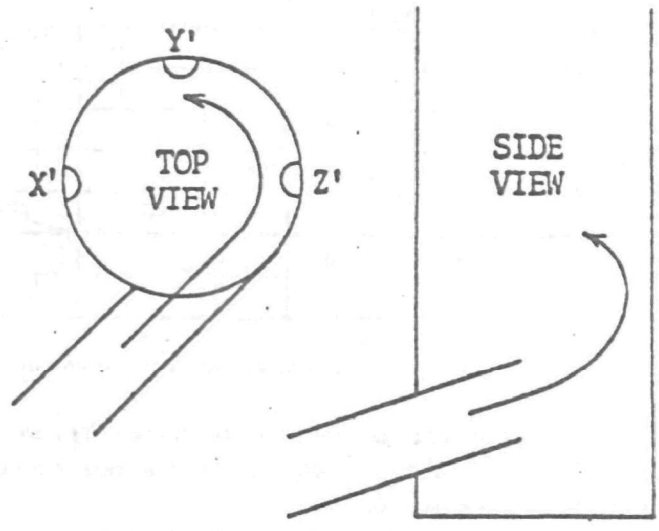


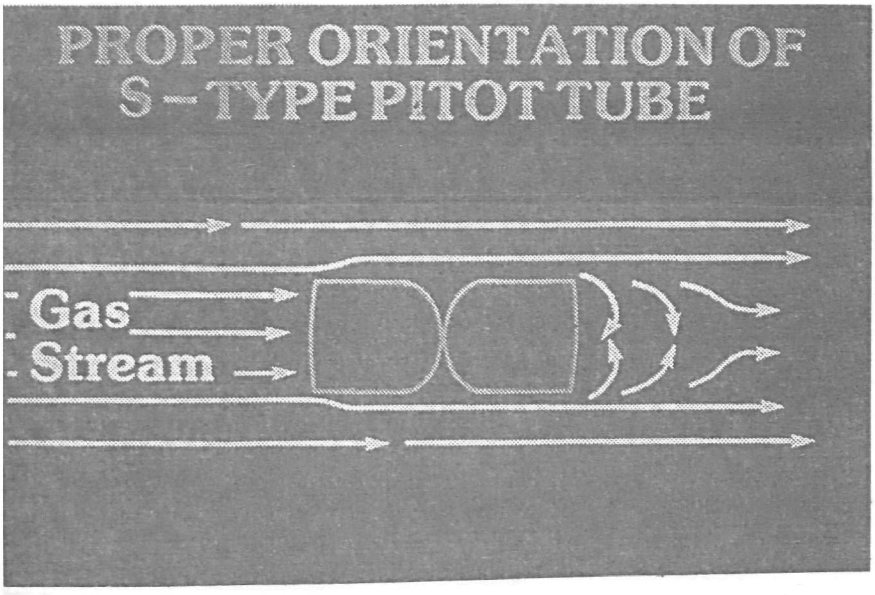
Figure 7. Tangential or cyclonic flow.

SLIDE 152-0

NOTES

PITOT TUBE MISALIGNMENT

SLIDE 152-1



SLIDE 152-2

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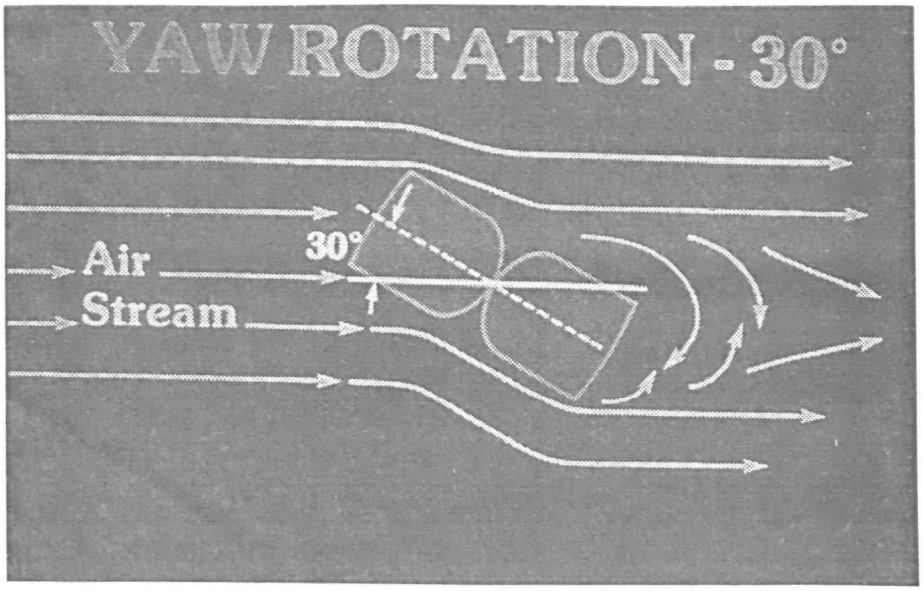
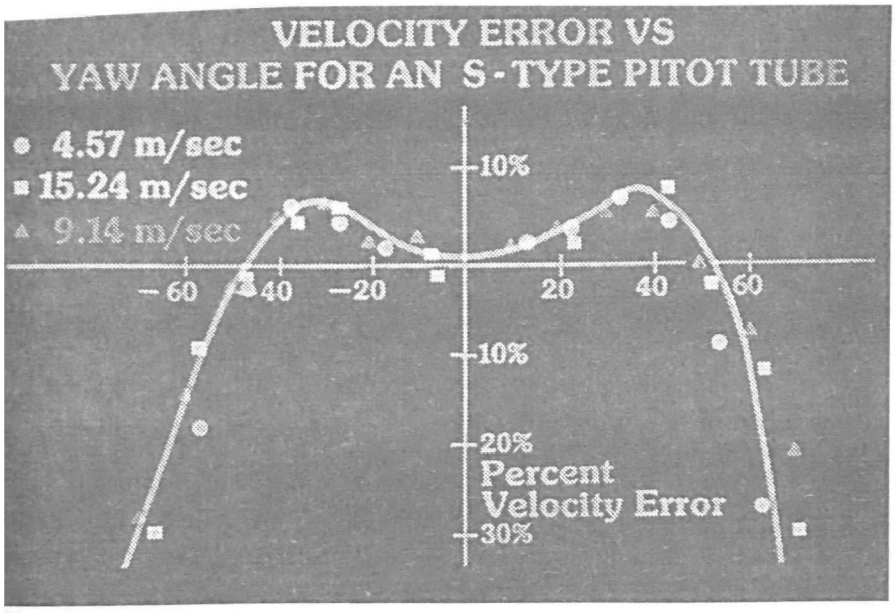


SLIDE 152-3



SLIDE 152-4

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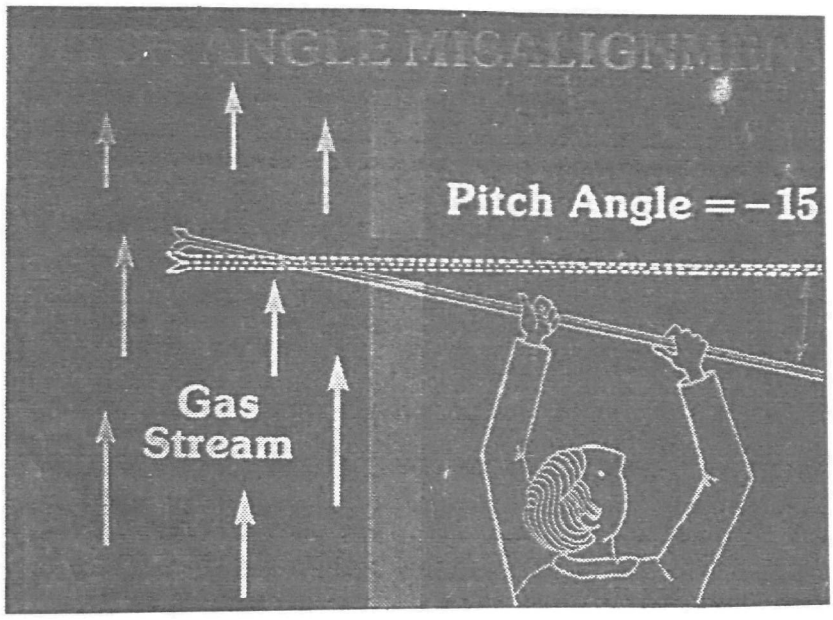
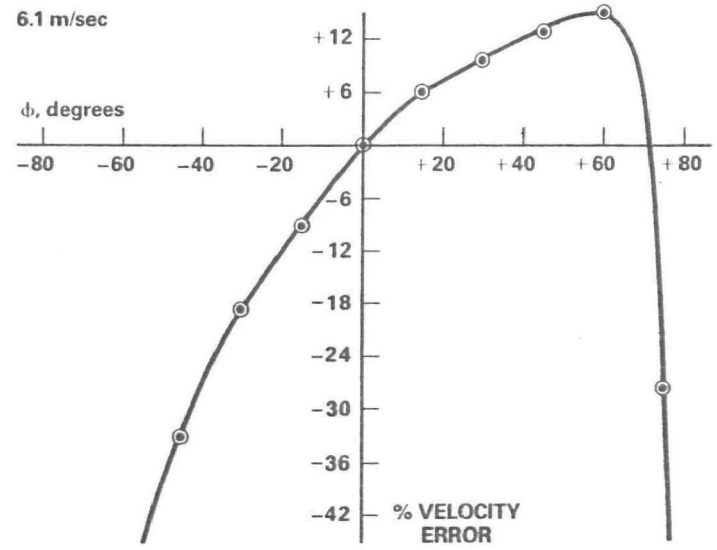


SLIDE 152-5

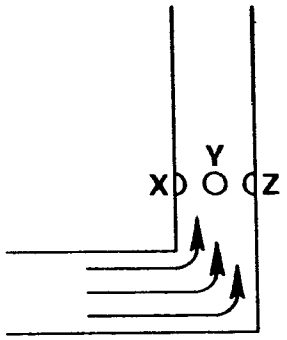
VELOCITY ERROR VS PITCH ANGLE FOR AN S-TYPE PITOT TUBE



SLIDE 152-6

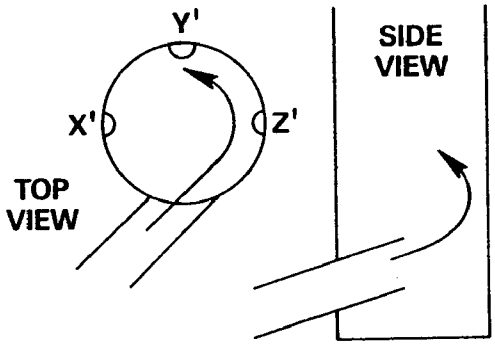
NOTES

FLOW AFTER BEND OR ELBOW



SLIDE 152-7

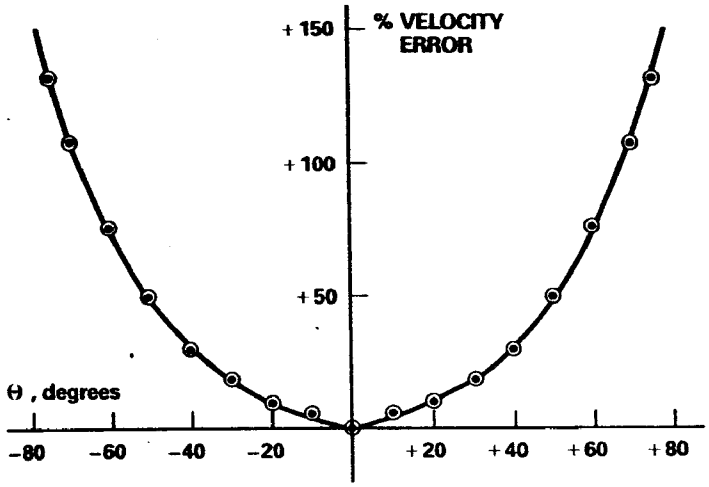
TANGENTIAL OR CYCLONIC FLOW



SLIDE 152-8

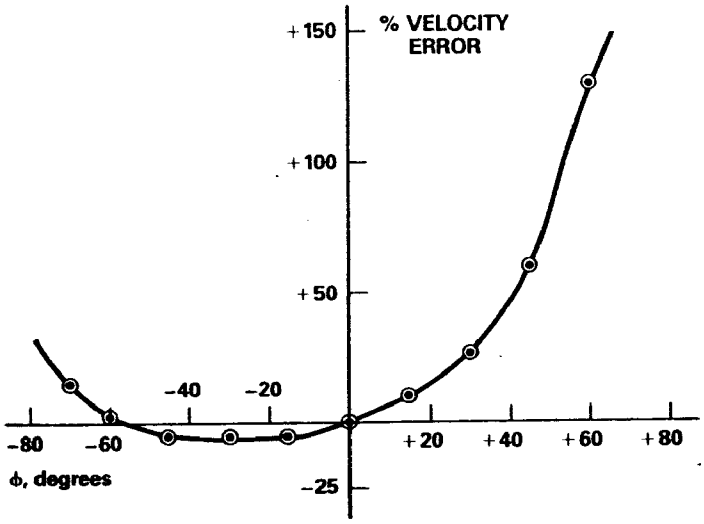
NOTES

VELOCITY ERRORS FROM TYPE A FLOW MISALIGNMENT



SLIDE 152-9

VELOCITY ERRORS FROM TYPE B FLOW MISALIGNMENT



SECTION M. ISOKINETIC SAMPLING AND BIASES FROM NONISOKINETIC SAMPLING

A Method of Interpreting Stack Sampling Data *

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INTRODUCTION

In order to obtain an average pollutant concentration ($\bar{c}_s$), two basic quantities are required: (1) total quantity of pollutant (pm_{st}) emitted from the stack per time period (θ_t), and (2) total quantity or volume of effluent gas (V_{st}) emitted from the stack for the same time period (θ_t). In equation form:

$$\bar{c}_s = \frac{pm_{st}}{V_{st}} \tag{1}$$

The objective of the stack sampler is to obtain these two basic quantities, pm_{st} and V_{st}. Since they generally cannot be measured in their entirety, multiple measurements are made in order to estimate them. The general approach is to divide the stack cross section into a number of equal areas (N_e) and to traverse the cross section by sampling at the midpoint or centroid of each equal area for equal lengths of time (θ_e). The sample is extracted at a regulated rate, isokinetically for particulates, and proportionally for gaseous pollutants.

The sampler's first assumption is that the sample extracted at the midpoint of the elemental area (A_e) is representative of that area; i.e., the concentration entering the sampling nozzle (c_n) is equal to the average concentration of the elemental area (c_e). The second assumption is that the average concentration ($\bar{c}_n$) obtained from the sampling train, upon traversing, is equal to the average stack concentration ($\bar{c}_s$) as defined by Equation 1. It has been pointed out that the correctness of the above assumptions is dependent on the time and cross-sectional variation of the pollutant concentration [1]. Whether or not the proper sampling approach was used in obtaining a representative sample concentration of the source, however, an evaluation of the stack sampling data can still be made to determine if proper sampling techniques were employed, i.e., isokinetic sampling rates for particulates and proportional rates for gases.

Hemeon and Haines [2] in 1954 pointed out two methods of calculating dust concentrations, and Badzioch [3, 4] expounded upon them in 1958 and 1960. For the purpose of this paper, these methods will be referred to as: (1) the ratio-of-area and (2) the sample-concentration calculation methods. Both utilize the inertial properties of the particles. In essence, the above authors have said that when anisokinetic conditions exist, the ratio-of-area calculation method gives better accuracy for very large or coarse particles, and the sample-concentration calculation method gives better accuracy for gases or fume-like (very small) particles. The decision of which method to use is based on prior knowledge of the particle size distribution.

The purpose of this paper is to show how the two methods can be used to interpret stack sampling data.

GENERAL PRINCIPLES

THEORY OF SAMPLING PROCEDURE AND CALCULATIONS

Consider a particulate sampling train with a nozzle of size A_n extracting a sample from an elemental area (A_e) for a time period (θ_e), where θ_e is the total sampling time (θ_t) divided by the total number of elemental or equal areas (N_e). When isokinetic conditions exist, i.e., the stack velocity at the centroid of the elemental area (v_{se}) is equal to the nozzle velocity (v_{ne}), the sampling train automatically integrates the instantaneous pollutant mass emission rate into the probe nozzle (pmr_{ne}) over the time period (θ_e). Mathematically:

* Taken from Stack Sampling News, Vol. 1, No. 2, August 1973.

$$m_{ne} = \int_{\theta_e} pmr_{ne} d\theta_e = \int_{\theta_e} A_n v_{ne} c_{ne} d\theta_e \tag{2}$$

where m_{ne} is the pollutant mass collected by the sampling train during time θ_e.

A sample traverse sums the individual m_{ne}'s for all the elemental areas or:

$$m_{nt} = \sum_{e=1}^{N_e} m_{ne} = \sum_{e=1}^{N_e} \int_{\theta_e} A_n v_{ne} c_{ne} d\theta_e \tag{3}$$

where m_{nt} is the pollutant mass collected by the sampling train during the total sampling time (θ_t). Hereafter, for simplicity, the summation sign (Σ) will denote the summation from e=1 to e=N_e.

The pollutant mass (m_{nt}) is one of the actual data that the sampler receives. This datum (m_{nt}) is used to estimate the average pollutant mass emission rate ($\overline{pmr}_s$) from the stack. This can be accomplished in the following manner.

Dividing Equation 2 through by A_n yields:

$$\frac{m_{ne}}{A_n} = \int_{\theta_e} c_{ne} v_{ne} d\theta_e$$

For isokinetic conditions ($v_{ne} = v_{se}$), using the *assumption* that the sample concentration is representative of the elemental area concentration ($c_{ne} = c_{se}$), it follows that the pollutant mass from the elemental area (m_{se}/A_e) is:

$$\frac{m_{se}}{A_e} = \int_{\theta_e} c_{se} v_{se} d\theta_e = \int_{\theta_e} c_{ne} v_{ne} d\theta_e = \frac{m_{ne}}{A_n}$$

$$\text{or } m_{se} = \frac{A_e}{A_n} m_{ne} \tag{4}$$

Dividing Equation 4 by θ_e yields the average pollutant mass emission rate ($\overline{pmr}_{se}$) from the elemental area (A_e):

$$\overline{pmr}_{se} = \frac{m_{se}}{\theta_e} \frac{A_e}{A_n} \frac{m_{ne}}{\theta_e} \tag{5}$$

The sample traverse yields the average pollutant mass rate from the entire stack:

$$\overline{pmr}_s = \Sigma\, pmr_{se} = \Sigma \frac{A_e}{A_n} \frac{m_{ne}}{A_e} = \frac{A_e}{A_n \theta_e} \Sigma\, m_{ne} \tag{6}$$

Substituting Equation 3 and using $\theta_e = \theta_t/N_e$ and $A_s = A_e N_e$, Equation 6 can be rewritten as:

$$\overline{pmr}_s = \frac{m_{nt} A_s}{\theta_t A_n} \tag{7}$$

Equation 7 is the ratio-of-area method of calculation.

The second method of estimating $\overline{pmr}_s$ is to use the average sample concentration ($\overline{c}_n$) and the average stack velocity ($\overline{v}_s$). Notice how this is effected.

The sample volume (V_{ne}) that the sampling train extracts during time (θ_e) from the elemental area (A_e) is also an integrated volume. Mathematically:

$$V_{ne} = \int_{\theta_e} A_n v_{ne} d\theta_e \tag{8}$$

A sample traverse again yields the sum of the volumes defined by Equation 8.

$$V_{nt} = \Sigma V_{ne} = \Sigma \int_{\theta_e} A_n V_{ne} d\theta_e \tag{9}$$

By combining Equations 3 and 9, the average sample concentration can be calculated.

$$\overline{c}_n = \frac{m_{nt}}{V_{nt}} \tag{10}$$

It is then assumed that $\overline{c}_n = \overline{c}_s$.

If velocity measurements are taken simultaneously with the pollutant sample and it is assumed that the average stack velocity ($\overline{v}_s = \psi \overline{v}_{se}/N_e$, $\overline{c}_n$ and $\overline{v}_s$ can be used to determine $\overline{pmr}_s$:

$$\overline{pmr}_s = \overline{c}_n \overline{V}_s A_s = \frac{m_{nt}}{V_{nt}} \overline{v}_s A_s \tag{11}$$

Equation 11 is the sample-concentration method of calculation.

The same analogy can be applied to pollutant concentration instead of the pollutant mass emission rate of Equation 7 and Equation 11, yielding the following:

$$\overline{c}_s = \frac{m_{nt}}{V_{nt}} \qquad \text{Sample-Concentration Method} \tag{12}$$

$$\overline{c}_s = \frac{m_{nt}}{\theta_t \overline{v}_s A_n} \qquad \text{Ratio-of-Area Method} \tag{13}$$

Equations 12 and 13 are the equations used by Hemeon and Haines, and by Badzioch. For the sake of brevity, the ensuing discussion will be limited to Equations 7 and 11.

At this point, Equations 7 and 11 will be redefined and summarized to distinguish between the two methods as follows:

$$\overline{pmr}_a = \frac{m_{nt}}{\theta_t} \frac{A_s}{A_n} \tag{7}$$

$$\overline{pmr}_c = \frac{m_{nt}}{V_{nt}} \overline{v}_s A_s \tag{11}$$

where: $\overline{pmr}_a$ = pollutant mass emission rate calculated on a ratio-of-area basis

$\overline{pmr}_c$ = pollutant mass emission rate calculated on a sample concentration basis

m_{nt} = pollutant mass collected by the sampling train in total sampling time (θ_t)

A_s = area of stack cross section

A_n = area of sampling nozzle

V_{nt} = actual metered sample gas volume corrected to stack conditions

$\overline{v}_s$ = average measured stack gas velocity at stack conditions

DETERMINATION OF AVERAGE SAMPLING RATE

The ratio $\overline{v}_n/\overline{v}_s$ is the ratio of isokinetic conditions on an average. By taking the ratio of $\overline{pmr}_a$ to $\overline{pmr}_c$, and noting that $\overline{v}_n = V_{nt} / A_n \theta_t$:

$$\frac{\overline{pmr}_a}{\overline{pmr}_c} = \frac{\dfrac{\overline{m}_{nt} A_s}{\theta_t A_n}}{\dfrac{\overline{m}_{nt} \overline{v}_s A_s}{V_{nt}}} = \frac{V_{nt}}{A_n \theta_t \overline{v}_s} = \frac{\overline{v}_n}{\overline{V}_s} \qquad (14)$$

We see that $\overline{pmr}_a/\overline{pmr}_c$ is also a measure of the degree of isokinetic conditions on an average. Summarizing in Table 1 as follows:

Table 1. Average Sampling Rate

Ratio, $\overline{pmr}_a / \overline{pmr}_c$	Average Condition
1	Isokinetic
< 1	Underisokinetic
> 1	Overisokinetic

Equation 14 says: (1) that only when isokinetic conditions exist will $\overline{pmr}_a = \overline{pmr}_c$, (2) that in order to determine the ratio $\overline{pmr}_a/\overline{pmr}_c$, it is necessary only to know V_{nt}, θ_t, A_n, and $\overline{v}_s$, and (3) that the ratio $\overline{pmr}_a/\overline{pmr}_c$ is not a function of the characteristic of the pollutant collector (an independent evaluation must be made on the efficiency of the collector).

DISCUSSION AND CATEGORIES OF SAMPLING RATES

It must be understood what the possibilities of Table 1 mean. When $\overline{pmr}_a/\overline{pmr}_c \neq 1$, it means that the results are definitely questionable. When $\overline{pmr}_a/\overline{pmr}_c = 1$, it means that isokinetic conditions were maintained on an average during the sampling period, but does not necessarily mean that the results are valid. The integrated volumes can be obtained by sampling at the average stack velocity ($\overline{v}_s$) while traversing such that $\overline{v}_n = \overline{v}_s$. But because of variation of velocity over the cross section, there are times when anisokinetic conditions will exist. The same situation could arise due to fluctuation of velocity. Since

$$V_{nt} = \bar{v}_s A_n \theta_t \tag{15}$$

when $\bar{v}_n = \bar{v}_s$, it should be obvious that the criteria of Table 1 are valid only if V_{nt} and $\bar{v}_s$ are obtained independently; i.e., V_{nt} is not calculated using Equation 15, or vice versa.

Use of Equation 15 is equivalent to the ratio-of-area method of calculation as this would make $\overline{pmr}_c = \overline{pmr}_a$. Such sampling trains using *only* a rate meter (orifice, rotameter, cyclone, etc.) or only a null-balance nozzle are not amenable to the criteria of Table 1 as they are not subject to any cross-check at all. For example, errors could be introduced through:

1. Miscalculation in the sampling rate leading to an improper meter setting.
2. Careless meter of equipment adjustments.
3. Improper calibration of meters or of null-balance nozzles.
4. Misuse through pure ignorance of the operating principles of the rate meters or null-balance nozzles.

In the above conditions, isokinetic rates can be claimed without dispute.

An evaluator can only:

1. Rely on reputation of the sampler.
2. Critically examine the procedure.
3. Check the results using a material balance.
4. Rely on past data whose reliability may also be questionable.

All this points to the fact that a volumetric device such as a dry gas meter is a must in a sampling train if any interpretation is to be made. An independent velocity reading (pitot tube), preferably simultaneous with sampling, is also a must.

With the understanding that the ratio $\overline{pmr}_a/\overline{pmr}_c$ be a function of the *independent* measurable quantities (V_{nt}, θ_t, A_n, and $\bar{v}_s$, the different sampling conditions can be categorized as shown in Table 2

Table 2. Sampling Rate Categories

Case 1:	$\overline{pmr}_a/\overline{pmr}_c$	=	1 or $\bar{v}_n$	= $\bar{v}_s$
Case 2:	$\overline{pmr}_a/\overline{pmr}_c$	≠	1 or $\bar{v}_n$	≠ $\bar{v}_s$

ERRORS IN PROPORTIONAL ANISOKINETIC CONDITIONS

Before any guidelines can be established, it must be understood how anisokinetic conditions affect the results because the practical situation says that Case 2 is the more general situation. Going back to Equation 3, it can be seen that this case ($\bar{v}_n \neq \bar{v}_s$) could affect the amount of pollutant collected.

$$m_{nt} = \Sigma \int_{\theta_s} A_n v_{ns} c_{ns} d\theta_s \tag{3}$$

The general premise is that when $v_n = v_s$, then $c_n = c_s$; but when $v_n \neq v_s$, $c_n \neq c_s$ except when sampling for gases and small particles. Thus the amount of pollutant collected by the sampling train depends on v_n/v_s and the variation of c_s. The degree of deviation from the time value (c_s) depends on the method of calculation and the particle size distribution.

Hemeon and Haines [2] and Badzioch [3, 4] illustrated the effect under steady state conditions (c_s = constant) of particle sizes depending on the calculation methods when *only* large particles and *only* small particles were present. Figures

1 and 2 illustrate this. Figure 3 is a combination of Figures 1 and 2, and Table 3 summarizes the biases relative to the true $\overline{pmr}_s$ [5].

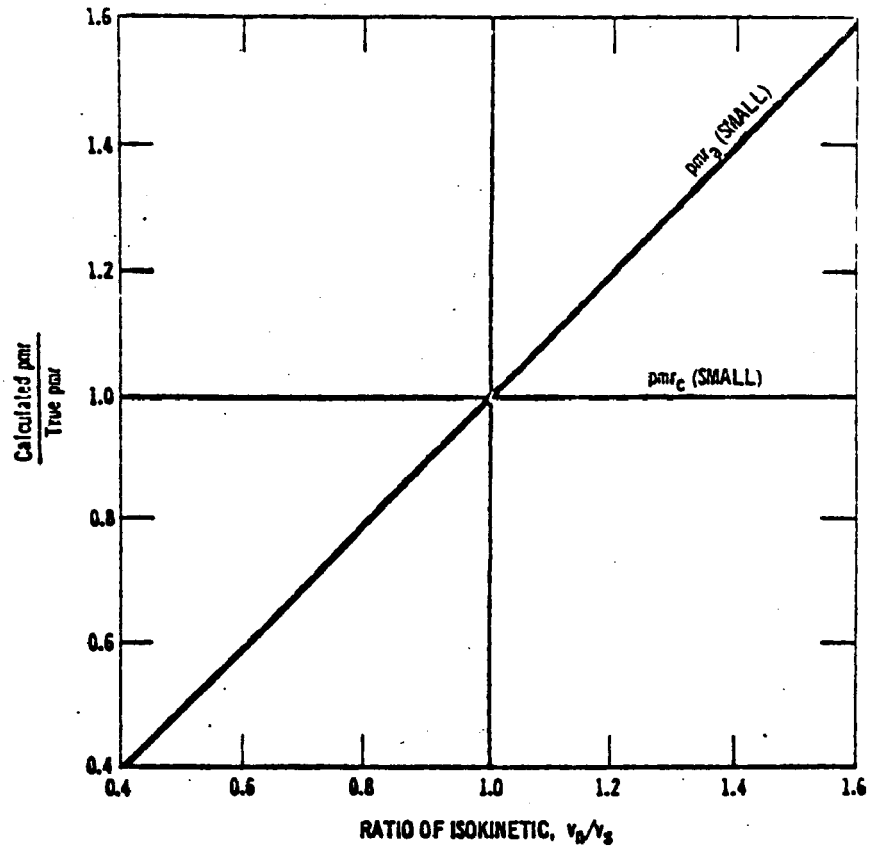


Figure 1. Errors due to anisokinetic conditions (small particles).

Table 3. Constant Proportional Condition

Ratio $\overline{pmr}_a/\overline{pmr}_c$	Condition	Bias relative to true $\overline{pmr}_s$	
		$\overline{pmr}_a$	$\overline{pmr}_c$
1	Isokinetic	=	=
< 1	Underisokinetic	<	>
> 1	Overisokinetic	>	<

According to Figure 1, when *only* small particles exist (including gaseous pollutants), the sample-concentration method of calculation gives accurate results; according to Figure 2, when *only* large particles exist, the ratio-of-area calculation method gives the most accurate result under anisokinetic conditions. Figure 3 shows the maximum deviation from the true value [as marked by pmr_a (small) and pmr_c (large)] that could possibly occur if constant proportional conditions are maintained.

Unfortunately, the two extremes, all large particles or all small particles, do not often occur. A mixture of large and small particles is the more general case. The dotted line in Figure 3 shows the case where 50 percent by weight are small and 50 percent are large particles. A mixture of different size particles, then, only tends to decrease or shrink the outer

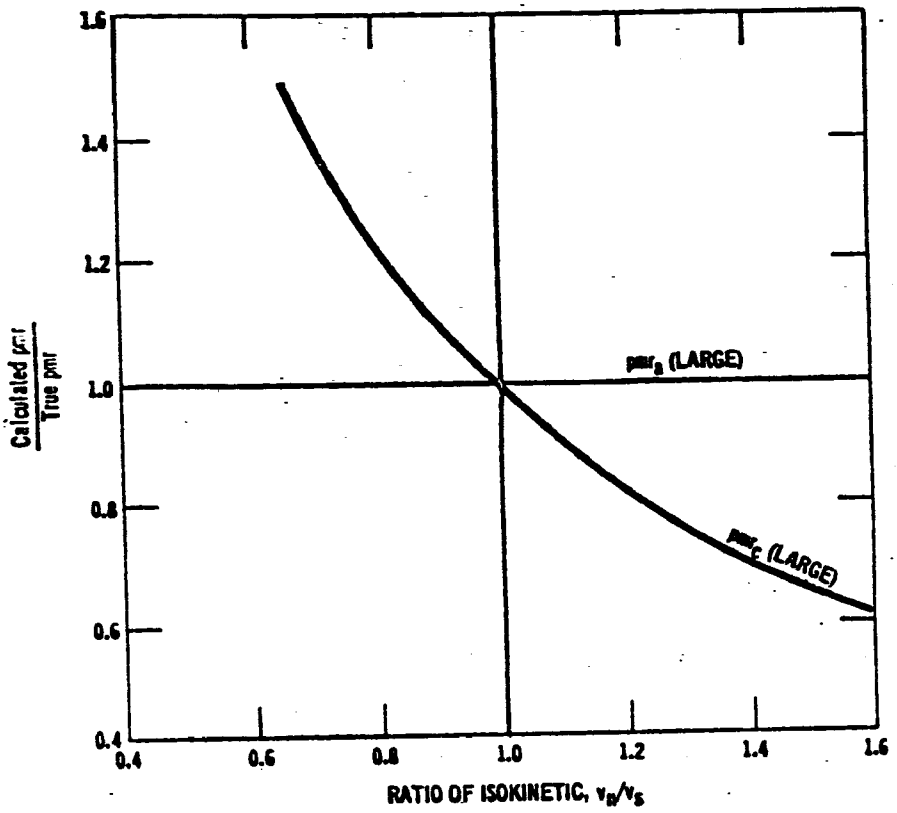


Figure 2. Errors due to anisokinetic conditions (large particles).

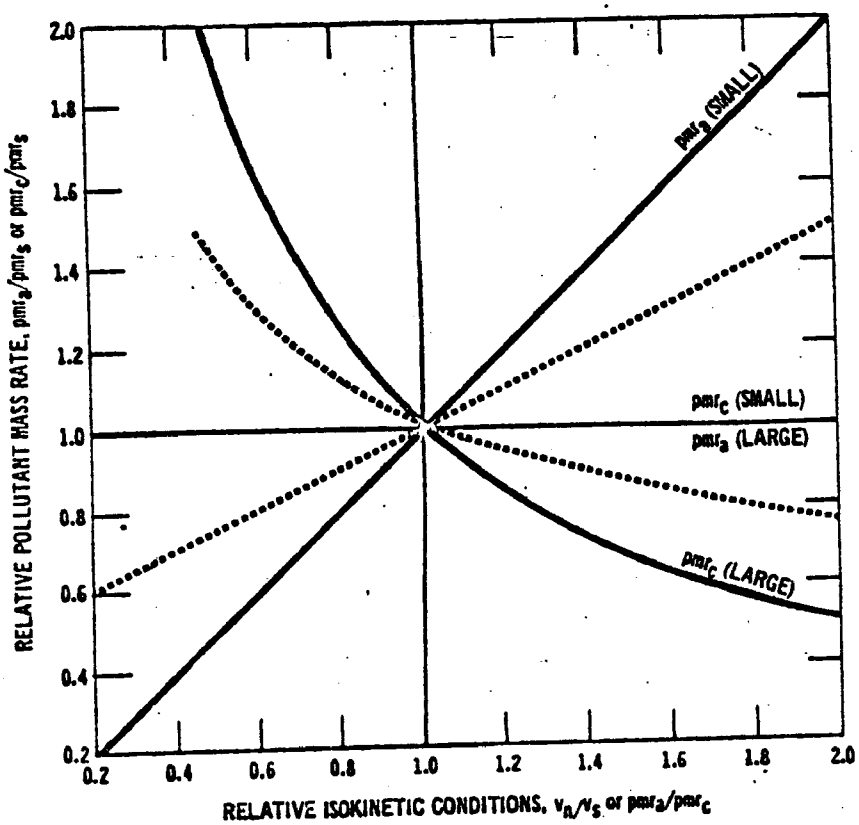


Figure 3. Errors due to anisokinetic conditions (50% small and 50% large particles).

boundaries. It is impossible to exceed the outer boundaries if exact, constant, proportional conditions are maintained.

Realizing this, it can be stated that the true $\overline{pmr}_s$ value lies somewhere between $\overline{pmr}_a$ and $\overline{pmr}_c$. It may be closer or even equal to one or the other, depending on the particle size distribution. Without this knowledge, the best that can be said is:

$$\widehat{\overline{pmr}}_s = \frac{\overline{pmr}_a + \overline{pmr}_c}{2} \pm \frac{\overline{pmr}_a - \overline{pmr}_c}{2} \tag{16}$$

when $\widehat{\overline{pmr}}_s$ is the estimate of the true $\overline{pmr}_s$.

Under the practical situation, it is impossible to maintain isokinetic or proportional conditions without some degree of fluctuation. We need to understand how this would affect the sampling results.

Badzioch wrote an equation relating the pollutant mass collected to the actual pollutant mass in the stack as:

$$\frac{m_{ne}}{m_{se}} = (1-\alpha)\frac{v_{ne}}{v_{se}} + \alpha$$

where α = parameter, depending on the inertia of the particle and the gas flow pattern at the sampling nozzle. Also, it was reported that α was a constant for $v_{ne}/v_{se} = 0.5$ to 4. Watson [6] approximated the range as 0.5 to 2.

Thus for a given particle size distribution, when c_{se} = constant, then the average $(\overline{\alpha})$ is a constant, and:

$$\frac{m_{ne}}{m_{se}} = (1-\overline{\alpha})\frac{v_{ne}}{v_{se}} + \overline{\alpha}$$

which is a straight-line function. Further, if $\overline{v}_{ne} = \overline{v}_{se}$ or $\overline{v}_{ne} = k\,\overline{v}_{se}$, the ratio of m_n to m_s remains fixed but is biased depending on the value $\overline{v}_n/\overline{v}_s$.

PROPOSED PROCEDURE

FACTS AND ASSUMPTIONS

The bases may now be set for the interpretation procedure.

1. There is no knowledge of particle size distribution or how the pollutant concentration is varying with time.
2. The following data are provided:
   a. $\overline{v}_s$, m_{nt}, A_n, V_{nt}, and θ_t. V_{nt} must be obtained independently of $\overline{v}_s$.
   b. V_{ne}, θ_e, and $\overline{v}_{se}$ for each elemental area.
3. Results within ± 10 percent of the true value are desired.
4. There is no deliberate attempt to bias the data.
5. Fluctuation within the stack of both velocity and pollutant concentration at any point are random.
6. Only averages can be calculated.

PROCEDURE AND EXPLANATION

The following steps should ensure valid results within ± 10 percent of the true value. The limit, ± 10 percent, is purely arbitrary; an understanding of this example should enable you to set your own desired limits.

With the principles set forth, a method of evaluating source sampling data can now be proposed.

1. Calculate $\overline{pmr}_a$ and $\overline{pmr}_c$, using independent measurements of V_{nt} and $\overline{v}_s$.

2. Calculate the ratio $\overline{pmr}_a/\overline{pmr}_c$ and $\dfrac{pmr_a - pmr_c}{pmr_a + pmr_c}$

   a. If $1.2 < \overline{pmr}_a/\overline{pmr}_c < 0.82$ or $\dfrac{pmr_a - pmr_c}{pmr_a + pmr_c} > \pm 0.10$, reject the results.

   b. If $0.82 \leq \overline{pmr}_a/\overline{pmr}_c \leq 1.2$ or $\dfrac{pmr_a - pmr_c}{pmr_a + pmr_c} \leq \pm 0.10$, go to step 3.

3. Calculate $\dfrac{V_{ne}}{\overline{v}_{se} A_n \theta_e} = \dfrac{\overline{v}_{ne}}{\overline{v}_{se}}$

   V_{ne} is an integrated volume over the sampling period θ_e, whereas V_{se} is a result of the readers estimation of the average reading of the inclined manometer. The inclined manometer of the pitot tube does not give V_{se} directly but, instead, yields the velocity pressure (Δp_{se}). This reading, the Δp_{se}, must be within ± 10 percent of the average value [7]. Therefore, if Δp_{se} varies more than this amount during θ_e, the rate must be adjusted, and the Δp_{se} and the time must be recorded so an average $\overline{v}_{se}$ can be calculated.

   a. If any $1.2 < \dfrac{V_{ne}}{\overline{v}_{se} A_n \theta_e} < 0.82$, reject the data.

   b. If all $0.82 \leq \dfrac{V_{ne}}{\overline{v}_{se} A_n \theta_e} \leq 1.20$, average $\overline{pmr}_a$ and $\overline{pmr}_c$.

   This value will be within ± 10 percent of the true $\overline{pmr}_s$.

SUMMARY

There are two approaches used to calculate the pollutant mass rate:

1. The ratio of area method in which the sample collected is weighted according to the ratio of the cross-sectional areas of the stack and the sample nozzle.
2. The concentration based method in which the sample collected is weighted according to the ratio of the volumetric flows in the stack and in the sample nozzle.

The source tester should be aware of how the two methods for calculating the pollutant mass rate can be biased by the particle size distribution of the pollutant when isokinetic conditions are not maintained. Although it is shown that the bias is predictable if either all large particles or all small particles are present, the real situation usually lies somewhere between these predictable limits. A compromise between these two limits then is the best estimate of the true value when exact knowledge of the particle size distribution is lacking. The procedure recommended is to obtain the average value of the two methods, which is a model based upon the concept of Hemeon and Haines [2] and others [3, 4, 6] and can be applied to real sources.

The two methods for calculating pollutant mass rate do not take into account time and cross-sectional variations of the pollutant mass rate. However the ratio of the methods, $\overline{pmr}_a/\overline{pmr}_c$, can be used to determine the average percent of isokinetic or proportional conditions. With additional real time data, e.g., velocity and sample volume reading, a more detailed evaluation can be made. This shows the necessity of having a velocity measuring instrument during sampling and a volume meter in the sampling train.

REFERENCES

1. W. C. Achinger, R. T. Shigehara, "A guide for selecting methods for different source conditions," *J. APCA 18*, 605–609 (September 1968).
2. W. C. L. Hemeon, G. F. Haines, Jr., "The magnitude of errors in stack dust sampling," *Air Repair 4*, 159–164 (November 1954).
3. S. Badzioch, "Collection of gas-borne dust particles by means of an aspirated sampling nozzle," *British J. of Applied Physics 10*, 26–32 (January 1959).
4. S. Badzioch, "Correction for anisokinetic sampling of gas-borne dust particles," *J. Inst. of Fuel*, 106–110 (March 1960).
5. W. D. Snowden, R. T. Shigehara, "Non-proportional sampling biases on concentration and pollutant mass rate," Unpublished paper (1969).
6. H. H. Watson, "Errors due to anisokinetic sampling of aerosols," *Am. Ind. Hyg. Ass. Quart.* 15–16, 21–25 (March 1954).
7. R. T. Shigehara, W. F. Todd, and W. S. Smith, "Significance of errors in stack sampling measurements," Presented at APCA Annual Meeting, St. Louis, Missouri (June 1970).

SLIDE 153-0

NOTES

ISOKINETIC SAMPLING AND BIAS FROM NONISOKINETIC SAMPLING

SLIDE 153-1

ISOKINETIC SAMPLING

1. The velocity entering the nozzle must be the same as the velocity in the stack.
2. The particles must be moving parallel to the stack wall and directly toward the nozzle.

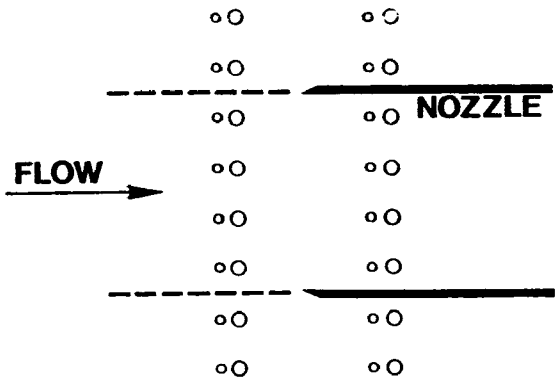
SLIDE 153-2

EXAMPLE

- o — Large particles. These particles are greater than 100 microns and are not affected by flow pattern due to their inertia.
- o — Small particles. These particles are less than 0.3 microns and move along with the flow pattern since they have very little inertia.

SLIDE 153-3

NOTES



100% ISOKINETIC

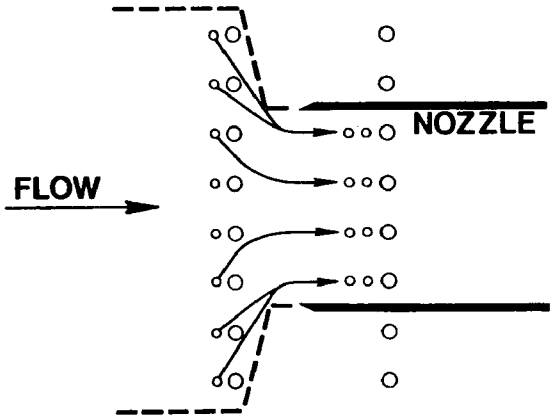
$v_n = v_s$

m_n = 8 grains/min

Q_n = 1 cfm

c_n = 8/1 = 8 grains/cf

SLIDE 153-4



200% ISOKINETIC

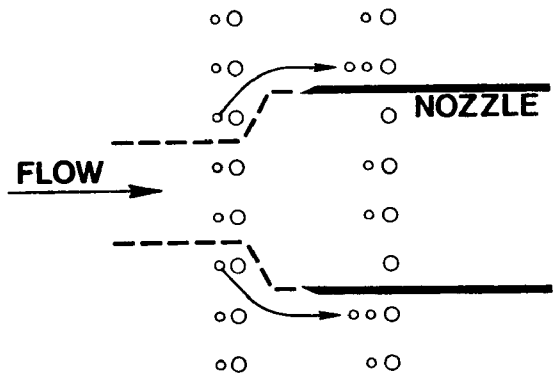
$v_n = 2v_s$

m_n = 12 grains/min

Q_n = 2 cfm

c_n = 12/2 = 6 grains/cf

SLIDE 153-5



50% ISOKINETIC

$v_n = 1/2\ v_s$

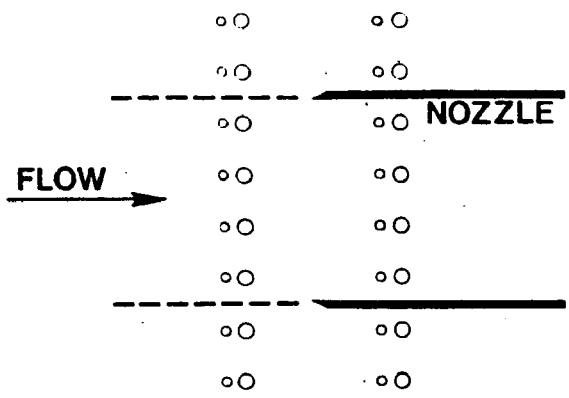
m_n = 6 grains/min

Q_n = 0.5 cfm

c_n = 6/0.5 = 12 grains/cf

SLIDE 153-3

NOTES



100% ISOKINETIC

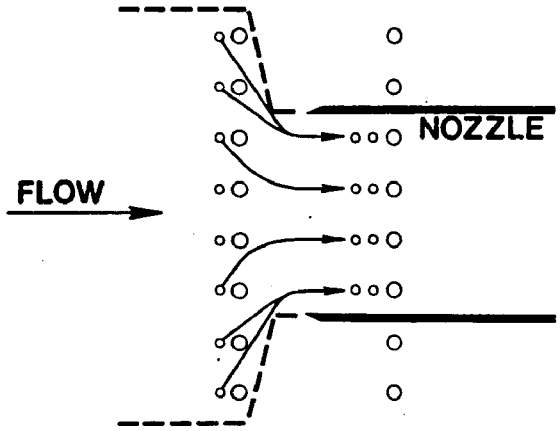
$v_n = v_s$

m_n = 8 grains/min

Q_n = 1 cfm

c_n = 8/1 = 8 grains/cf

SLIDE 153-4



200% ISOKINETIC

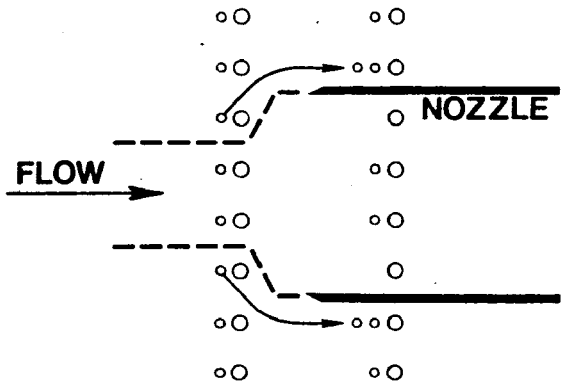
$v_n = 2v_s$

m_n = 12 grains/min

Q_n = 2 cfm

c_n = 12/2 = 6 grains/cf

SLIDE 153-5



50% ISOKINETIC

$v_n = 1/2\ v_s$

m_n = 6 grains/min

Q_n = 0.5 cfm

c_n = 6/0.5 = 12 grains/cf

SLIDE 153-6

NOTES

RESULTS

100% Isokinetic — 8 gr/ft³

200% Isokinetic — 6 gr/ft³

50% Isokinetic — 12 gr/ft³

SLIDE 153-7

POLLUTANT MASS RATE EQUATIONS

$$PMR_c = \frac{M_n}{V_{m_{std}}} v_{s_{std}} As$$

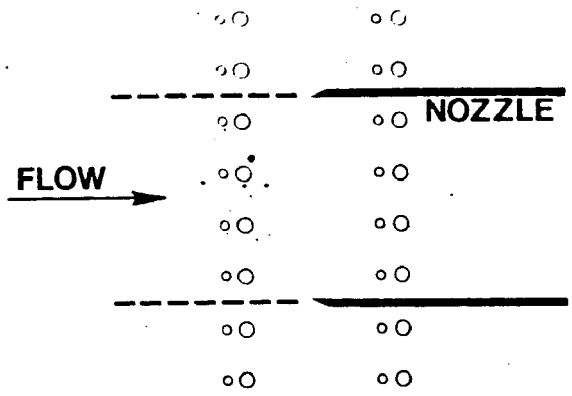
$$PMR_a = \frac{M_n}{\theta} \frac{As}{An}$$

Whereas

- PMR_c — pollutant mass rate calculated on the concentration basis
- m_n — mass collected on the filter and probe
- $V_{m_{std}}$ — volume metered by the sample train at standard conditions
- $v_{s_{std}}$ — velocity of the stack at standard conditions
- As — area of the stack
- PMR_a — pollutant mass rate calculated on the ratio of areas basis
- θ — time tested
- An — area of the nozzle

SLIDE 153-3

NOTES



100% ISOKINETIC

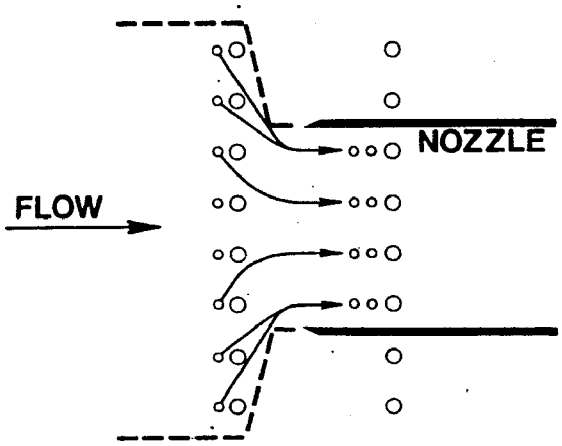
$v_n = v_s$

m_n = 8 grains/min

Q_n = 1 cfm

c_n = 8/1 = 8 grains/cf

SLIDE 153-4



200% ISOKINETIC

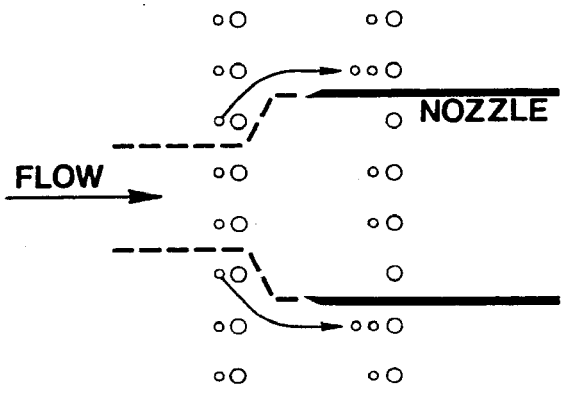
$v_n = 2v_s$

m_n = 12 grains/min

Q_n = 2 cfm

c_n = 12/2 = 6 grains/cf

SLIDE 153-5



50% ISOKINETIC

$v_n = 1/2\ v_s$

m_n = 6 grains/min

Q_n = 0.5 cfm

c_n = 6/0.5 = 12 grains/cf

SLIDE 153-8

NOTES

SIMPLIFIED MASS RATE EQUATION

The equations below have all constant values removed.

$$PMR_c = \frac{M_n}{V_m}$$

$$PMR_a = M_n$$

$V_{s_{std}}$, As, θ, and An are all constant

Note: The pollutant mass rate on the concentration basis (PMR_c) is a result of both the mass and sample volume collected. The pollutant mass rate on the area basis is dependent on only mass.

SLIDE 153-9

RESULTS OF PMR_c FOR SMALL PARTICLES

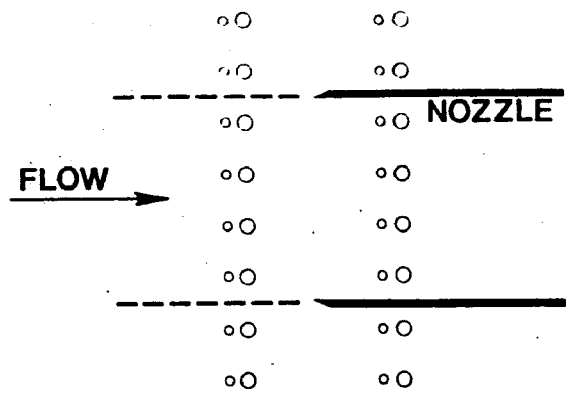
100% Isokinetic – $C = \frac{4}{1} = 4$ gr/ft³

200% Isokinetic – $C = \frac{8}{2} = 4$ gr/ft³

50% Isokinetic – $C = \frac{2}{0.5} = 4$ gr/ft³

SLIDE 153-3

NOTES



100% ISOKINETIC

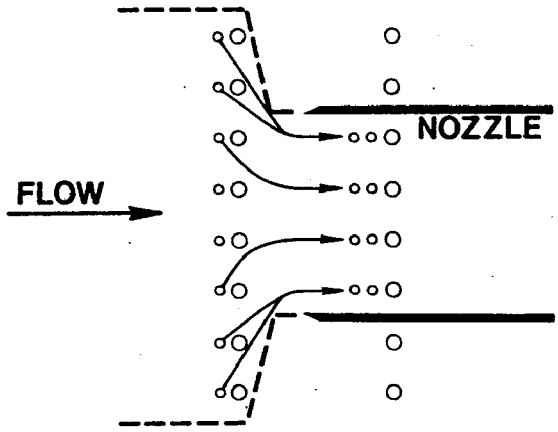
$v_n = v_s$

m_n = 8 grains/min

Q_n = 1 cfm

c_n = 8/1 = 8 grains/cf

SLIDE 153-4



200% ISOKINETIC

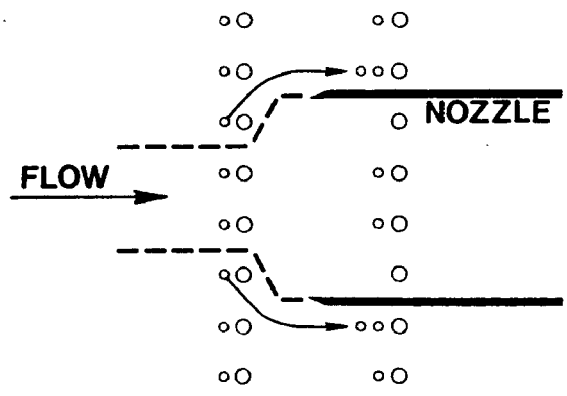
$v_n = 2v_s$

m_n = 12 grains/min

Q_n = 2 cfm

c_n = 12/2 = 6 grains/cf

SLIDE 153-5



50% ISOKINETIC

$v_n = 1/2\ v_s$

m_n = 6 grains/min

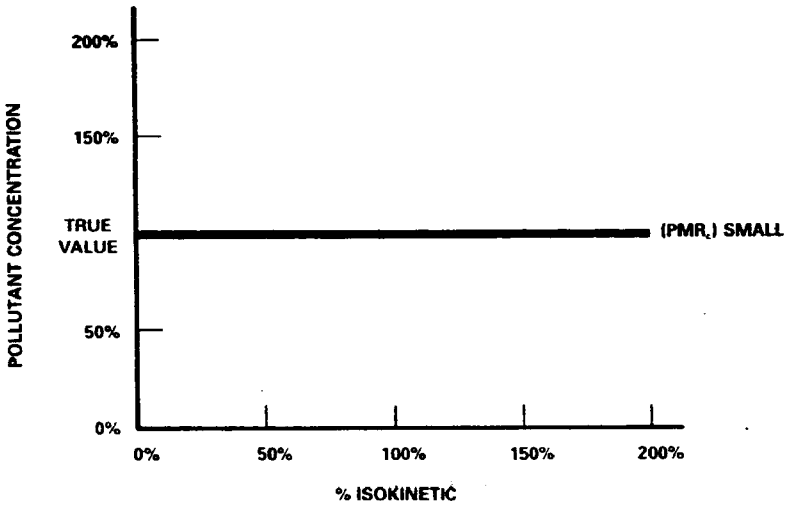
Q_n = 0.5 cfm

c_n = 6/0.5 = 12 grains/cf

SLIDE 153-10

NOTES

PLOT OF % ISOKINETIC VS. POLLUTANT CONCENTRATION



SLIDE 153-11

RESULTS OF PMR_c FOR LARGE PARTICLES

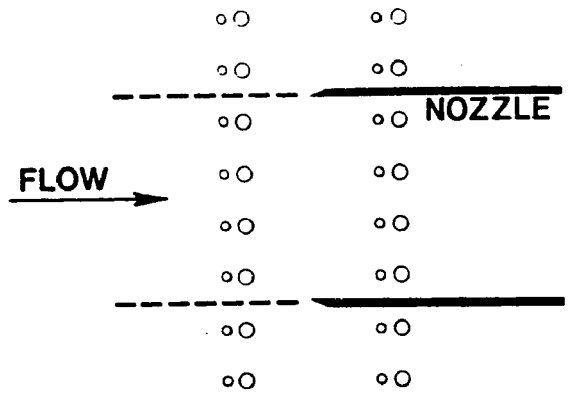
100% Isokinetic – $C = \frac{4}{1} = 4$ gr/ft^3

200% Isokinetic – $C = \frac{4}{2} = 2$ gr/ft^3

50% Isokinetic – $C = \frac{4}{0.5} = 8$ gr/ft^3

SLIDE 153-3

NOTES



100% ISOKINETIC

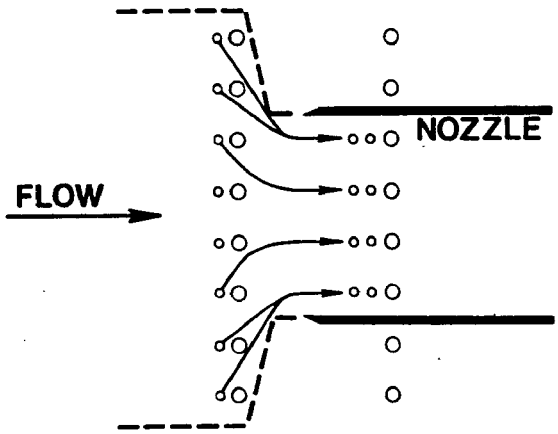
$v_n = v_s$

m_n = 8 grains/min

Q_n = 1 cfm

c_n = 8/1 = 8 grains/cf

SLIDE 153-4



200% ISOKINETIC

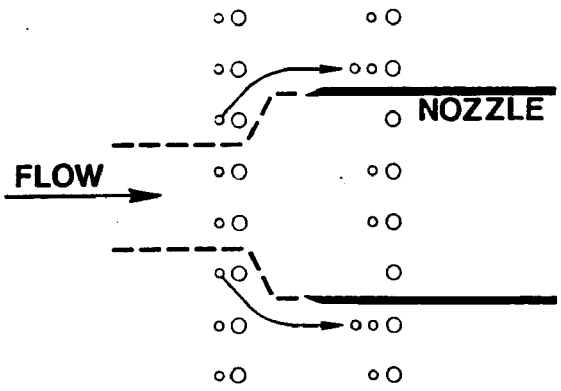
$v_n = 2v_s$

m_n = 12 grains/min

Q_n = 2 cfm

c_n = 12/2 = 6 grains/cf

SLIDE 153-5



50% ISOKINETIC

$v_n = 1/2\ v_s$

m_n = 6 grains/min

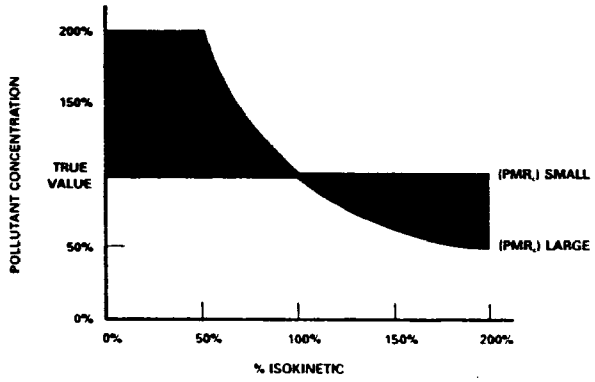
Q_n = 0.5 cfm

c_n = 6/0.5 = 12 grains/cf

SLIDE 153-12

NOTES

PLOT OF % ISOKINETIC VS. POLLUTANT CONCENTRATION



NOTE:

Since no stack contains either all large or all small particles, the bias will be in the colored area.

SLIDE 153-13

RESULTS OF PMR_a FOR SMALL PARTICLES

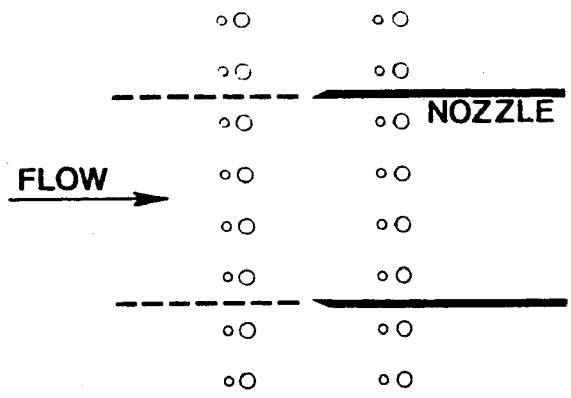
100% Isokinetic — 4 gr

200% Isokinetic — 8 gr

50% Isokinetic — 2 gr

SLIDE 153-3

NOTES



100% ISOKINETIC

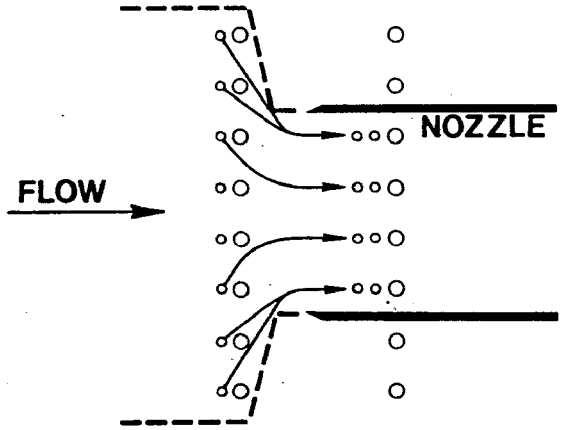
$v_n = v_s$

m_n = 8 grains/min

Q_n = 1 cfm

c_n = 8/1 = 8 grains/cf

SLIDE 153-4



200% ISOKINETIC

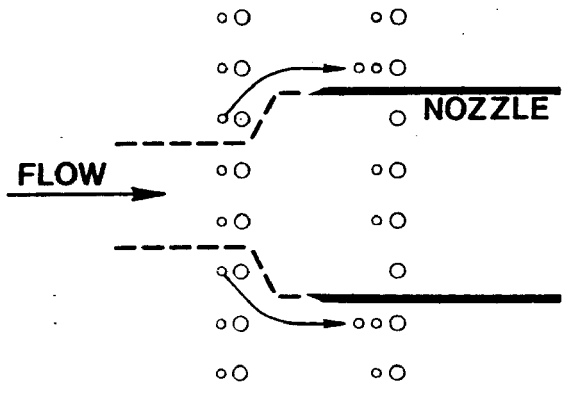
$v_n = 2v_s$

m_n = 12 grains/min

Q_n = 2 cfm

c_n = 12/2 = 6 grains/cf

SLIDE 153-5



50% ISOKINETIC

$v_n = 1/2\ v_s$

m_n = 6 grains/min

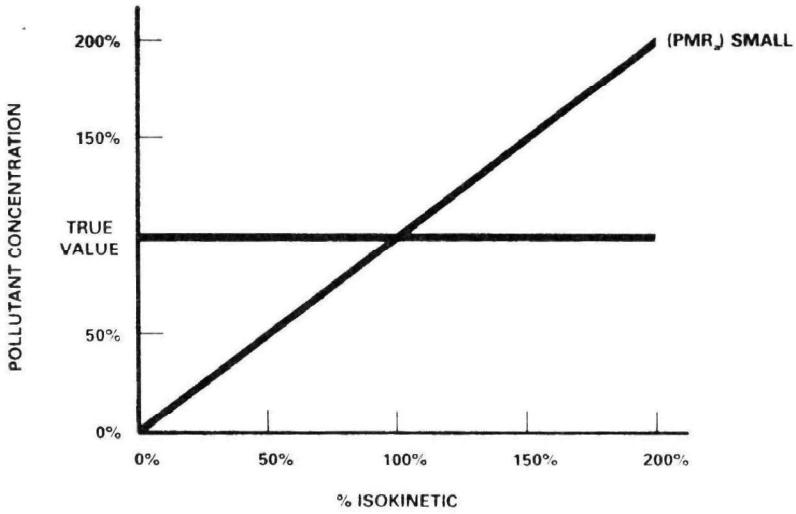
Q_n = 0.5 cfm

c_n = 6/0.5 = 12 grains/cf

SLIDE 153-14

NOTES

PLOT OF % ISOKINETIC VS. POLLUTANT CONCENTRATION



SLIDE 153-15

RESULTS OF PMR_a FOR LARGE PARTICLES

100% Isokinetic — 4 gr

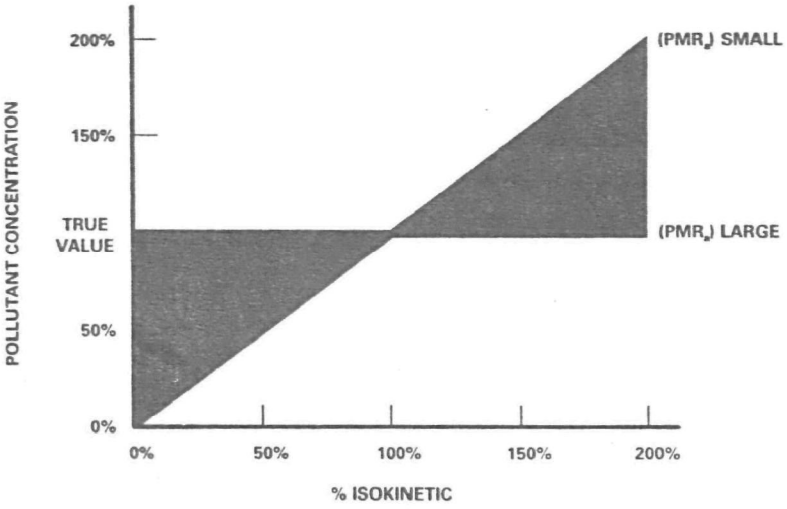
200% Isokinetic — 4 gr

50% Isokinetic — 4 gr

SLIDE 153-16

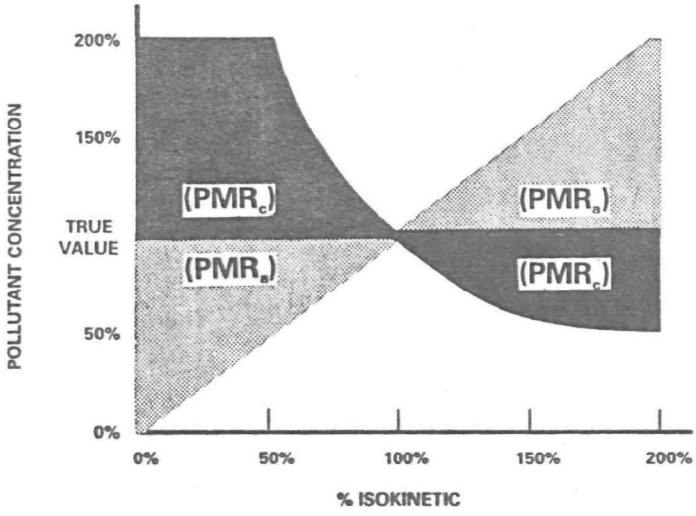
NOTES

PLOT OF % ISOKINETIC VS. POLLUTANT CONCENTRATION



SLIDE 153-17

PLOT OF % ISOKINETIC VS. POLLUTANT CONCENTRATION



SLIDE 153-18

NOTES

The pollutant concentration can be calculated by either equation; however, the EPA chose to use PMR_c since most particles for new sources are small and PMR_c gives the true value for small particles. The proposed regulations had the tester calculate the results by both methods and average the results.

SLIDE 153-19

Conclusion

$$PMR_a = \frac{\%\ Isokinetic}{100} \times PMR_c$$

SECTION N. PRECISION AND ACCURACY OF TEST METHODS

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Research Triangle Park, N.C. 27711

DATE: October 10, 1980

SUBJECT: Information to Support Data Quality Acceptance Criteria for Performance Audits and Routine Monitoring

FROM: Dr. Thomas R. Hauser, Director
Environmental Monitoring Systems Laboratory, RTP (MD-75)

TO: Dr. Courtney Riordan
Deputy Assistant Administrator
for Monitoring and Technical Support (RD-680)

Your memo of September 22, 1980, has generated a large amount of data gathering and documentation. I have divided it into two sections, ambient and source. I hope it is some help to you.

Enclosure

cc: D.J. von Lehmden
M.R. Midgett
J.C. Puzak
L.J. Purdue
T.A. Clark
F.J. Burmann

Provided here in Table A-3 are the estimates of precision and accuracy for those source emission test methods that the Source Branch of the Quality Assurance Division has either collaboratively tested or subjected to a multi-sample, single-laboratory evaluation. Because of the great expense involved in the collaborative testing of source emission methods, such tests are not normally performed unless they are justified by the particular situation. For example, a method for a pollutant of critical importance, and that will incur widespread usage, might warrant collaborative testing whereas one that would be used intermittently for a few industries would not. Where collaborative testing is not justified and/or budgeting restrictions do not allow the expense, a multi-sample, single-laboratory evaluation is routinely conducted during the development and evaluation of the test method to obtain within-laboratory precision estimates. The estimates in Table A-3 reflect this; there is no between-laboratory standard deviation shown for those methods that have not been collaboratively tested.

Because the true pollutant concentration of a stack gas is unknown and constantly changing, estimates of method accuracy are particularly difficult to obtain. These estimates are frequently obtained from analysis of standard cylinder gases, analysis of reference materials that test the accuracy of the analytical procedure only, or comparison with another Reference method or instrument to establish relative accuracy.

Table A-4 provides a listing of source emission methods for which precision and accuracy data are not available.

TABLE A-3. SOURCE EMISSION METHODS FOR WHICH PRECISION/ACCURACY DATA EXISTS BASED ON COLLABORATIVE TESTS OR SINGLE-LABORATORY EVALUATIONS

EPA Method	Description/Application	Condition of test	Standard Deviation Within Lab	Standard Deviation Between Lab	Accuracy
2	Velocity	Real sample,	3.9% of flow	5.0% of flow	Accurate within limits
	Volumetric Flow	Multi-laboratory	5.5% of flow	5.6% of flow	of method precision
3	CO_2 (manual)	" "	0.2%	0.4%	" " "
	O_2 (manual)	" "	0.3%	0.6%	" " "
	Molecular Weight	" "	0.35 g/g mole	0.048 g/g mole	" " "
5	Particulate Emission	" "	10.4% of conc.	12.1% of conc.	Not determinable
	Stack Moisture Content	" "	0.1%	0.1%	Within limits of method precision
6	SO_2-Power Plant	" "	4.0% of conc.	5.8% of conc.	Accurate within limits of method precision
7	NO_x-Nitric Acid	" "	14.9% of conc.	18.5% of conc.	" " "
	NO_x-Power Plant	" "	6.6% of conc.	9.5% of conc.	" " "
8	SO_2-Sulfuric Acid Plant	" "	8.0 mg/m^3	11.2 mg/m^3	" " "
	H_2SO_4-Sulfuric Acid Plant	" "	2.7 mg/m^3	3.0 mg/m^3	" " "
9	Stack Gas Opacity	" "	2.0% of opacity	2.0% of opacity	5% opacity at level of standard
10	CO-Refinery FCC	" "	13 ppm	25 ppm	$\leq$ 24 ppm
11	H_2S-Refinery Fuel Gas	Simulated sample, Multi-laboratory	2.1% of conc.	4.5% of conc.	4% at level of standard
12	Pb	Real sample, Single laboratory	5% of conc.	—	Accurate within limits of method precision

----CONTINUED----

TABLE A-3. (continued)

EPA Method	Description/Application	Condition of test	Standard Deviation Within Lab	Standard Deviation Between Lab	Accuracy
13A	F^- by SPADNS Analysis	Real sample, Multi-laboratory	0.044 mg/m^3	.064 mg/m^3	-.08 mg/l
13B	F^- by SIE Analysis	" "	0.037 mg/m^3	0.056 mg/m^3	-.10 mg/l
15	H_2S, COS, CS_2 Sulfur Recovery	Real sample, Single laboratory	8% of conc.	—	10% at level of standard
16	Total Reduced Sulfur - Kraft	" "	8% of conc.	—	10% at level of standard
17	Particulate	" "	6% of conc.	—	Not determinable
23	Chlorinated Hydrocarbons	" "	3% of conc.	—	-3%
24	Volatile Organics from Paint	" "	8% Water Based Paint 0.5% Solvent Based Paint	— —	Not determined
101/102	Hg/Chlor-Alkali Plants[a]	Real samples, Multi-laboratory	1.6 µg/ml	1.8 µg/ml	-0.4 µg/ml
104	Be	" "	0.4 µg/m^3	0.6 µg/m^3	-0.13 µg/m^3
105	Hg in Sewage Sludge	Real samples, Single laboratory	0.2 µg/g	—	Accurate within precision of method
106	Vinyl Chloride	Real samples, Multi-laboratory	2.5% of conc.	6.3% of conc.	-2% at level of standard
110	Benzene	Real samples, Single laboratory	9% of conc.	—	+3.1% at level of standard
111	Hg in Sludge Incinerator Stacks	Real samples, Single laboratory	4.8 µg/m^3	—	Unknown

[a]Precision for analytical portion only.

TABLE A-4. SOURCE EMISSION METHODS FOR WHICH PRECISION AND ACCURACY DATA ARE NOT AVAILABLE

EPA Method	Application	Present Status	Comments on Precision/Accuracy
1	Sampling point selection	Promulgated	Precision/accuracy not applicable
4	Estimate of stack moisture	Promulgated	Expected to be similar to Method 5 moisture determination
14	Design of Al Plant Roof Monitoring System	Promulgated	Precision/accuracy not applicable
18	NH_4 NO_3 emissions from fertilizer plants	Future Proposal	Method under development
19	Calculation of F factor	Promulgated	Precision/accuracy not applicable
20	NO_x, SO_2 from gas turbines	Proposed	
21	Leak test method for valves and pumps in organic processes	Future Proposal	Precision/accuracy not applicable
22	Fugitive emissions estimate by visual observation	Future Proposal	
25	Non-methane organic emissions	Proposed	Method being evaluated
26	Organic emissions from asphalt processing	Future Proposal	Method under development
27	Reserved	—	
28	Urea emissions from fertilizer plants	Proposed	
29	Volatile organic contents of printing ink	Future Proposal	Method under development
103	Screening method for Be	Promulgated	Precision/accuracy expected to be similar to Method 104
107	Vinyl chloride monomer in resin	Promulgated	
108	As emissions from smelters	Future Proposal	Method under development
109	Visual emissions from coke ovens	Future Proposal	Method under development

ERROR ANALYSIS*

Introduction

The problem of accuracy in stack sampling measurements is considered and debated in almost every report or journal article in which stack sampling data appear. There exists, however, a great deal of misunderstanding in the engineering community on the difference between error, precision, and accuracy. This misunderstanding often leads to a misinterpretation of analytical studies of stack sampling methods. The type of error analysis often used applies only to "randomly distributed error with a normal distribution about the true value."

A discussion of the definitions of terms normally used in error analysis will be given in a course lecture. The definitions are also included in this manual for your future reference. It is hoped that by studying this section the student will realize the limitations of error analysis procedures and will be able to more carefully design experiments that will yield results close to the "true" value.

Definitions

Error: This word is used correctly with two different meanings (and frequently incorrectly to denote what properly should be called a "discrepancy"):

(1) To denote the difference between measured value and the "true" one. Except in a few trivial cases (such as the experimental determination of the ratio of the circumference to the diameter of a circle), the "true" value is unknown and the magnitude of the error is hypothetical. Nevertheless, this is a useful concept for the purpose of discussion.

(2) When a number such as $\sigma = \pm 0.000008 \times 10^{10}$ is given or implied, "error" refers to the estimated uncertainty in an experiment and is expressed in terms of such quantities as standard deviation, average deviation, probable error, or precision index.

*Adapted from Y. Beers, Theory of Errors, Addison-Wesley, Reading, Mass, (1958) pp. 1-6.

Discrepancy: This is the difference between two measured values of a quantity, such as the difference between those obtained by two students, or the difference between the value found by a student and the one given in a handbook or textbook. The word "error" is often used incorrectly to refer to such differences.

Many beginning students suffer from the false impression that values found in handbooks or textbooks are "exact" or "true." All such values are the results of experiments and contain uncertainties. Furthermore, in experiments such as the determination of properties of individual samples of matter, handbook values may actually be less reliable than the student's because the student's samples may differ in constitution from the materials which were the basis of the handbook values.

Random Errors: Sometimes when a given measurement is repeated the resulting values do not agree exactly. The causes of the disagreement between the individual values must also be causes of their differing from the "true" value. Errors resulting from these causes are called random errors. They are also sometimes called experimental or accidental errors.

Systematic or Constant Errors: If, on the other hand, all of the individual values are in error by the same amount, the errors are called systematic or constant errors. For example, all the measurements made with a steel tape that includes a kink will appear to be too small by an amount equal to the loss in length resulting from the kink.

In most experiments, both random and systematic errors are present. Sometimes both may arise from the same source.

Determinate and Indeterminate Errors: Errors which may be evaluated by some logical procedure, either theoretical or experimental, are called determinate, while others are called indeterminate.

Random errors are determinate because they may be evaluated by application of a theory that will be developed later. In some cases random or systematic errors may be evaluated by subsidiary experiments. In other cases it may be inherently impossible to evaluate systematic errors, and their presence may be inferred only indirectly by comparison with other measurements of the same quantity employing radically different methods. Systematic errors may sometimes be evaluated by calibration of the instruments against standards, and in these cases whether the errors are determinate or indeterminate depends upon the availability of the standards.

Corrections: Determinate systematic errors and some determinate random errors may be removed by application of suitable corrections. For example, the measurements that were in error due to a kink in a steel tape may be eliminated by comparing the tape with a standard and subtracting the difference from all the measured values. Some of the random error of this tape may be due to expansion and contraction of the tape with fluctuations of temperature. By noting the temperature at the time of each measurement and ascertaining the coefficient of linear expansion of the tape, the individual values may be compensated for this effect.

Precision: If an experiment has small random errors, it is said to have high precision.

Accuracy: If an experiment has small systematic errors, it is said to have high accuracy.

Adjustment of Data: This is the process of determining the "best" or what is generally called the most probable value from the data. If the length of a table is measured a number of times by the same method, by taking the average of the measurements we can obtain a value more precise than others, then a weighted average should be computed. These are examples of adjustment of data for directly measured quantities. For computer quantities the process may be specialized and complicated.

Classification of Errors

Systematic Errors:

(1) Errors of calibration of instruments.

(2) Personal errors. These are errors caused by habits of individual observers. For example, an observer may always introduce an error by consistently holding his head too far to the left while reading a needle and scale having parallax.

(3) Experimental conditions. If an instrument is used under constant experimental conditions (such as of pressure or temperature) different from those for which it was calibrated, and if no correction is made, a systematic error results.

(4) Imperfect technique. The measurement of viscosity by Poiseuille's Law requires the measurement of the amount of liquid emerging from an apparatus in a given time. If a small amount of the liquid splashes out of the vessel which is used to catch it, a systematic error results.

Random Errors:

(1) Errors of judgment. Most instruments require an estimate of the fraction of the smallest division, and the observer's estimate may vary from time to time for a variety of reasons.

(2) Fluctuating conditions (such as temperature, pressure, line voltage).

(3) Small disturbances. Examples of these are mechanical vibrations or, in electrical instruments, the pickup of spurious signals from nearby rotating electrical machinery or other apparatus.

(4) Definition. Even is the measuring process were perfect, repeated measurements of the same quantity might still fail to agree because that quantity might not be precisely defined. For example, the "length" of a rectangular table is not an exact quantity. For a variety of reasons the edges are not smooth (at least if viewed under high magnification) nor are the edges accurately parallel. Thus even with a perfectly accurate device for measuring length, the value is found to vary depending upon just where on the cross section the "length" is measured.

Illegitimate Errors: These errors are almost always present, at least to a small degree, in the very best of experiments and they should be discussed in a written report. However, there are three types of avoidable errors which have no place in an experiment, and the trained reader of a report is justified in assuming that these are not present.

(1) Blunders. These are errors caused by outright mistakes in reading instruments, adjusting the conditions of the experiment, or performing calculations. These may be largely eliminated by care and by repetition of the experiment and calculations.

(2) Errors of computation. The mathematical machinery selected for calculating the results of an experiment (such as slide rules, logarithm tables, adding machines) should have errors small enough to be completely negligible in comparison with the natural errors of the experiment. Thus if the data are accurate to five significant figures, it is highly improper to use a slide rule capable of being read to only three figures, and then to state in the report that "slide rule error" is a source of error. Such a slide rule should be used for calculating the results of an experiment having only three or preferably two significant figures. On the other hand, if the experiment does give five significant figures, five or six-place logarithm talbes or some other more accurate means of calculation should be used.

(2) Chaotic Errors. If the effects of disturbances become unreasonably large - that is, large compared with the natural random errors - they are called chaotic errors. In such situations are experiment should be discontinued until the source of the disturbance is removed.

SLIDE 154-0

NOTES

PRECISION AND ACCURACY OF EPA REFERENCE METHODS

SLIDE 154-1

PRECISION AND ACCURACY METHODS EVACUATION

- REAL SAMPLE (Multi-Laboratory)
- SIMULATED SAMPLE (Multi-Laboratory)
- REAL SAMPLE (Single Laboratory)

SLIDE 154-2

REAL SAMPLE (Multi-Laboratory)

Approximately 10 different testing laboratories test the same source simultaneously. This is usually referred to as a "collaborative test." This testing is expensive and testing parameters are not usually varied.

SLIDE 154-3

NOTES

SIMULATED SAMPLE
(Multi-Laboratory)

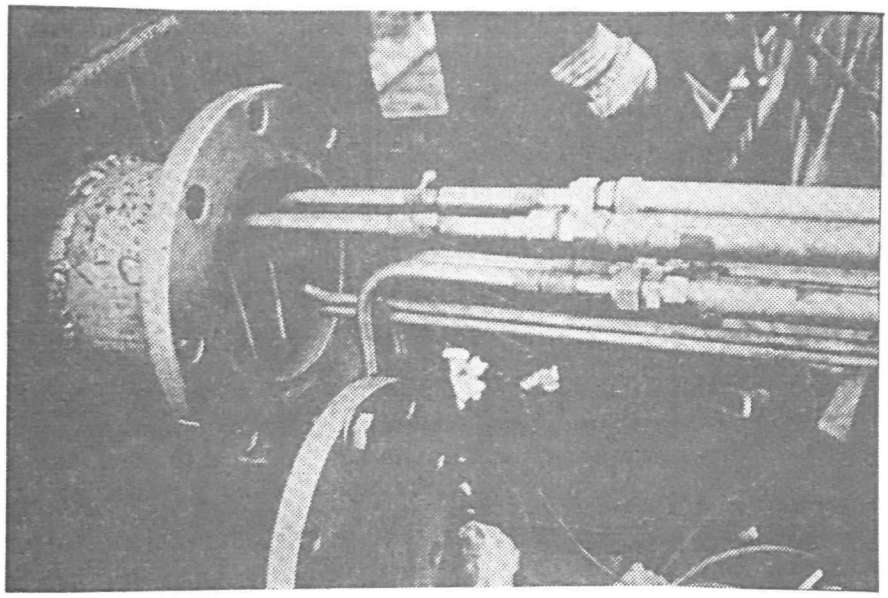
Approximately 10 different testing laboratories test a common manifold containing a known concentration of pollutant. This testing is expensive but the pollutant concentration is known and can be varied.

SLIDE 154-4

REAL SAMPLE
(Single Laboratory)

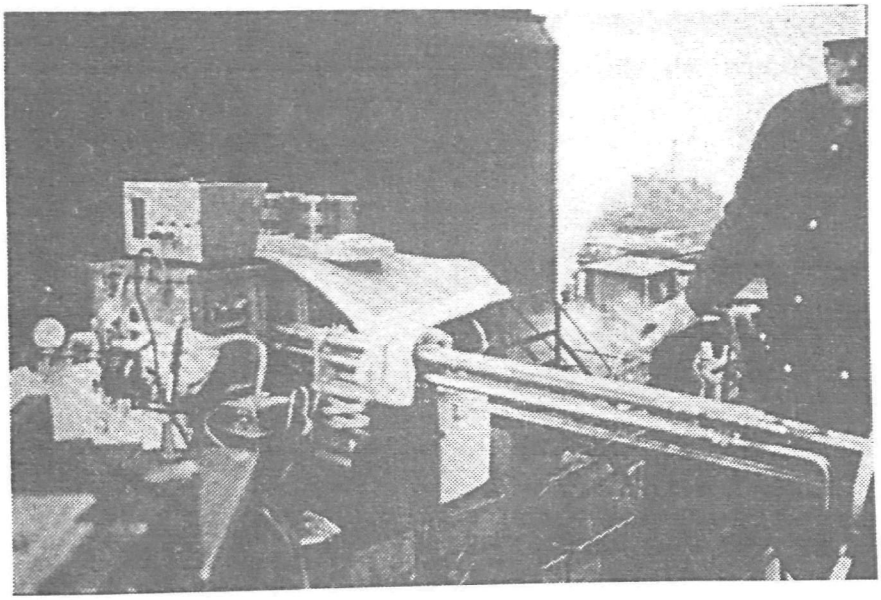
A single laboratory utilizes a "methods evaluation" train. This train collects a minimum of four samples for a single point. This testing is less expensive and any sampling parameters can be varied to determine its effect.

SLIDE 154-5



SLIDE 154-6

NOTES

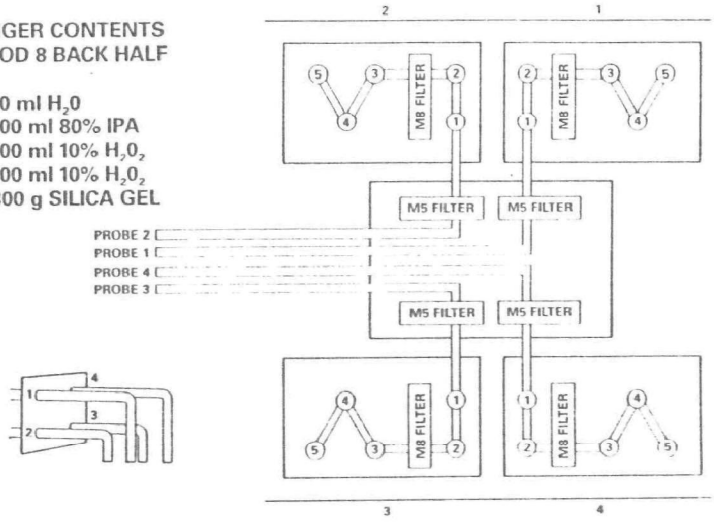


SLIDE 154-7

QUAD TRAIN CONFIGURATION

IMPINGER CONTENTS
METHOD 8 BACK HALF

1 — 50 ml H_2O
2 — 100 ml 80% IPA
3 — 100 ml 10% H_2O_2
4 — 100 ml 10% H_2O_2
5 — 300 g SILICA GEL



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NOTES

EPA METHOD	DESCRIPTION	STANDARD DEVIATION	
		Within Lab	Between Lab
2	Velocity	3.9%	5.0%
	Volumetric Flow	5.5%	5.6%
3	CO_2 (manual)	0.2%	0.4%
	O_2 (manual)	0.3%	0.6%
	Molecular Weight	0.35 g/g mole	0.48 g/g mole

SLIDE 154-9

EPA METHOD	DESCRIPTION	STANDARD DEVIATION	
		Within Lab	Between Lab
5	Particulate	10.4%	12.1%
6	SO_2 - Power Plant	4.0%	5.8%
7	NO_x - Power Plant	6.6%	9.5%

SECTION O. SIGNIFICANCE OF ERROR FOR SOURCE TEST OBSERVERS

USE OF SIGNIFICANCE OF ERROR FOR SOURCE TEST OBSERVER'S DECISIONS

by

Bill DeWees

The addition of numerous test methods, requirements, quality assurance procedures, allowable options by the tester, alternative methods and published articles, has made the average agency performance tester and/or observer all ask the same question. What is really important? If there were an easy answer, the question would not have to be asked. The only easy answer is the standard military or politician's answer: It varies with the conditions. Unfortunately, this is the correct answer.

This paper is designed to provide the necessary information and concepts to allow the agency staff to determine what is important as the conditions vary.

The tester or observer needs to know three things to determine what is important.

1) What is the data to be used for, (i.e., proof of compliance, proof of violation or engineering evaluation)?
2) What are the direction and magnitude of any biases?
3) What is the acceptable bias that will be allowed before rejecting the results?

Let's look at the three uses of the data and explore what biases would be acceptable. First, the source is attempting to prove itself in compliance with the regulation. If the test results conclusively show compliance with the regulation, any magnitude of bias in the data that increased the measured results (high bias) could be accepted for proof of compliance. These results may not, however, be valid for future use in emissions trading or banking. An acceptable low bias could be any amount that does not change what would be violation into compliance.

Since the final results are not known during the on-site testing, the observer generally would rather have a fixed value to allow or disallow the test run while on site. Past collaborative tests have shown most of the NSPS reference methods to have a reproducibility of about ±10 percent. Therefore, when the source is trying to demonstrate compliance, a good rule of thumb for allowing biases determined on-site may be less than 10 percent high bias and 5 percent low bias. Setting more stringent criteria than these for each sample run could be costly in time and money for both the industry and agency.

The second case is when the agency is trying to prove that the source is in violation with the applicable regulation. Any test data that prove conclusively that the source is in violation could contain any magnitude of low bias. The low bias will not be challenged by the industry unless the testing does not comply with the legal requirements as stated by the reference method. When the agency bears the proof of violation, a good rule of thumb for acceptable bias would be up to a 5 percent high bias and down to a 10 percent low bias. Again, let me stress that for proof of violation, meeting the requirements of the reference method is manadatory.

The final example is data used for engineering evaluation. Any bias may be acceptable in this case as long as it is quantifiable. The data can then be adjusted or a confidence limit will be placed on it.

Both on-site and posttest data analysis will be made by the agency. The agency observer should be aware of his authority and acceptable biases on-site. It is the belief of the author that an agency observer that is not allowed to make decisions on-site, does not have the right to observe the test and later reject data based on his on-site observations. Rejection of a testing series is both time and cost consuming for the agency and industry. The agency should not compromise its standards but should make every effort both on-site and in posttest analysis to obtain test results acceptable for their purpose.

Error from the measurement of most of the sampling parameters have very little effect on the final data results. Table 1 was designed to provide a useful tool in estimating the magnitude and direction of biases from measurement errors of most sampling parameters. The parameters are shown separately but can be added together when more than one parameter has been erroneously measured. The table was developed using the testing values in the expected average range of testing. Also, it was assumed that a 2 percent isokinetic

error makes a 1 percent error in sample concentration. These values, although not always exact, should be good enough to make the required on-site or post-test decisions.

To better explain the table, let's use three examples.

EXAMPLE 1

The NSPS test is on an asphalt plant which has a concentration standard of 0.04 g/scf. It has been discovered that the test team incorrectly measured the stack temperature. The test team measured value was about 350°F, but the correct reading should be about 320°F. The question is how much error is there from a 30°F high measurement? Table 1 shows that for ts, a 10°F error will cause a -0.4 percent bias for a concentration measurement. Therefore, a +30°F error would cause a -1.2 percent bias in the concentration measurement. The 30°F error does not meet the requirements of $\pm$1.5 percent of the absolute temperature, however, the error would have little effect on the final data results and could therefore be allowed. The -1.2 percent bias means that the value that will be shown in the source test report will be 1.2 percent less than the value that should have been measured. This bias does aid the source in trying to show compliance, but the error is so small, it is almost meaningless.

EXAMPLE 2

The test is on a dog food fixing tank. The allowable mass emission rate standard is about 5 lb/h. The calibration factor of the dry gas meter has been checked with two critical orifices supplied by EPA. The results of the check shows the Y factor should be 0.91 but the tester reports a Y factor of 0.97. The error is 0.06, which should be multiplied by the error in the table for Y at 0.01 which causes a +1 percent. You inform the tester that the data will likely be biased high by 6 percent. You decide at this point to allow the tester to proceed as long as he assures you that a good posttest calibration will be made for the dry gas meter.

At the conclusion of the first run, the posttest leak-check indicated a leak rate of 0.15 cfm. This is well above the allowable, but the tester said he will just subtract the leak rate from the final sample volume as

shown in the reference method. Since the rate of sampling was only about 0.6 cfm, this could potentially cause a 25 percent high bias. You should suggest that the run be invalidated and that another run be made. The tester informs you he has the right to use the procedures that could cause up to a 25 percent high bias. You should then inform him that you have an obligation to protect the source as well as the general public and proceed to inform the facility contact of the consequences that the tester has decided to impose on the source.

EXAMPLE 3

The test is at a Subpart D power plant. The concentration standard is 0.1 lb/10^6 Btu. You are familiar with the testing crew and they usually do a good job. This test happens to be on one of those days when nothing seems to go right.

You first spot that the pitot tube and nozzle are not the minimum distance of 3/4 of an inch apart. This is corrected by simply bending the pitot tube away from the nozzle. Next, you check the stack thermocouple and both dry gas meter thermometer readings against each other at ambient temperature. The stack thermometer reads 81^{o}F; the inlet dry gas thermometer reads 79^{o}F; and the outlet thermometer reads 63^{o}F. You inform the team of the discrepancy but they have no way of adjusting the thermometer. You inform them that a posttest calibration must be performed on both thermometers. To estimate what error might occur, you use Table 1 to determine the expected error from the average temperature of the meter (tm), being low by 10^{o}F. The table shows that 5^{o}F gives an error of -0.9, therefore, a -10^{o}F error would be +1.8 percent error. This means the measured value would 1.8 percent higher than the true value.

After the second run, the orsat results seem wrong. There must have been a leak or some procedure was in error. The tester knows that the O_2 value should have been about 4.5 percent; however, his results were 7.7 percent O_2. Going back to the table, a 1 percent error at 5 percent O_2 will cause a +6.7 percent error, therefore a 3.2 percent error causes a +21.4 percent error. Since the test team is a reputable one, the tester requests that the test be invalidated since there was an obvious error. The observer agrees and another run is performed.

When the observer receives the final report for review, he notes that the test team has the static pressure as +6.7 in. H_2O. The observer thought that the value should have been -6.7 in. H_2O; however, he was not sure. To see if this really makes any difference, he refers to Table 1 and finds that for Ps, a 2 in. H_2O error will cause a +0.1 percent change. His change from +6.7 to -6.7 in. H_2O is -13.2 in. H_2O, which would cause about a -0.6 percent error. The error is insignificant on the impact of the data, however, the observer may wish to call the source and determine the actual sign of the static pressure to enhance accuracy of data reporting.

OTHER ERRORS

In addition to the table, four other potential major errors are listed: 1) two leak check procedures, 2) nozzle-pitot tube spacing, and 3) calibration at the magnehelic.

Generally, the most critical period for noting errors by the observer and creating errors by the tester is during the sample recovery phase. The observer should be present, if possible, during the sample recovery phase. Both the probe rinse and used filter should be observed. The filter should have a clear space all around its border to show that no particulates have been circumvented. Generally, no particulate should exist that can be easily seen by the naked eye. No particles of that size should be able to get through the control device, but if they do, they would not be a health hazard as they would immediately fall to the ground after existing in the stack. Their presence usually means that foreign debris has been caught by the sample train. The color and appearance can also give information on process operation and failure of the sample box to maintain the proper temperature. A drastic color change for one of the runs may indicate a process malfunction which should be checked. Also, as discussed in the paper on the "Role of the Agency Observer", the apparent mass of particulate collected should be compared to the visible emissions during test.

CONCLUSION

There are numerous errors and nontypical conditions that can occur during each sample run. The observer should have a good understanding of the intent of the test and the magnitude of error that will be tolerated by the agency;

he should then proceed to make his decision in a logical manner, using all facts available at the time. When the observer is worried about not meeting the requirements of the reference method(s), usually a little creative thinking on the part of the observer and some posttest work by the test team can solve the problem. An example would be if the process shuts down with one point to go in a sample run, test the last point with the process off provided the fan is still running. If the fan shuts down, you have in fact sampled the last point at the correct rate (nothing). The posttest rejection of a test is costly in both time and money to the source and agency. If a sample run will likely be rejected, reject it on-site since this is far less costly and time consuming.

TABLE 1. ERROR ANALYSIS OF SAMPLING PARAMETERS

Parameter	True value	Error	Erroneous value	Concentration (%)[a]	Q_s	Qs_{std}	Pollutant mass rate (%)[b]	Requirement
P_{bar}	30 in. Hg	1 in. Hg	31 in. Hg	+ 4.1	-1.6	+1.6	+5.9	±0.1 in. Hg
ΔH	30 in. Hg	1 in. H_2O	30.07 in. Hg	+ 0.2	-[c]	-[c]	+0.2	none
tm	528°R	5°R	533°R	- 0.9	-[c]	-[c]	-0.9	±5.4°F
Ps	30 in. Hg	2 in. H_2O	30.15 in. Hg	+ 0.1	-0.2	+0.2	+0.3	±1.36 in. H_2O
ts	600°R	10°F	610°R	- 0.4	+0.8	-0.8	-1.2	±1.5%
Δp	1 in. H_2O	0.1 in. H_2O	1.1 in. H_2O	- 2.5	+4.9	+4.9	+2.4	equip. spec.
Moisture	10%	1%	11%	+ 0.1	-0.2	+1.3	+1.4	±1%
Md	29	1% CO_2	29.15	+ 0.1	-0.3	+0.3	+0.4	±0.1%
Md	29	1% O_2	29.03	+ 0.1	-0.1	+0.1	+0.2	±0.1%
Md	29	1% CO	29	-	-[c]	-[c]	-	±0.1%
Mn	100 mg	1 mg	101 mg	1.0	-[c]	-[c]	1.0	±0.5 mg
Time	120 min	1 min	121 min	- 0.4	-[c]	-[c]	-0.4	none
Y	1.00	0.01	1.01	1.0	-[c]	-[c]	1.0	±2%
Cp	0.84	0.01	0.85	- 0.6	+1.2	+1.2	+0.6	±0.03
Nozzle diameter	0.250 in.	0.005 in.	0.255 in.	- 2.0	-[c]	-[c]	-2.0	±0.002 in.
Isokinetic	100%	10%	110%	- 5.0[d]	-[c]	-[c]	-5.0	+10%
F factor	5% O_2	1% O_2	6% O_2	+ 6.7[d]	N/A[e]	N/A[e]	N/A[e]	±0.1% O_2
F factor	10% O_2	1% O_2	11% O_2	+10.1[d]	N/A[e]	N/A[e]	N/A[e]	±0.1% O_2
F factor	15% O_2	1% O_2	16% O_2	+20.4[d]	N/A[e]	N/A[e]	N/A[e]	±0.1% O_2

Note: All errors are a theoretical calculation and it is assumed that a 2% error in isokinetics will cause a 1% error in both concentration and pollutant mass rate emissions. The percentage of error may vary slightly depending on the magnitude of the true value and the different assumptions made.

[a]Error due to calculation and combined isokinetic bias.

[b]Error due to calculation and combined isokinetics bias.

[c]No effect on the measured value.

[d]Lb/10^6 Btu is actually a concentration standard.

[e]N/A - not applicable.

(continued)

TABLE 1 (continued)

- Leak Check of the Pitot Tube:

  A posttest leak check of the pitot tube must be performed at a minimum of 3 in. H_2O. It is not possible to posttest calculate the error from a pitot tube that does not leak check. However, if only one side of the pitot tube does not leak check, the test or observer may wish to determine the maximum error that could have occurred by the following procedures. Place the pitot tube back into the stack at a point of average velocity using the same orientation as in the test. Take the Δp reading. Turn the pitot tube over (180^0 reorientation). Again take the Δp reading. The difference in the two readings is the expected error in in. H_2O. If both sides of the pitot tube leak no error analysis is possible.

- Spacing of the Pitot Tube to the Nozzle:

  A minimum of a 3/4 in. spacing is required between the pitot tube and nozzle. If the nozzle-pitot tube spacing is only 1/2 in. an error of a 7 percent high bias in the flow rate may be caused. A lesser spacing can cause even greater high bias error.

- Calibration of the Magnehelic vs. the Inclined Manometers;

  The error would be the same as reported for Δp.

- Leak Check of the Sample Train:

  The train is to be posttest leak checked at the maximum vacuum operated during the test. If the maximum leak exceeds 0.02 cfm, the leak (leak rate times the sample time) is to be substacted from the sample volume. It is suggested when leak check at the nozzle exceeds the allowable leak rate, the leak check be performed at the nozzle 2 in. Hg (or the static pressure, whichever is higher). The train should then be leak checked at the inlet to the filter. The higher of the two leakage rates can then be subtracted from the sample volume. Both approaches could cause a high bias for both concentrations and pmr standards. The percent bias will be somewhere between the maximum (assuming there was no leakage during the test) and the minimum of zero (assuming the maximum measured leakage actually occurred during the test).

SLIDE 155-0

NOTES

SOURCE TEST OBSERVER'S USE OF SIGNIFICANCE OF ERROR

SLIDE 155-1

BASIS FOR OBSERVER'S DECISIONS

1. What is the pupose of test?
2. What is direction and magnitude of bias?
3. What is the acceptable allowed bias?

SLIDE 155-2

USE OF ERROR ANALYSIS TABLE

(From Table 1)

- Errors are calculated using values shown.
- It is assumed that a 2% error in the isokinetic sample rate results in a 1% error in concentration.
- When more than one error occurs, use the sum of the errors.

Note: The true value and erroneous measured value are shown to eliminate confusing the terms "measured high" and "measured low."

SLIDE 155-3

NOTES

EXAMPLE 1
For Concentration Standard

1. ERROR T_s: measured value is 30°F too high.
2. 10°F causes a -0.4% bias (from Table 1).
3. Therefore, +30°F will cause a -1.2% bias.

SLIDE 155-4

EXAMPLE 2
For Mass Emission Standard

PART 1

1. ERROR Y: reported value is 0.06 too high.
2. 0.01 causes a +1% bias (from Table 1).
3. Therefore, +0.06 will cause a +6% bias.

SLIDE 155-5

PART 2

1. ERROR: leak rate is 0.15 cfm.
2. Sample rate is $\approx$ 0.6 cfm.
3. Therefore, if total leak rate is subtracted, bias could be as high as +25%.

SLIDE 155-6

NOTES

EXAMPLE 3
For Concentration Standard

PART 1

1. ERROR: pitot tube is < ¾ in. from nozzle.
2. Table 1 shows that a ½ in. spacing causes a 7% bias.
3. Therefore, pitot tube must be bent until correct spacing is obtained.

SLIDE 155-7

PART 2

1. ERROR: oxygen reading is 3.2% too high.
2. At 5% O_2 a 1.0% O_2 error causes a +6.7% bias (from Table 1).
3. Therefore, a +3.2% O_2 error will cause a +21.4% bias.

SLIDE 155-8

PART 3

1. ERROR: Static pressure reading of +6.7 in. H_2O may have an incorrect sign.
2. A 2.0 H_2O error causes a 0.1% bias (from Table 1).
3. Therefore, sign should have been negative not positive. A -13.4 in. H_2O error will cause a -0.6% bias.

SLIDE 155-9

NOTES

OTHER ERRORS

- Pitot tube leak-check
- Pitot tube/nozzle spacing
- Magnehelic calibration
- Sample train leak-check

SLIDE 155-10

CRITICAL PHASE FOR OBSERVATION

- Check probe rinse for large particles.
- Check filter for large particles and color.
- Check filter for evidence of particle contamination.
- Check ratio of apparent mass collected to visible emission readings.

SLIDE 155-11

CONCLUSIONS

1. Determine purpose of test.
2. Determine magnitude and direction of biases.
3. Know acceptable bias allowed.
4. Make every effort to meet test requirements and to use the data.

Note: Test rejection is costly in time and money for both the source and agency. If sample run will likely be rejected, reject it on-site.

SECTION P. STACK SAMPLING NOMOGRAPHS

INTRODUCTION

This publication is composed of nomographs which Entropy Environmentalists, Inc. has found to be very helpful in estimating or checking parameters measured in stack sampling. The charts are to be used as guides and should not be considered as accurate to the third significant figure (even though some of them are that accurate). The nomographs are separated into three groups: Moisture Content, Excess Air, and Volume and Velocities.

Two hints about using nomographs. If you plan to use the nomographs often, put Scotch "Magic Transparent Tape" over each line; and when too many marks are made on the lines, just erase them from the tape. This saves the printed markings from being erased or obliterated because of excess use. The other hint is that if you plan to copy any of these nomographs on a copy machine, be sure that the machine does not shrink or expand the copy in one direction more than the other.

GROUP I - MOISTURE CONTENT

The stack sampler is frequently confronted with the need to estimate the moisture content of the stack gas prior to sampling. If he estimates incorrectly, he will create an isokinetic error of about one percent for every one percent error in his moisture estimates. For this reason, a package of moisture nomographs has been assembled which will assist and hopefully improve the sampler's estimates.

A. Wet and Dry Bulb Temperatures

Chart 1 is the most useful of the psychrometric charts for stack samplers, since it not only corrects for pressure, but it also gives the results in percent moisture (absolute) on a volume basis. Many stack samplers distrust the wet and dry bulb method because it has given them erroneous data in the past. This is often due to lack of pressure corrections, although the following conditions can also cause problems:

Acid gases (>10 ppm SO_3) yield high moisture results.
Low velocity (<5 ft/sec) yields high moisture results.

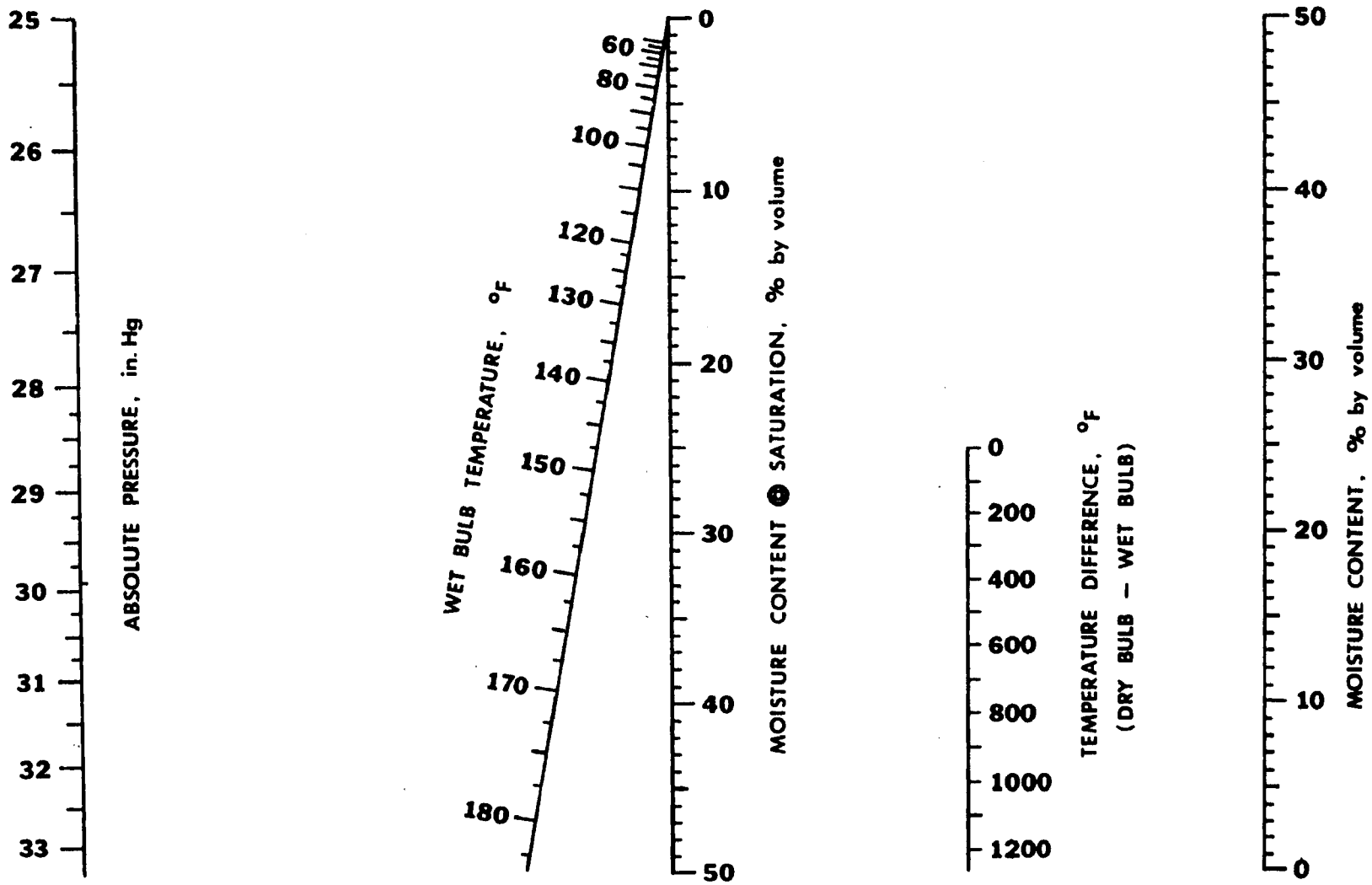


CHART 1

AIR - WATER VAPOR PSYCHROMETRIC CHART

(CORRECTS FOR ABSOLUTE PRESSURE CHANGES)

High velocity (>50 ft/sec) yields erratic moisture results.
High temperature (>250°F) yields erratic moisture results. (HINT: Put wet bulb in hot water instead of cold water.)

Chart 2 is just a simpler chart than Chart 1. It does not have any pressure corrections and is only good at 29.92 inches of mercury. It is included because it can give you approximate answers at a glance. Use Chart 3 to correct for pressure in Chart 2 or any other psychrometric chart.

B. Combustion Calculations

Since most fuels are made of carbon and hydrogen, it is fairly easy to calculate the moisture content for combustion sources from the fuel analysis, the free water in the fuel, the humidity of the combustion air, and Chart 4. First, you must know the excess air. Charts 8, 9, and 10, later in this document, will assist you in this venture.

Next, you need to know the type of fuel. This is usually known unless a combination of fuels or refuse is being fired. For a combination of fuels, calculate from Chart 4-A the answer for each fuel and estimate some reasonable middle answer. For refuse, since refuse is usually paper, choose wood.

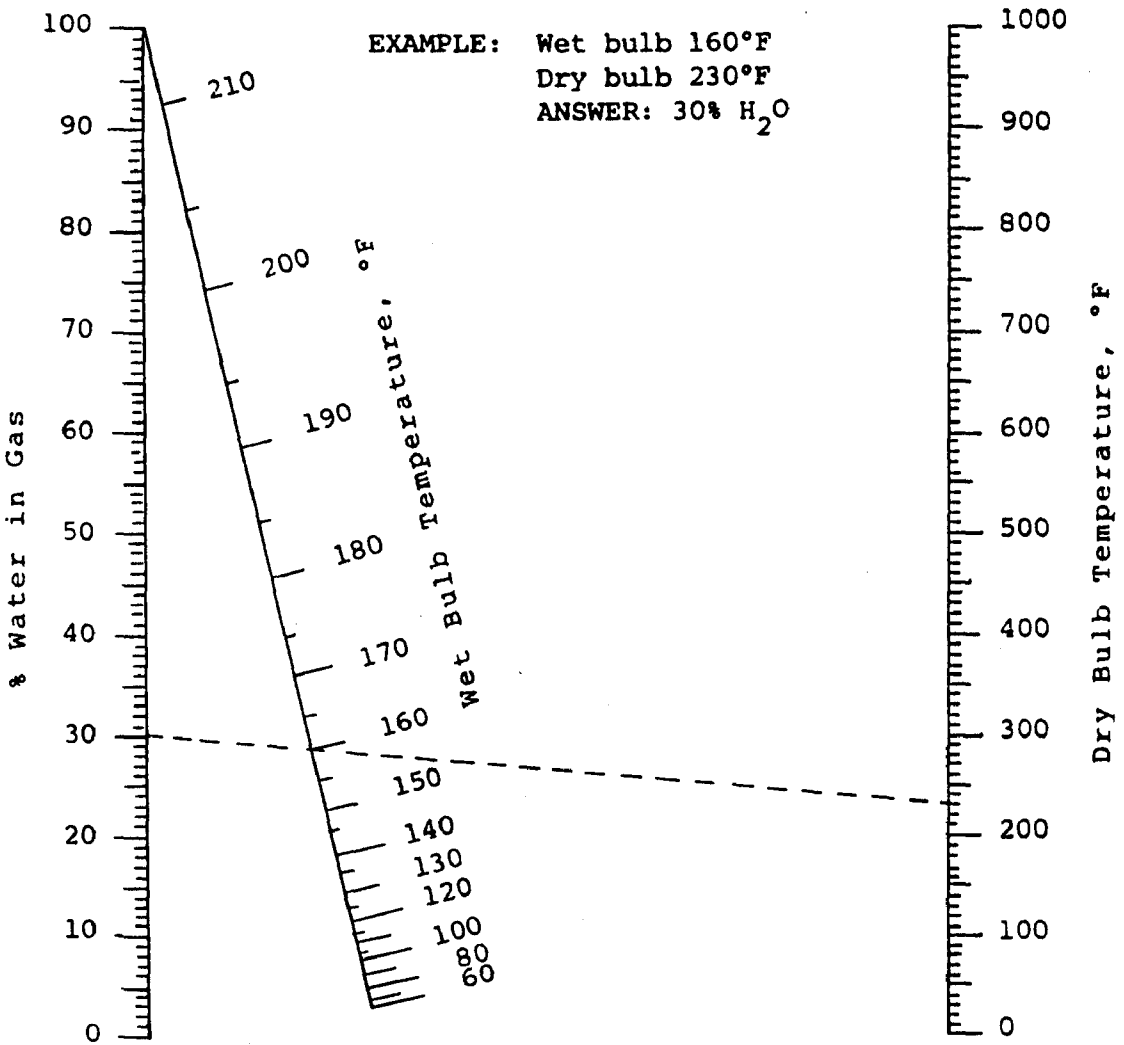
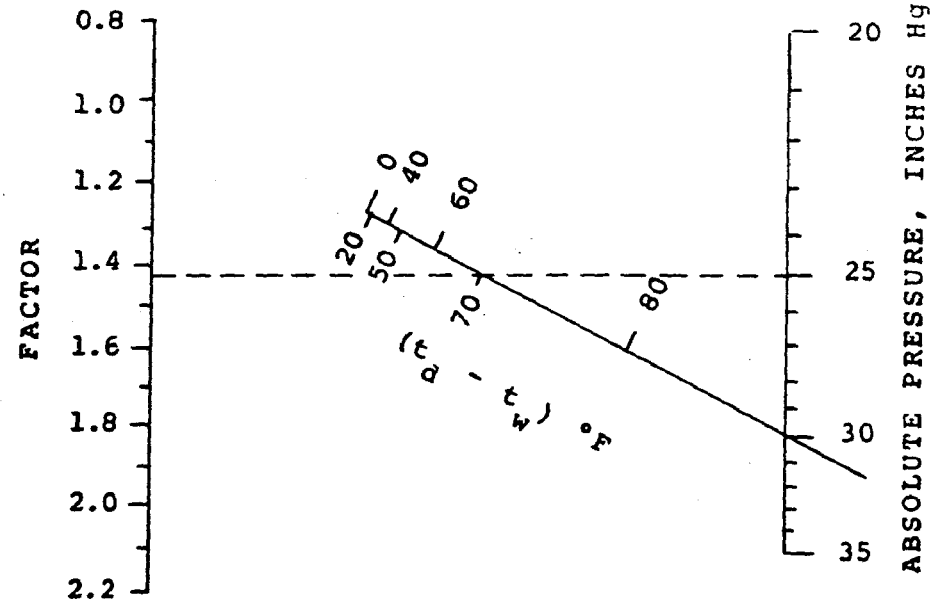


CHART 2

Nomograph for calculating percent moisture in stack using wet and dry bulb thermometers

Assumes 29.92 in. Hg Stack Pressure



EXAMPLE: A Psychrometric chart for t_d = 200°F and t_w = 130°F, yields 11% moisture; however, the absolute stack pressure is 25 inches Hg.

True Moisture - 1.4 x 11% = 15.5% moisture

CHART 3

CORRECTION FACTOR PRESSURE CORRECTIONS FOR THE PSYCHROMETRIC CHARTS

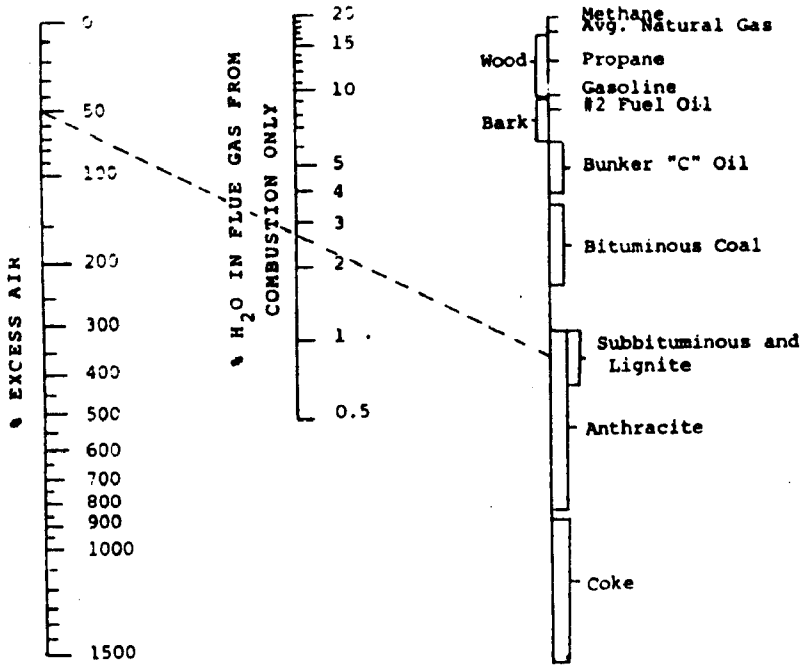


Figure A

FOR DETERMINING MOISTURE IN FLUE GAS FROM COMBUSTION OF FUEL

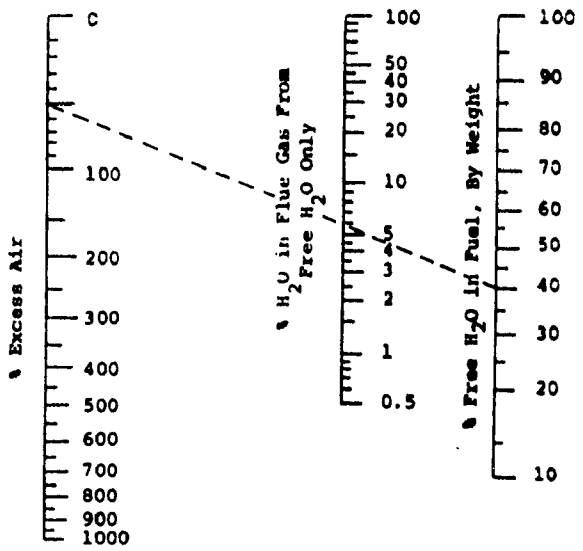


Figure B

FOR DETERMINING MOISTURE IN FLUE GAS FROM FREE WATER IN FUEL

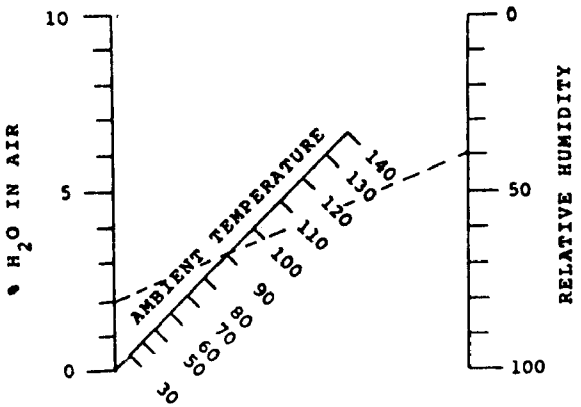


Figure C

MOISTURE FROM THE AMBIENT AIR

MUST KNOW:

1) Type of Fuel
2) Free Water in Fuel
3) Humidity of Ambient Air

EXAMPLE: Lignite with 40% free water is burned at 50% Excess Air, which is 90 °F at 40% Relative Humidity. Add 2.5% (from fuel - Figure A), 6% (from free water - Figure B), and 2% (from ambient air - Figure C) to get 10.5% moisture in the stack gases.

CHART 4

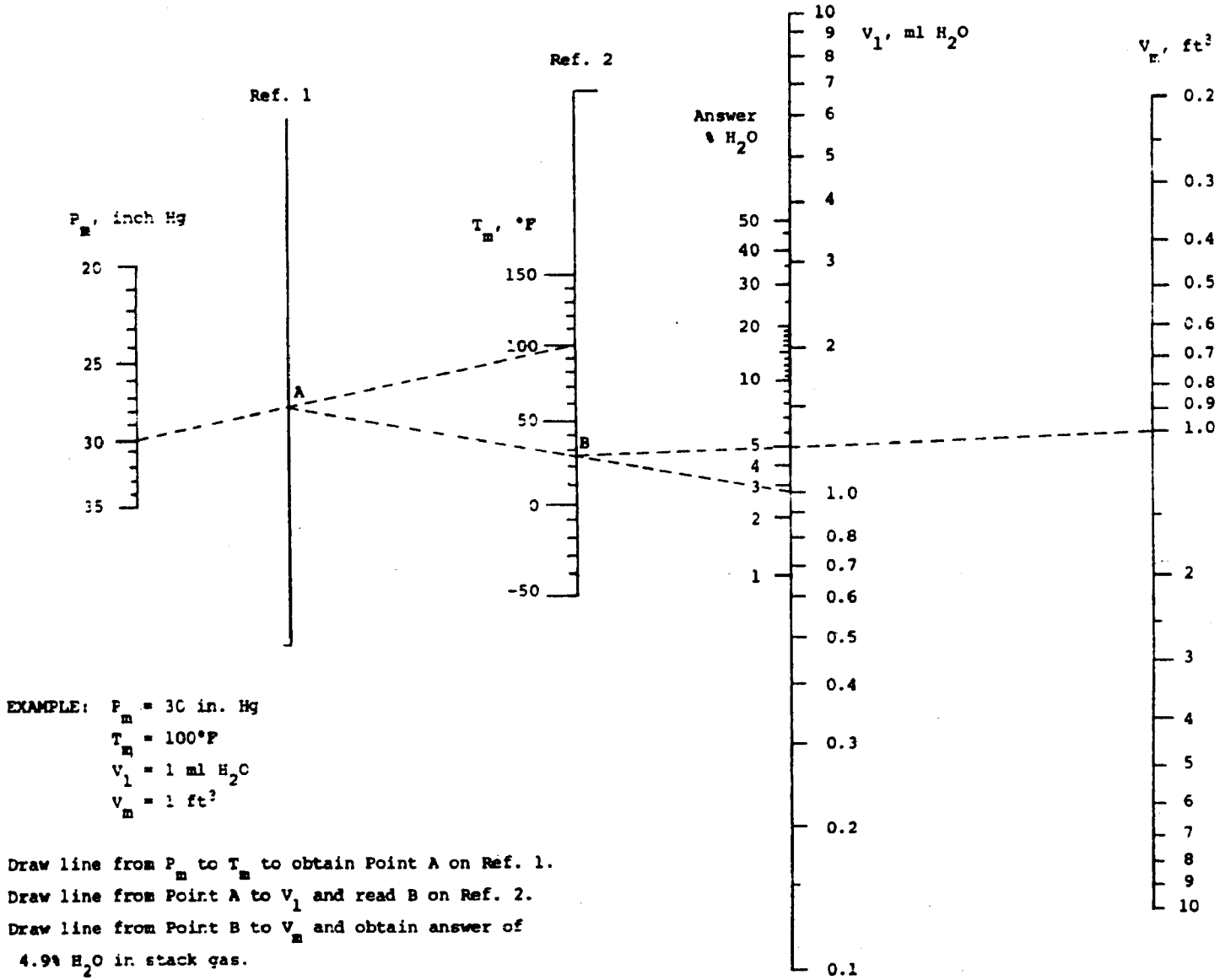
NOMOGRAPHS FOR ESTIMATING MOISTURE IN COMBUSTION SOURCES

The free moisture in the fuel is not readily apparent to the casual observer. Coal and anthracite usually contain from three to fifteen percent, lignite and bark, ten to forty percent, and wood and refuse, five to fifty percent. If it is raining on the fuel, pick a higher number. The source may have some measured data you can use.

The ambient air moisture is usually known. The local radio station usually gives it as percent relative humidity, so Chart 4-C corrects that to percent moisture (absolute).

C. Condenser and Silica Gel Methods

If all estimating methods fail, try measuring the moisture directly. Chart 5 is for low volumes which is associated with silica gel tube methods. Chart 6 is for larger volumes, using the EPA Method 4.

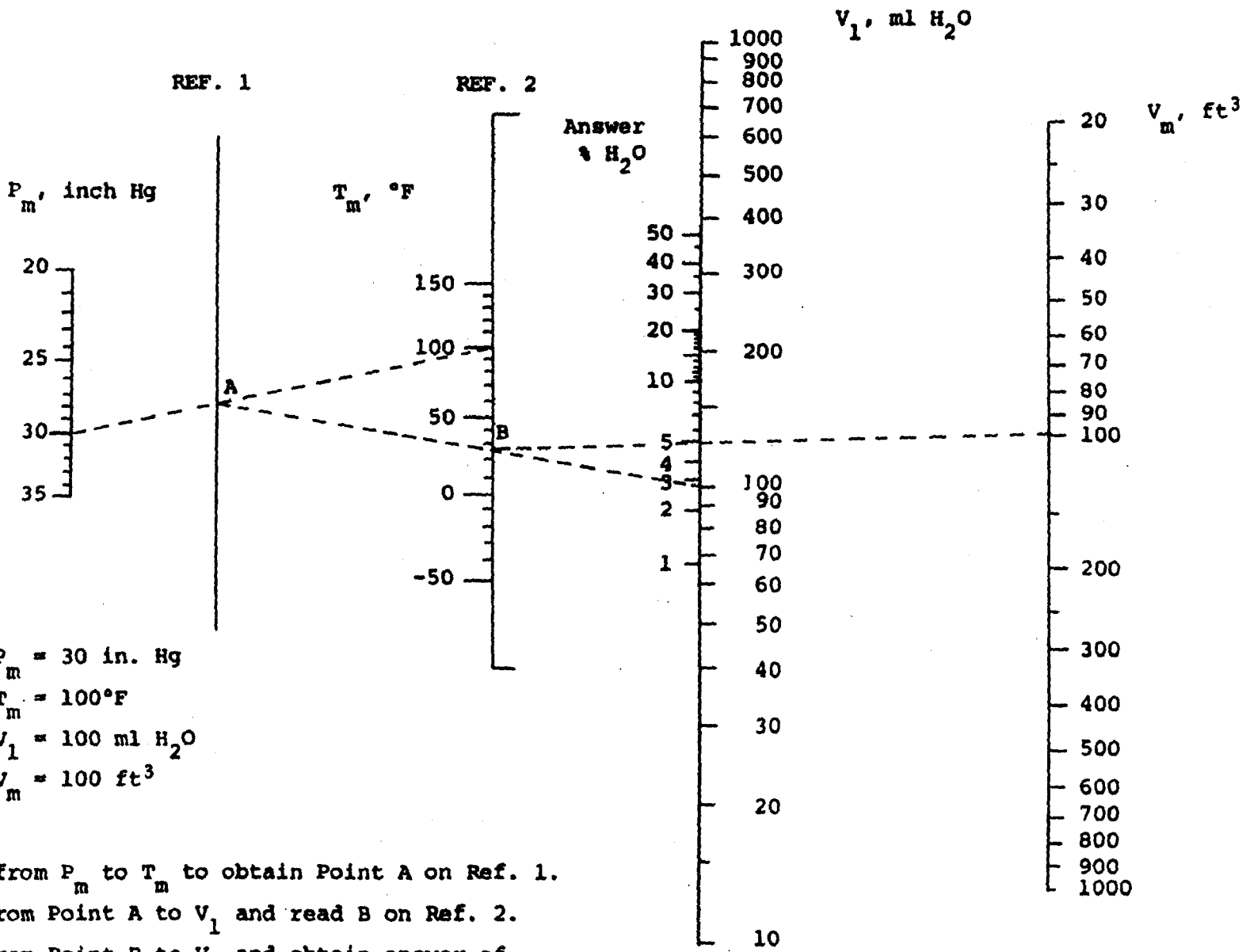


EXAMPLE: P_m = 30 in. Hg
T_m = 100°F
V_l = 1 ml H_2O
V_m = 1 ft^3

Draw line from P_m to T_m to obtain Point A on Ref. 1.
Draw line from Point A to V_l and read B on Ref. 2.
Draw line from Point B to V_m and obtain answer of
4.9% H_2O in stack gas.

CHART 5

NOMOGRAPH FOR CALCULATING MOISTURE CONTENT FROM LOW VOLUMES



EXAMPLE: P_m = 30 in. Hg
T_m = 100°F
V_1 = 100 ml H_2O
V_m = 100 ft^3

Draw line from P_m to T_m to obtain Point A on Ref. 1.

Draw line from Point A to V_1 and read B on Ref. 2.

Draw line from Point B to V_m and obtain answer of 4.9% H_2O in stack gas.

CHART 6

NOMOGRAPH FOR CALCULATING MOISTURE CONTENT FROM HIGH VOLUMES

GROUP II - EXCESS AIR

Knowledge about excess air in a combustion source is necessary not only for use in nomographs such as Chart 4, but it is also helpful for interfacing between stack samplers and combustion experts.

A. Excess Air Versus Flue Gas Composition

Let's say a source is burning a waste oil which is like pentane. If a line is drawn from pentane to 240 percent excess air on Chart 7, the nomograph says the flue gas should have about 15.0 percent O_2 and 4.2 percent CO_2. Conversely, if a line is drawn from pentane to 15.0 percent O_2, it says that there should be 4.2 percent CO_2 and 240 percent excess air. This Chart is useful for calculating excess air, or for validating orsat data, or for calculating CO_2 from O_2 data.

B. Low Excess Air Versus Composition

Chart 8 is the same as Chart 7, except it is for lower excess air and has a dry molecular weight line on it. Some may recognize this as being similar to a chart published in "Atmospheric Emissions from

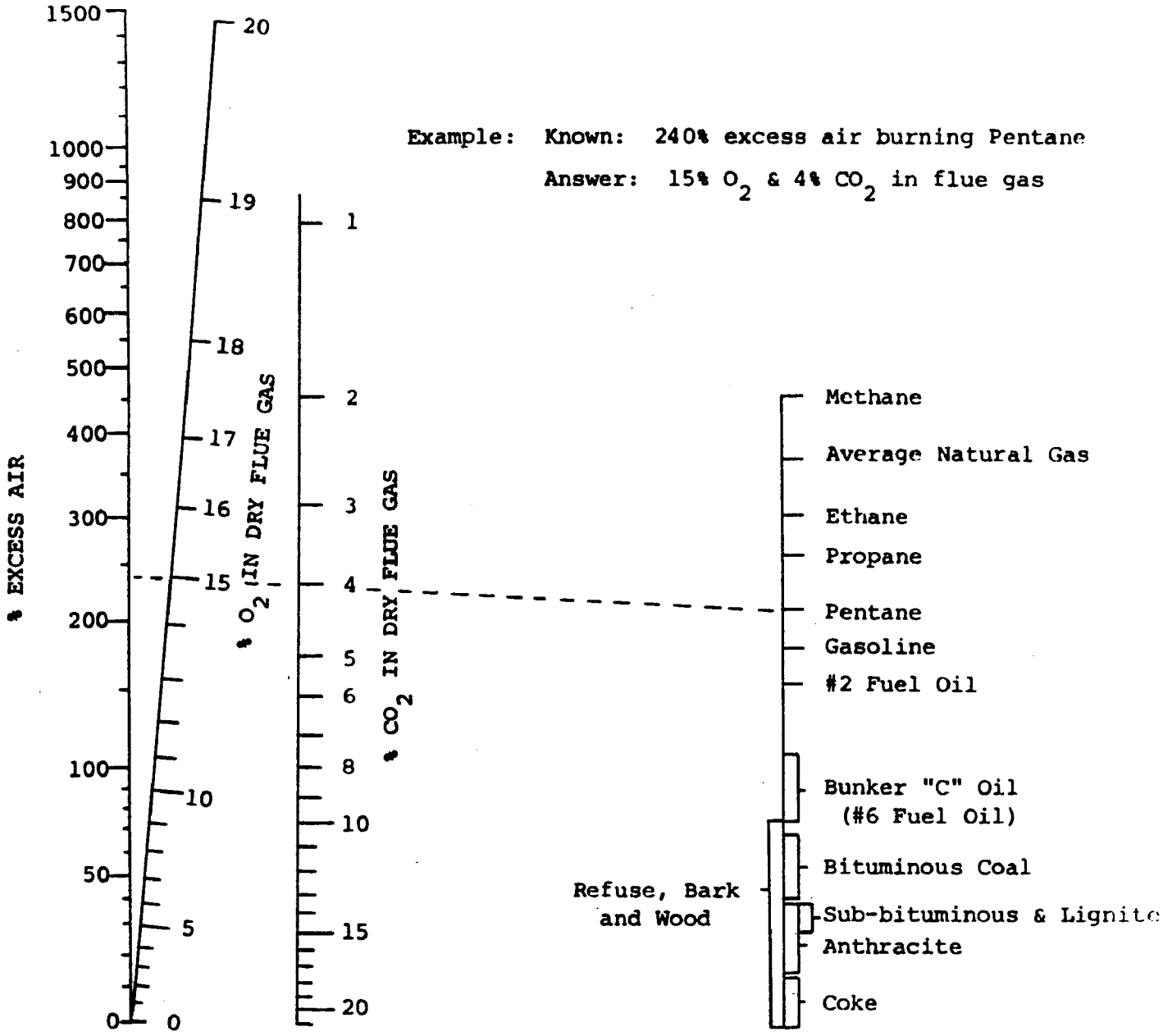


CHART 7

NOMOGRAPH FOR ESTIMATING FLUE GAS COMPOSITION,

EXCESS AIR OR TYPE OF FUEL

Example: Known: 50% Excess Air
burning No. 6 oil

Answer: 7.2% O_2, 10.8% CO_2
and a dry molecular weight of 30

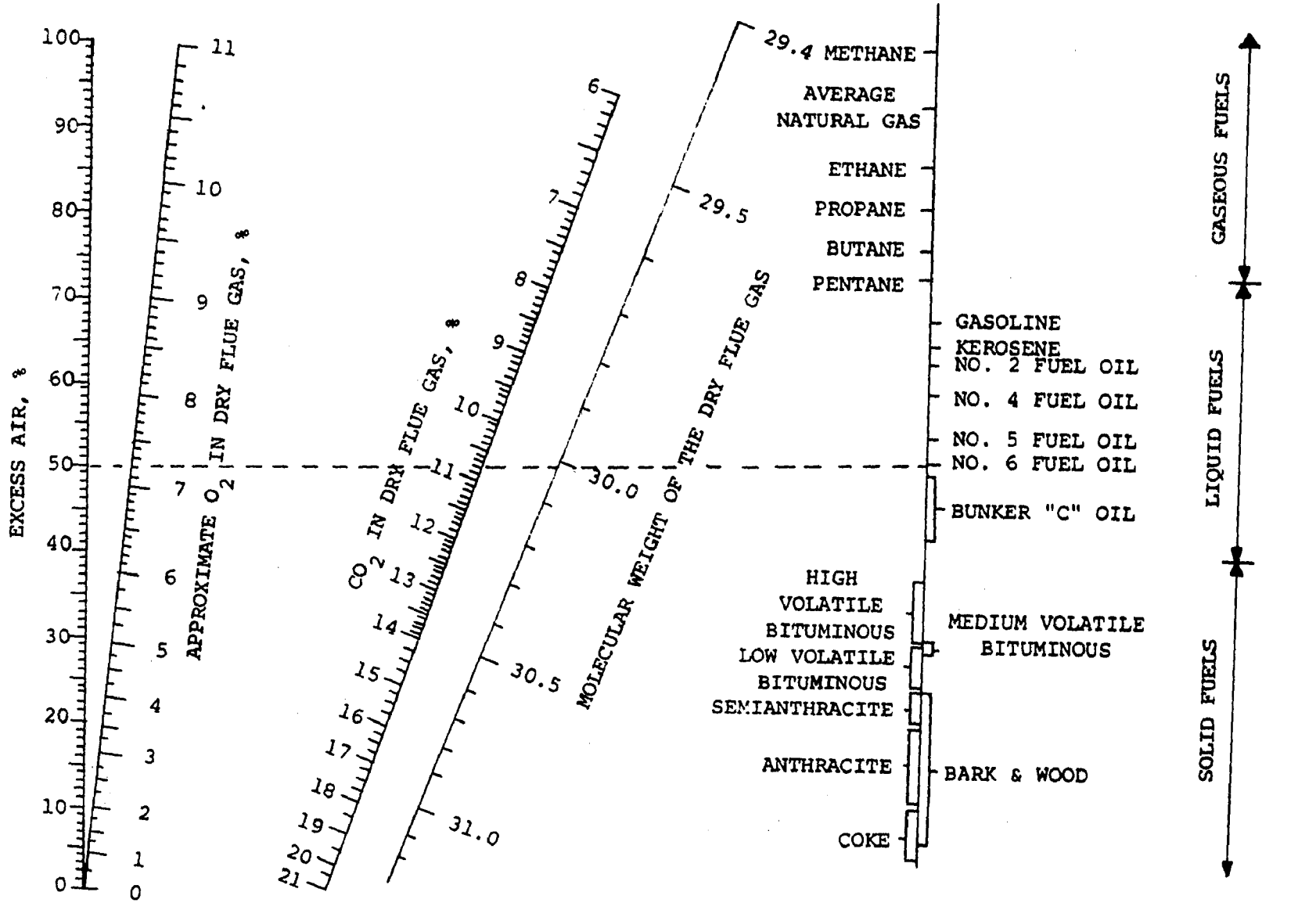


CHART 8

NOMOGRAPH FOR ESTIMATING MOLECULAR WEIGHT OF DRY FLUE GAS

Coal Combustion - An Inventory Guide", by W. S. Smith and C. W. Gruber, P.H.S. No. 999-AP-24. Since then the chart has been improved and the information increased.

C. Molecular Weight Versus Excess Air

Chart 9 gives dry molecular weights for different fuels and amounts of excess air. Chart 10 can be used to convert dry molecular weights to wet molecular weights.

EXAMPLE: Lignite is burned at 50% excess air

Answer: the dry molecular weight is 30.25

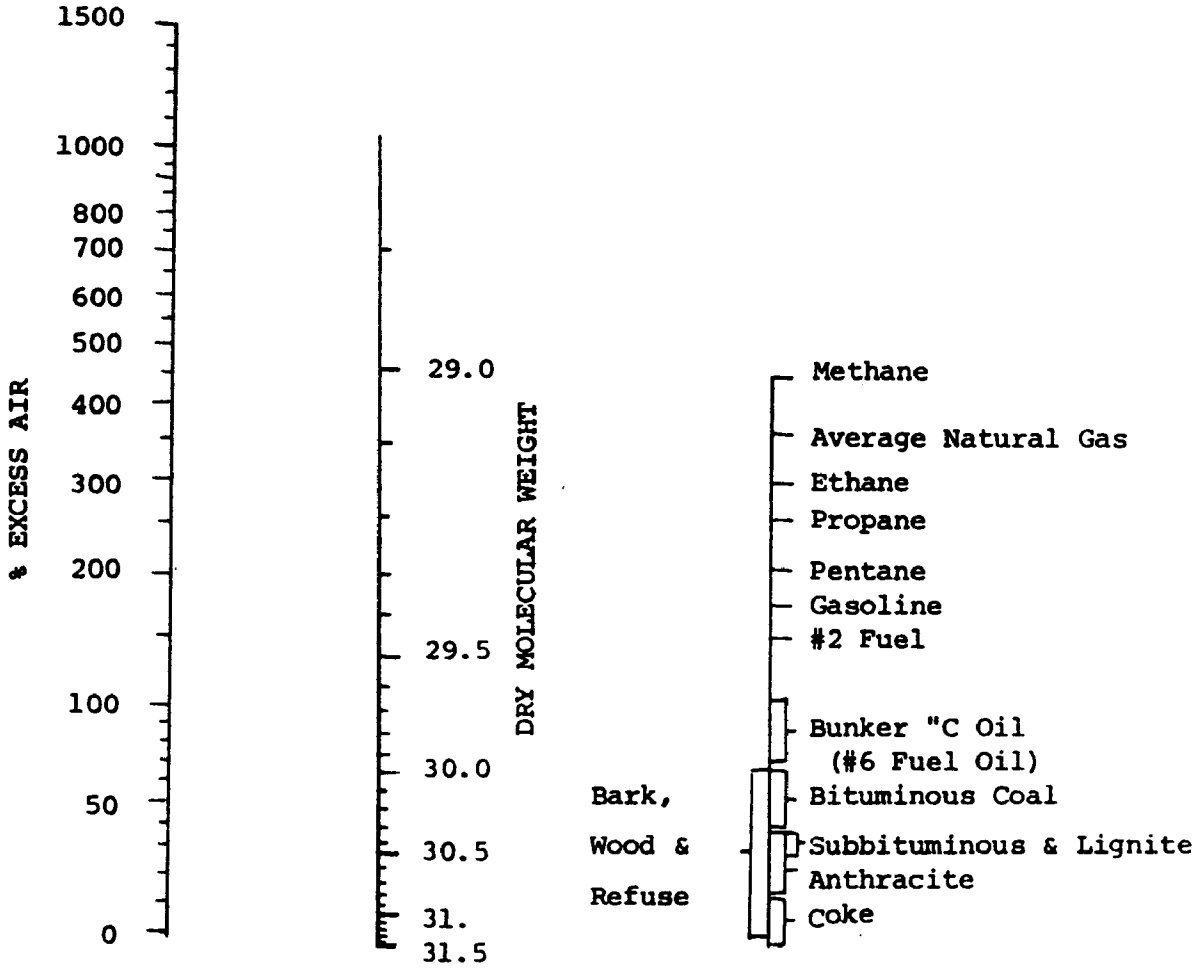


CHART 9

NOMOGRAPH FOR ESTIMATING DRY MOLECULAR WEIGHT FROM EXCESS AIR AND TYPE OF FUEL

EXAMPLE: a dry molecular weight of 30.25 in 10% moisture gas stream gives a 29 wet molecular weight

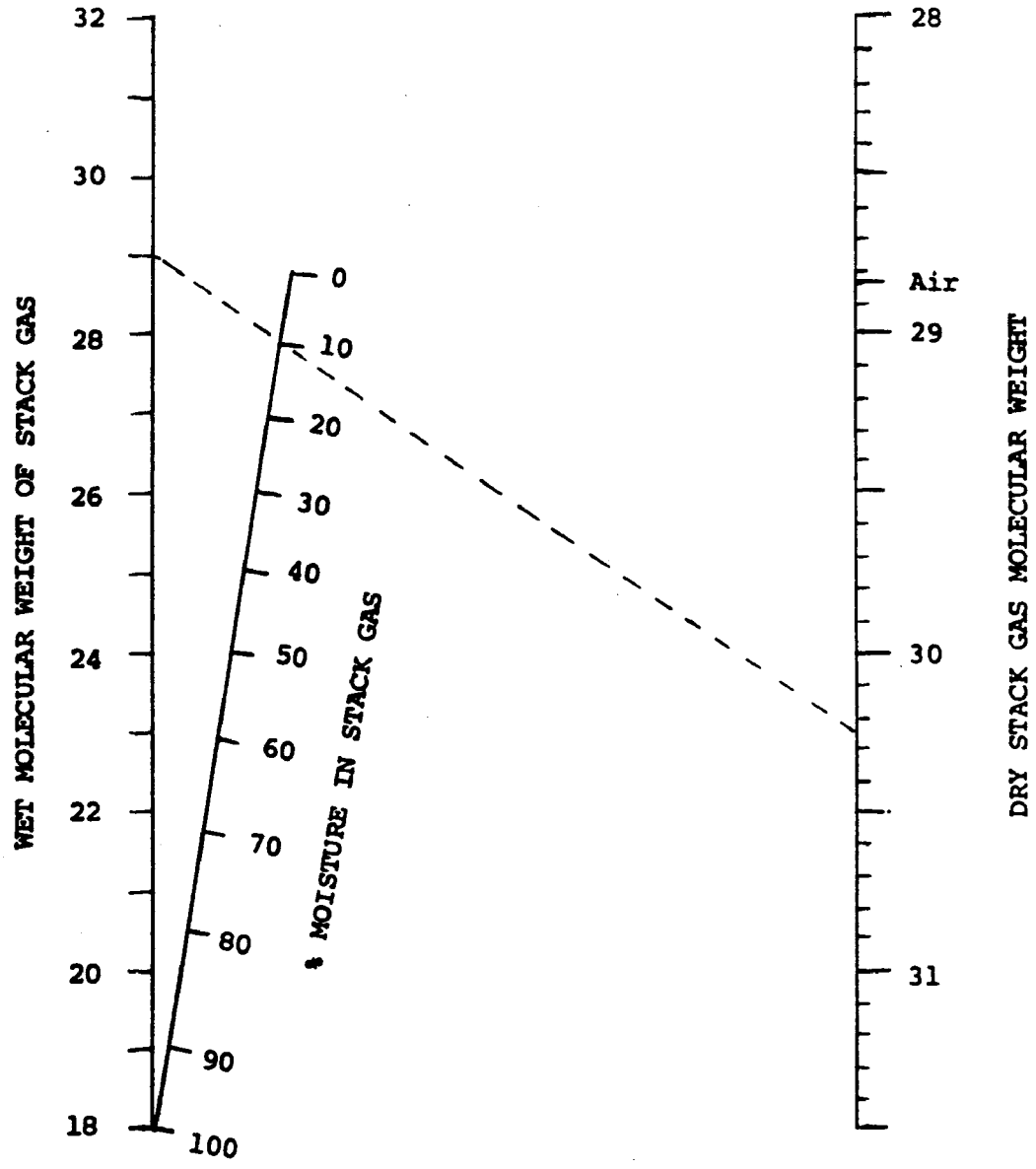


CHART 10

NOMOGRAPH FOR ESTIMATING WET MOLECULAR WEIGHT FROM DRY MOLECULAR WEIGHT AND MOISTURE

GROUP III - VOLUME AND VELOCITY

Sizing nozzles, estimating flow rates, and just checking data requires a decent handle on stack velocity or total volumetric flow.

A. Characteristics of Coal-Fired Equipment

Chart 11 is a very handy chart which was included in "Atmospheric Emissions from Coal Combustion - An Inventory Guide". The volumes appear to be correct except those for stokers with control equipment, which reflect the volume at maximum capacity. As an example, we have a 100,000 pounds of steam per hour spreader stoker fitted with draft-controlled (constant volume) multiple cyclones. The chart would tell us that the boiler input was about 120 million Btu per hour, burning about 8000 pounds of coal per hour, it could generate about 10 megawatts, the stack gas was about 28,000 scfm at 35 percent excess air, coal to steam efficiencies in the lower 80 percent, stack gas temperatures about 500°F, and could be used in a hospital or even as a large industrial boiler. The important thing to note is that if this boiler is running at half load, the

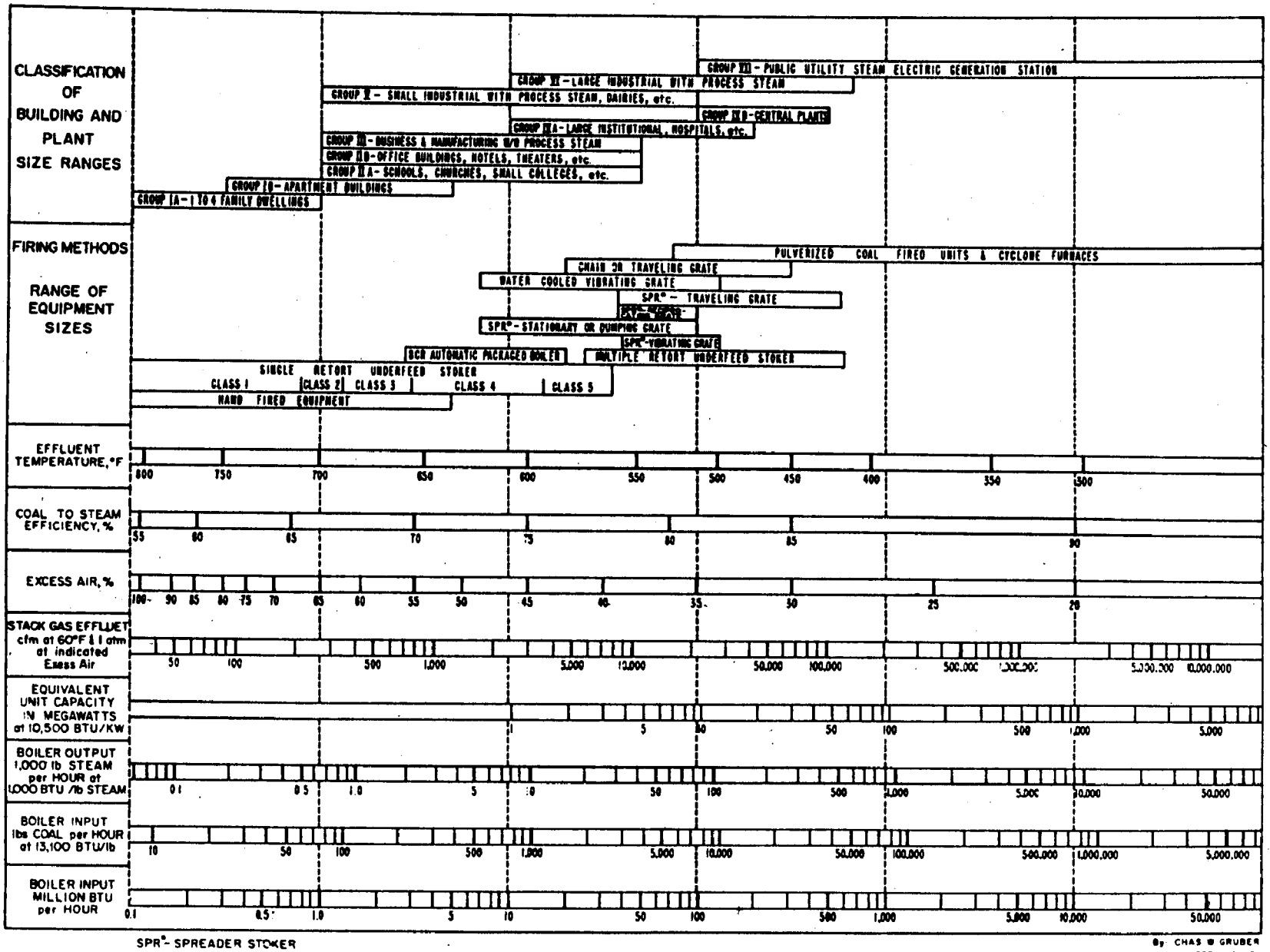


CHART 11

SUMMARY OF CHARACTERISTICS OF COAL-FIRING EQUIPMENT

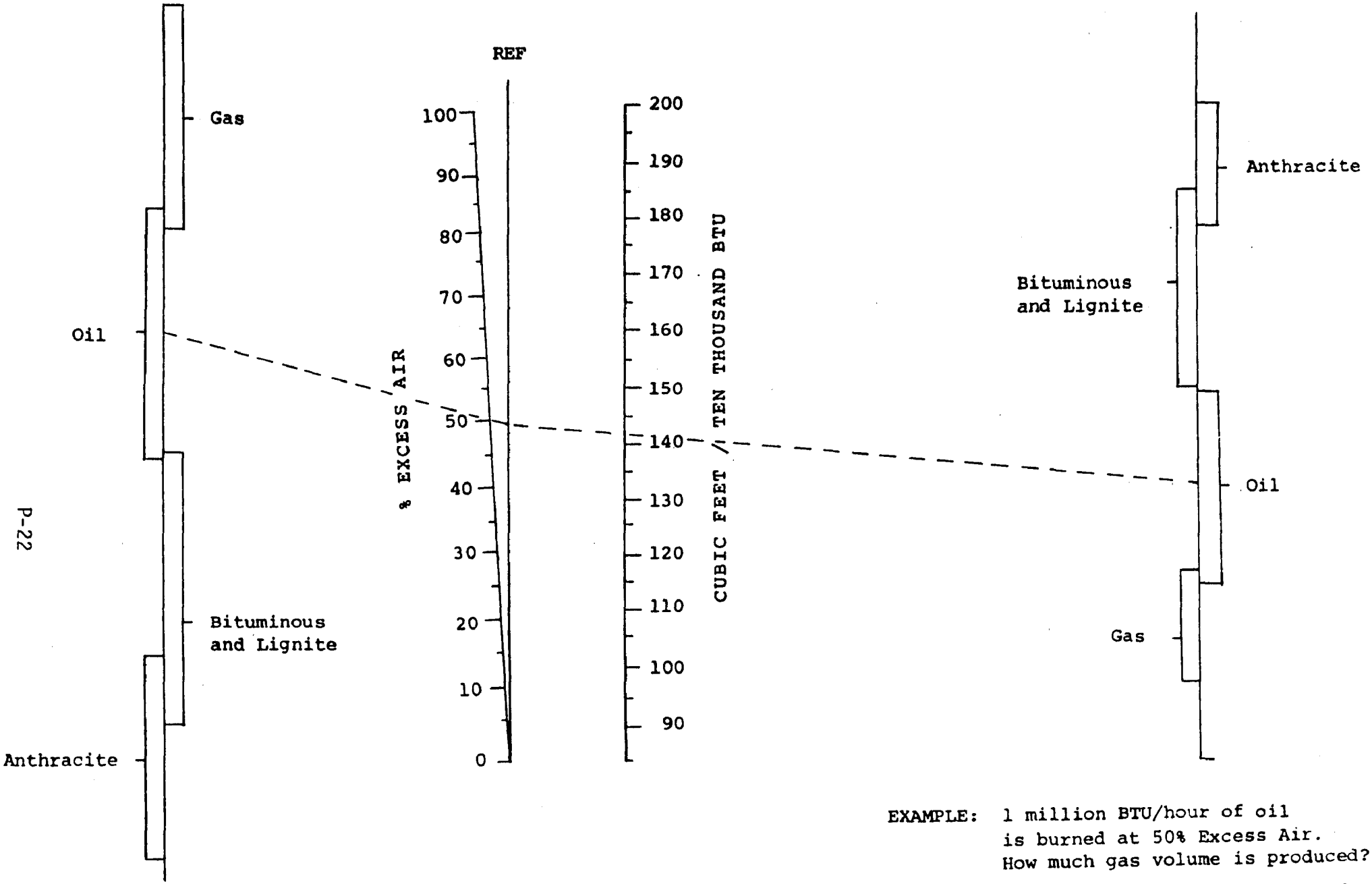
volume will be about the same, the excess air about 270 percent, and a gas temperature of about 200°F. This sort of thing does not happen with pulverized boilers with electrostatic precipitators.

B. Volume Versus Heat Input

Chart 12 is a more exacting chart which can calculate the flow rate up a stack on a combustion source if the heat input is known. The data for this chart is in an unpublished paper as of April 1973, called "A Method of Calculating Power Plant Emission Rates", by R. T. Shigehara, R. M. Neulicht, and W. S. Smith. As can be seen from the chart, the heat input need not be measured if the volumetric flow rate and excess air is known.

C. Pitot Velocity Readings

The novice often finds it difficult to relate pitot tube readings into actual velocities. By using Chart 13, one can quickly relate an "S" type pitot tube reading into a velocity which is usually near the actual value. This chart also has a scale so one can correct the velocity to standard (STP) conditions which are 68°F and 29.92 inches of mercury pressure.



EXAMPLE: 1 million BTU/hour of oil is burned at 50% Excess Air. How much gas volume is produced?

ANSWER: 14,200 cf/hr. @ 70°F dry

CHART 12
VOLUME VERSUS HEAT INPUT CHART

EXAMPLE: Stack temperature 500°F
ΔP = 0.5 inches H_2O

Answer: Actual approximate Velocity = 54 ft/sec
Approximate velocity at STP* = 30 ft/sec

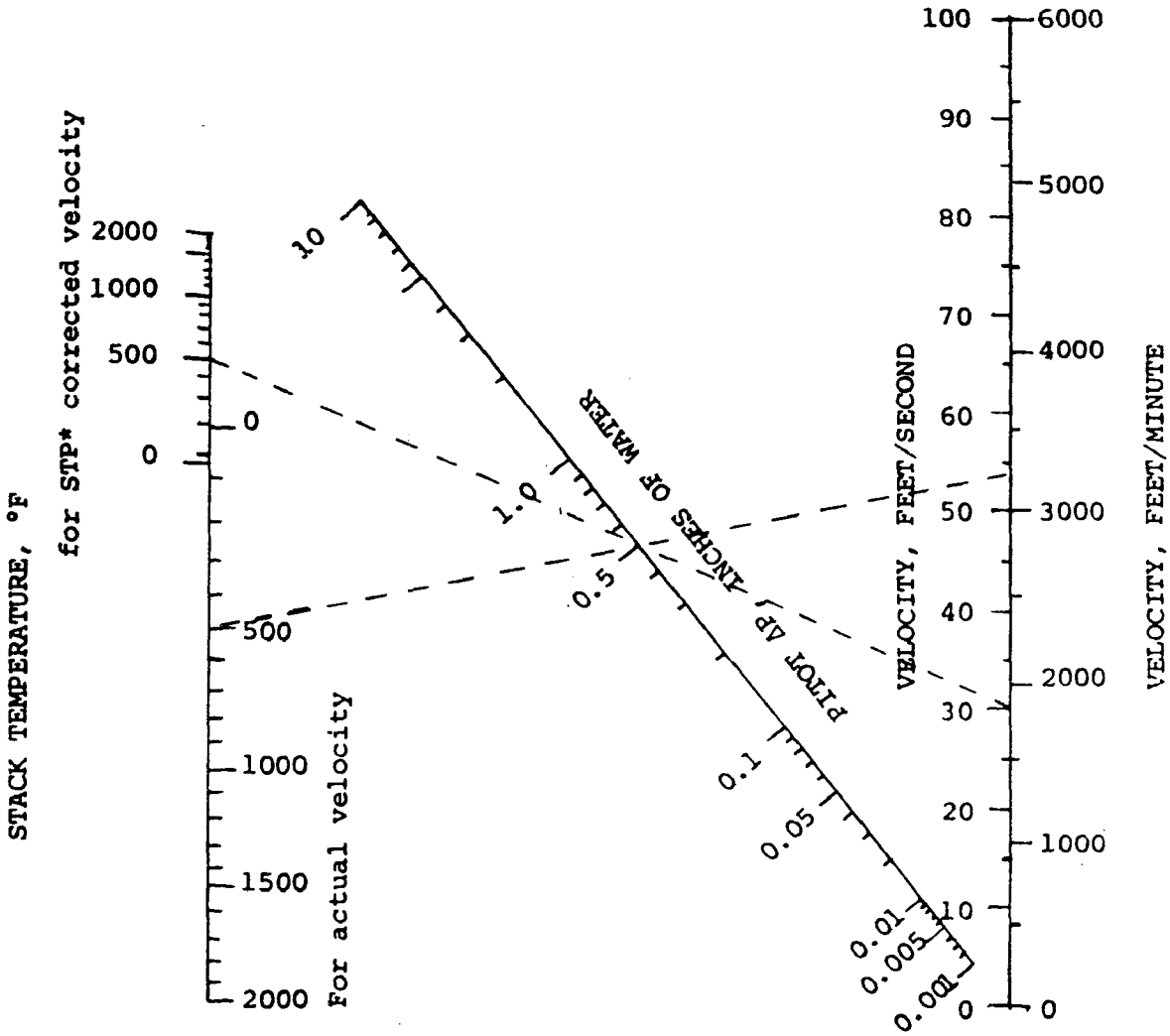
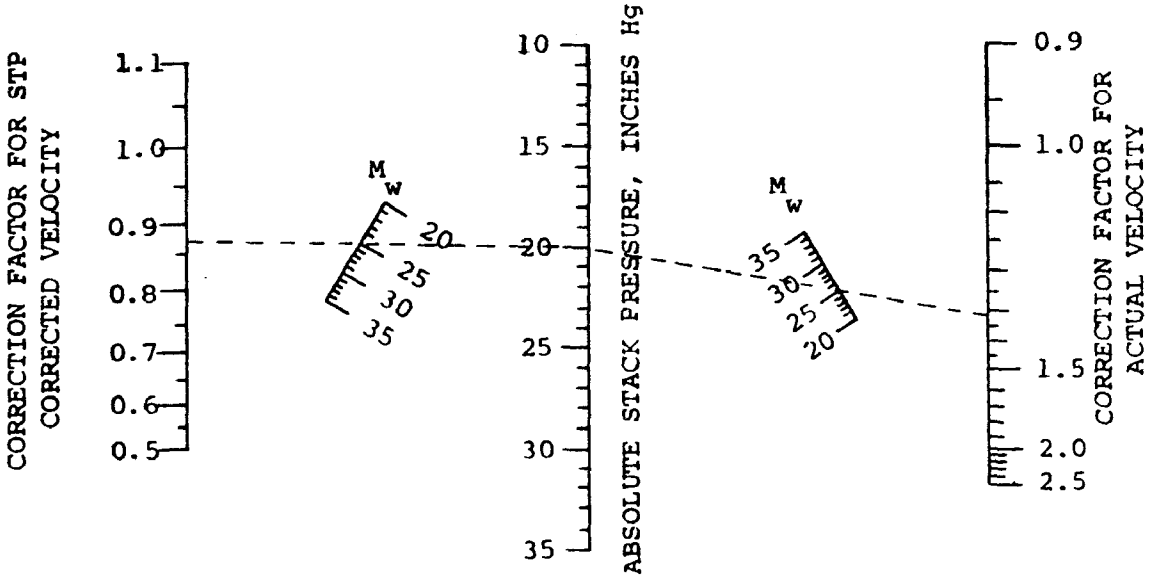


CHART 13

Approximate velocity Nomograph

Assumes Type "S" pitot tube used at 1 ATM in Air
STP = 68°F & 29.92 inches of mercury

Chart 14 corrects for the errors in Chart 13, not only for the actual velocity but also for velocity that has been corrected to STP. The example given is a very extreme case which could exist after a venturi scrubber, but before a fan (high moisture gives low molecular weights). This extreme example was given in order that you not implicitly trust Chart 13, especially under extreme conditions.



EXAMPLE: From Chart 13

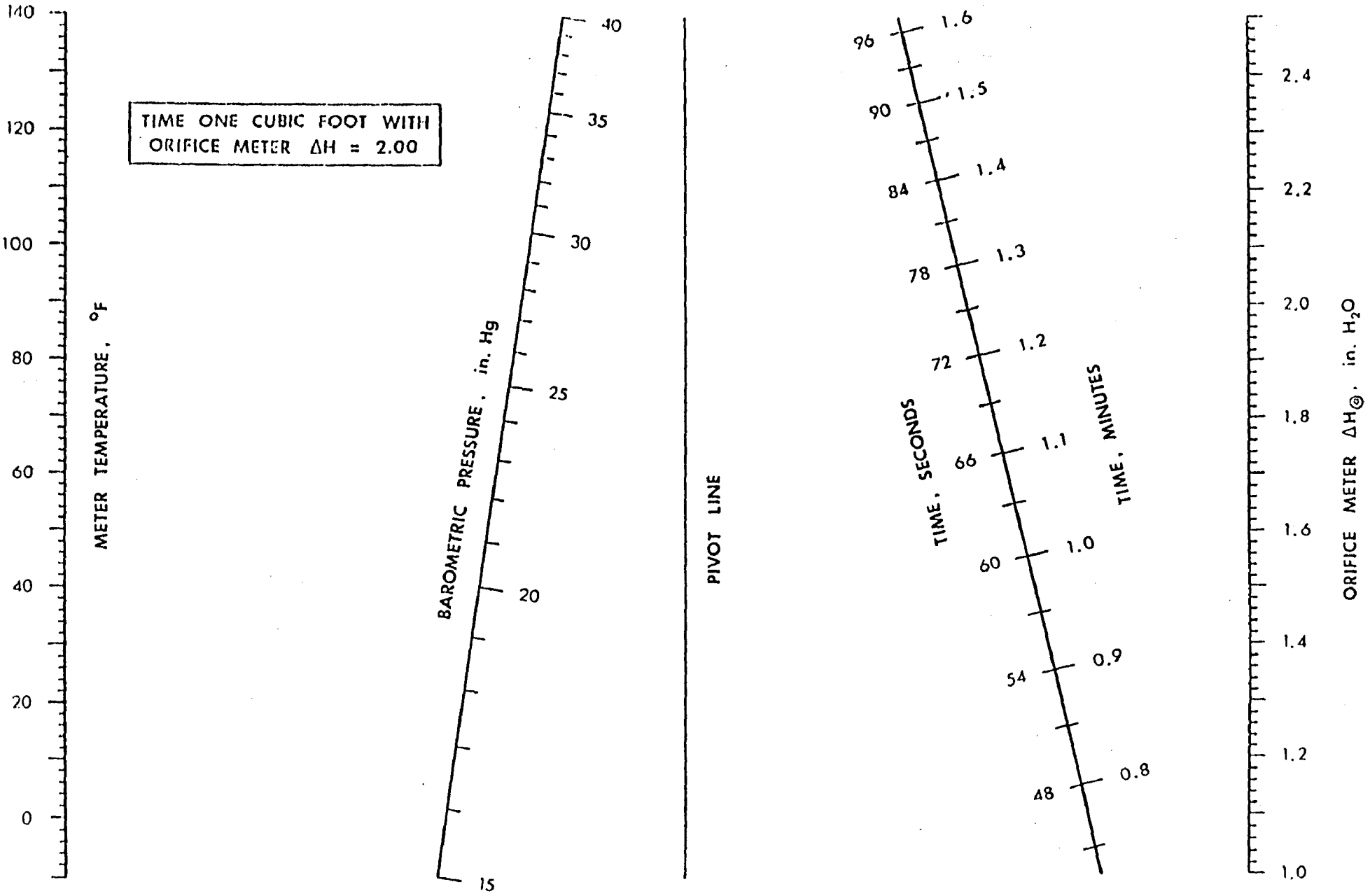
Approx. Actual Velocity 54 ft./sec.
Approx. STP Velocity 30 ft./sec.
Actual Molecular Weight (M_w) = 25
Actual Stack Pressure = 20 inches Hg

ANSWER: Actual Velocity 1.32 x 54 = 71 ft./sec.
STP Velocity 0.87 x 30 = 26 ft./sec.

CHART 14

CORRECTION FACTORS FOR VELOCITY CHART

SPECIALISTS IN
SOURCE
SAMPLING



Date:

Meter Box:

CALIBRATION CHECK - EPA TRAIN METER BOX

Given ΔH_A:

Graph ΔH_A:

SLIDE 156-0

NOTES

STACK SAMPLING NOMOGRAPHS FOR FIELD USE

SLIDE 156-1

Example: **Wet Bulb - 160°F**
Dry Bulb - 230°F
Answer: **30% H_2O**

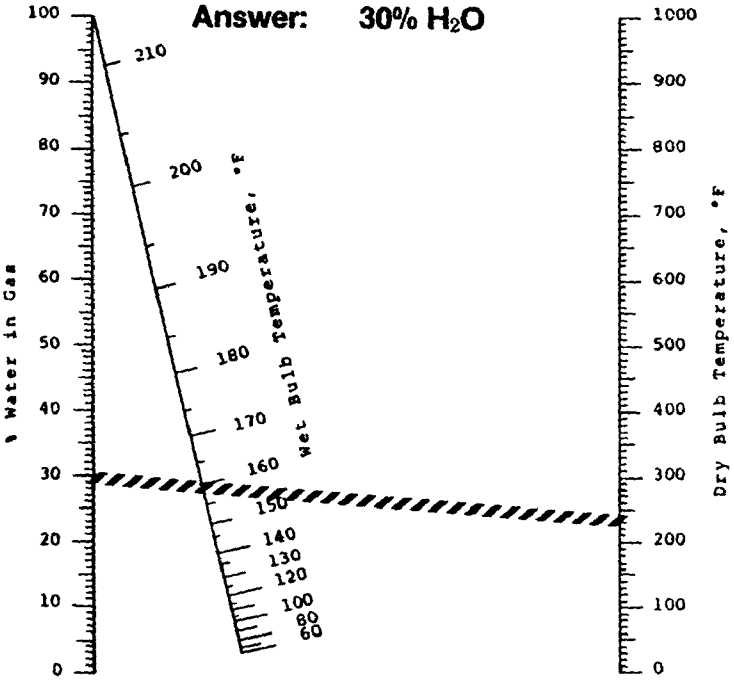
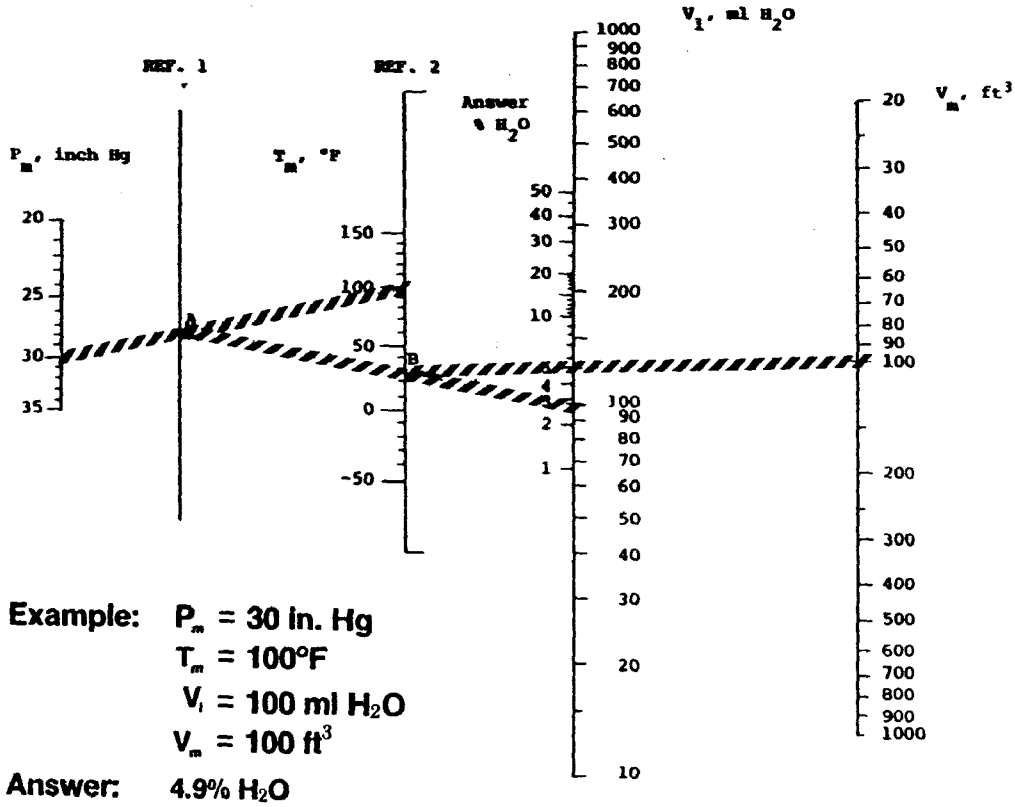


chart 2

SLIDE 156-2

NOTES



Example: P_m = 30 in. Hg
T_m = 100°F
V_l = 100 ml H_2O
V_m = 100 ft^3

Answer: 4.9% H_2O

chart 6

SLIDE 156-3

NOTES

Example: **Known: 50% Excess Air**
Burning No. 6 Oil

Answer: **7.2% O_2, 10.8% CO_2**
Dry Molecular Weight of 30

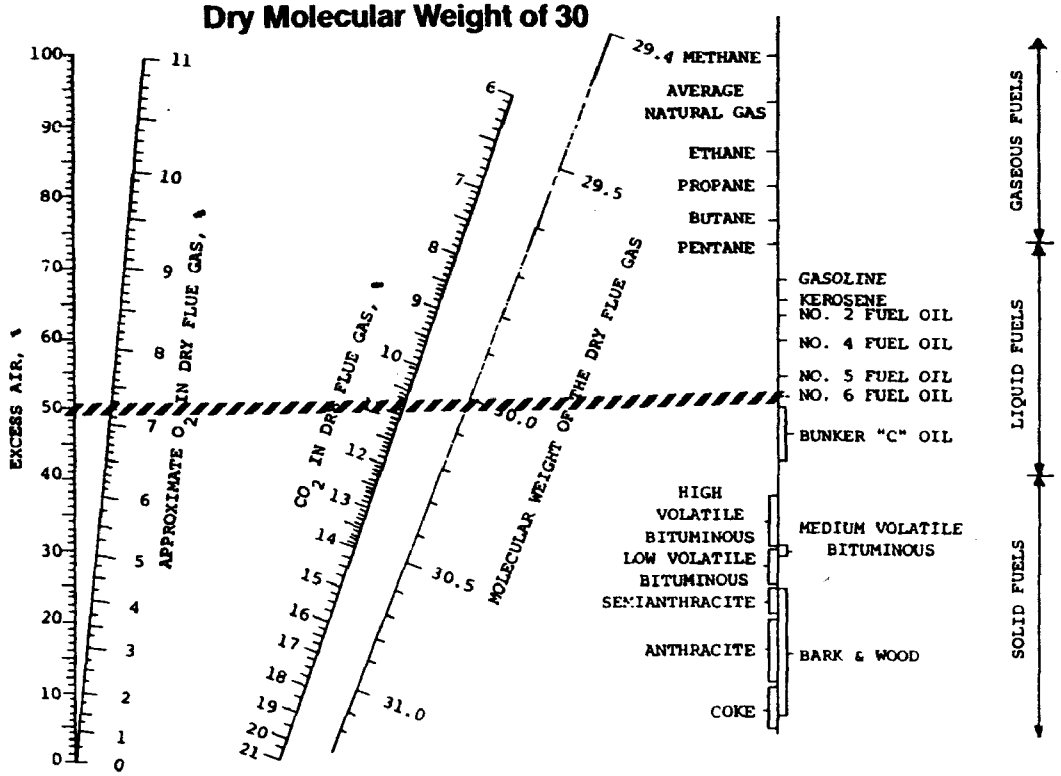


chart 8