WHITE MESA MILL

ENVIRONMENTAL SURVEILLANCE
PROCEDURES MANUAL

January 1985

APPENDIX D

ENVIRONMENTAL SURVEILLANCE PROCEDURES MANUAL

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1.0 AIR MONITORING

WHITE MESA PROCEDURES MANUAL

Monitoring Guideline
Environmental

AIR MONITORING - PARTICULATE

RADIONUCLIDES

1.0 EQUIPMENT

Equipment utilized in monitoring levels of ambient particulate radionuclides is composed of high volume (40 cfm) air samplers manufactured by General Metals Works and equipped with mass flow controllers and vacuum switch controlled timers. The mass air flow controller, once calibrated, varies the actual air flow rate, as affected by changing temperature, filter loading, and barometric pressure, to maintain a constant standard air flow rate. The timer, recording continuous run time, is controlled by a vacuum switch which automatically deactivates the timer when no vacuum exists in the system, i.e., when the motor/blower assembly is disconnected or malfunctions.

The high volume air sampler used is the General Metal Works Model 2310 Accu-Vol, with a modification on the timer. These samplers utilize an eight by ten inch glass microfiber filter such as:

1. Whatman EPM 1000 or

2. Schliecher & Schuell #1 HV.

Calibration equipment consists of a Model GMW-6000 Primary Calibration System Roots Meter and Model GMW-25 calibration orifice assembly.

2.0 MONITORING METHODOLOGY

The air samplers are mounted on wooden towers two meters above ground plane and operate through use of 110-volt line power. The samplers run continuously except for calibration, mechanical or electrical failure, and maintenance downtime.

Calibration of equipment occurs monthly, or following maintenance downtime, using the orifice plate method.

The sampler flow rate is regulated to a standard air volume, using twenty-five degrees centigrade and twenty-nine point nine-two inches of mercury as standard conditions.

2.0 MONITORING METHODOLOGY (continued)

Sample media replacement is scheduled weekly due to particulate loadings. Maximum filter use duration will be weekly.

Sample handling is as follows:

The filter media is removed from the package and is examined by naked eye by passing the media between the eye and a light source to detect any visible tears or pin pricks. Any abnormality in the filter is cause for discardment of the filter.

The filter is stamped in code signifying the filter number. The filter is pre-weighed to the nearest 0.0001 gram utilizing a Sartorious Model 2432 analytical balance. The filter tare weight is then recorded on the filter envelope along with the location, filter number, and date. On return from the field, the filter is again re-weighed and gross weight is recorded on the filter envelope. The filter number, net weight, and duration is recorded on the sample station log sheet.

At the sampler, following shutdown of the system, the "loaded" filter is removed, folded, and placed within it's original envelope. The envelope is marked by dates for the duration of the sample, time changed, and actual operating time in minutes. If any part of the filter remains on the seal gasket, it is removed and added to the envelope contents. The new, clean filter is removed from its envelope and placed over the suction head with the filter holder tightened enough for a seal, but not tight enough to rupture the filter. The sampler is then activated.

On a quarterly basis, each station filters are composited. The samples are forwarded, along with an analytical sheet to the Analytical Laboratory in a sealed transport container. The analytical results are then returned via a copy of this form. Blank filters are sent in quarterly for background radionuclide concentrations.

3.0 CALIBRATION

Samplers are checked monthly using the orifice plate assembly for calibration documentation. In addition, samplers are checked by this method following each motor replacement.

The calibration procedure is:

1. Obtain air temperature and barometric pressure levels at the site.

2. Place a clean filter on the sample platform and mount the calibrated orifice on it.

3.0 CALIBRATION (continued)

3. Place the filter under vacuum and obtain pressure differential across orifice plate.

4. Using this pressure differential, refer to orifice calibration curve graph to obtain the uncorrected flow rate (Qa).

5. Determine the corrected flow rate (Qs) using the following equation:

$$Qs = Qa \times \left[\frac{T_1P_2}{T_2P_1} \right]^{1/2}$$

Note: T^1 = Absolute Temperature at orifice calibration (273+ ^{o}C = degrees Kelvin)

T^2 = Absolute Temperature at hi-vol calibration (degrees Kelvin)

P^1 = Atmospheric Pressure at orifice calibration - Standard - 760 MM Hg

P^2 = Atmospheric Pressure at hi-vol calibration

6. Qs is the standard flow rate.

7. To correct equipment to the required standard flow rate, use the following equation:

$$Qa = Qs \times \left[\frac{T_2P_1}{T_1P_2} \right]^{1/2}$$

where: Qs is the required standard flow rate.

8. Determine the manometer reading from the orifice calibration curve which gives a flow rate of Qa.

9. Set the hi-vol to this manometer reading. (Flow through the equipment setup is regulated by the flow controller through adjustment of the screw mounted on transformer P^1 of the flow controller).

10. Disconnect the orifice and reassemble the filter head assembly for normal operation.

The orifice is recalibrated semi-annually against a positive displacement Roots meter to obtain the above-required orifice calibration curve.

4.0 RECORD KEEPING

Information included on the individual sample station log sheet is:

1. Sample period;
2. Sampling Location;
3. Suration of each sample, minutes;
4. Sample ID Number;
5. Sampler Flow Rate, Total Sample Volume, Standard Conditions;
6. Filter net weight, milligrams;
7. Analytical values in concentrations per composite sample;
8. Computed air concentrations (standard air conditions); and,
9. General Remarks.

Filters are analyzed for: U-nat., Ra-226, Th-230 and Pb-210.

Documentation of calibrations is maintained on Form UMC-A-1 attached.

The above form is maintained in that year's Environmental Operating log book under the appropriate sections or in computer files.

5.0 MONITORING LOCATIONS AND FREQUENCY

Particulate sampling is on a continuous basis with weekly or twice weekly rotation of filters and quarterly compositing of filters for analysis.

The monitoring locations are:

1. BHV-1; northeast of mill;
2. BHV-2; nearest resident;
3. BHV-3; Black Mesa background;
4. BHV-4; Southwest of mill; and
5. BHV-5; east southeast of mill.

AIR SAMPLER CALIBRATION WORKSHEET

UMETCO MINERALS CORPORATION
WHITE MESA

SITE	DATE	AIR TEMP. °C	BARO. PRESS	INT. "H_2O	INT. RATE	FIN. "H_2O	FINAL FLOW RATE
Sp.D							
TABE.							
CLAR.							
SEW.							
SWP.							
C-B							
F-B							
W#2							
FUS.							
		°C	"H_2O		scfm		scfm

CALCULATIONS ARE:

To calculate Initial Flow Rate:

1. Determine actual flow rate (Qa) from calib. curve for manometer reading for orifice
2. Determine the standard flow rate (Qs) using:

$$Qs = Qa \times \left[\frac{T_1 P_2}{T_2 P_1}\right]^{1/2}$$

To calculate final flow rate:

1. Determine Qa for a Qs of 40 scfm using:

$$Qa = Qs \times \left[\frac{T_1 P_2}{T_2 P_1}\right]^{1/2}$$

2. Determine the manometer reading from the orifice calibration curve which gives a flow rate of Qa.
3. Adjust sampler using flow controller adjustment.

NOTE:

T_1=ABSOLUTE TEMPERATURE AT ORIFICE CALIBRATION (273+°C). (298°K)

T_2=ABSOLUTE TEMPERATURE AT AIR SAMPLER CALIBRATION. (273 + °C)

P_1=ATMOSPHERIC PRESSURE AT ORIFICE CALIBRATION (29.92 "H_g)

P_2=ATMOSPHERIC PRESSURE AT AIR SAMPLER CALIBRATION ("H_g).

6.0 ANALYSIS PROCEDURE

The analytical procedures utilized are presented in detail in the Analytical Procedures Manual of the representative laboratory.

Analytical results are forwarded to White Mesa showing values for requested time intervals as curies or micrograms per composite sample.

In calculating radionuclide concentrations in air, analytical data is entered into a computer program which draws corresponding data from the field information file, sample dates, flow rates, sample duration, and net particulate loading, calculates concentrations and stores them in another file.

1. For uranium, the reported sample assay value is subtracted by the reported blank filter value. This result is then divided by the known standard air volume and conversion constants to achieve a concentration in air value. If any value is of a less than value, then fifty percent of that value is utilized and a less than value is assigned to the result.
2. For thorium, radium, and lead-210, the reported sample assay values and the blank assay values are reported with a counting error term. The resultant value is the difference between the sample assay value and the blank value divided by the known sampled air volume. The counting error deviation is the square root of the sum of the counting error terms squared and divided by the known sampled air volume.

7.0 QUALITY ASSURANCE

Installation/Removal of Filters

Quality assurance is maintained by:

1. Inspecting all new filters for aberrations and discarding those that are;
2. Maintaining seals on equipment connections;
3. Careful installation and removal of filters, retaining all abraded filter media;
4. Proper sealing of all gaskets;
5. Proper sequential handling of filters; and,
6. Filters are inspected for fingerprint contamination by visual observation of fingerprints.

7.0 QUALITY ASSURANCE (continued)

Sample Duration

Record maintenance of sample duration is assured by:

1. Installation of a vaccum-actuated timer which operates the timer only when motor is running and pulling the minimum allowable vacuum; and,
2. Weekly monitoring of motor brush and alternator bearing wear/proper changeout at appropriate intervals.

Sample Flowrate

Quality assurance of sample flowrate is by monthly checks of flow controller operation and documentation thereof. Samplers are checked for calibration at motor rotation intervals. Sample flowrates are maintained within seven percent of required flowrate.

Calculations

Calculations are checked on a random basis for inconsistencies and are documented.

Upon retrieval of the data analytical sheet from the support laboratory, the date of receipt is noted along with the date of transcribing to the sample station log sheet and the transcriber's initials. The transcription of data is reviewed by another person of the department to minimize transposition of numerical values. Calculation and data storage is by computer program.

Sampler Performance

A record of sampler operation time versus total possible duration time is maintained as a flag against excessive equipment downtime. Sampler performance is reviewed monthly.

WHITE MESA PROCEDURES MANUAL

MONITORING GUIDELINE

ENVIRONMENTAL

AIR MONITORING - AMBIENT

RADON GAS

1.0 EQUIPMENT

Equipment utilized in monitoring ambient levels of Rn-222 gas is the Passive Environmental Radon Monitors, (PERMS), issued by Western Radiation Consultants, which utilize an ionized TLD chip (at 670 volts) as the collector for alpha particles and an unionized TLD chip as a collector of gamma level radiation. A humidity trap of approximately three point three (3.3 lbs.) pounds of silica gel (with indicator coloration) is used to control humidity conditions within the ionized collection cone of the sampler. The silica gel is changed based solely upon the change in color of the indicator, approximately monthly, at White Mesa. The TLD's utilized are Harshaw TLD-100 (Lithium Fluoride) for gamma and Harschal TLD-200 (Calcium Fluoride) for alpha collection.

2.0 MONITORING METHODOLOGY

The "PERMs" are located at three meters above ground level.

The PERM's TLD's are rotated the first week of each month for analysis. Two sets of TLD's are utilized for each PERM. The alpha chip, covered by a aluminized mylar shield, is mounted on a cathode with a plastic "O" ring holding it in place. The gamma chip is mounted within a sealed plastic container which is fastened by an "O" ring in the vicinity of the alpha chip and cathode assembly. Handling of the chips, themselves, is done solely by the technical staff of the support laboratory.

One set of chips, in their individual holders, is rotated monthly to the Western Radiation Consultants laboratory for counting purposes while the other is placed in the monitor. The chips installed are forwarded monthly to White Mesa where the rotation occurs within two days. Each set of chips, one set containing a lithium fluoride chip assembly and a calcium fluoride assembly, per monitor station, is transported in its individual container. The chips are contained within their individual labeled holders, one set marked "A", and the other "B", and are changed thereby to prevent transposition of chips.

3.0 CALIBRATION

Calibration of the PERM's and TLD chips is performed by Western Radiation Consultants. This calibration occurs between receiving the units from the manufacturer and installation in the field.

The reaction activity level of the TLD chip, when heated to the proper temperature, is related to the known radon gas concentration and gamma levels and test duration in determining individual chip performance and calibration coefficients. Analytical error is the summation of counting error, TLD chip performance error, and sampling duration error.

The batteries used for the cathode energization are Eveready Photoflash No. 497 batteries wired to give 670 volts. The battery life is approximately one year and they are changed annually. Voltage potential is checked by a high voltage electrostatic voltmeter quarterly.

4.0 RECORD KEEPING

The month's TLD chips, in their respective containers, are forwarded by sealed container via the postal service to the support laboratory in Fort Collins, Colorado. Accompanying the samples is a transmittal analytical sheet on which the analytical and chain of custody data is recorded. Upon completion of analysis, the analytical sheet is transmitted to White Mesa where the data is transcribed from the transmittal to computer files by the White Mesa Environmental Staff. The transmittal sheet is filed under the ambient radon gas section of the maintained files, separated by year of record.

5.0 MONITORING LOCATIONS AND FREQUENCY

Radon 222 is monitored continuously with monthly rotation of the sensors (chips) and correspondingly an average monthly result of concentration and deviation.

The monitoring locations are:

1. BHV-1, northeast of mill,
2. BHV-2; nearest resident,
3. BHV-3; Black Mesa background,
4. BHV-4; southwest of mill,
5. BHV-5; east southeast of mill, and
6. BHV-6; nearest resident (duplicate).

6.0 ANALYSIS PROCEDURE

The analytical procedures utilized are presented in detail in the Western Radiation Consultants Procedure Manual. The concentration represented on the lithium fluoride (gamma) assay is subtracted from that represented on the calcium fluoride (gamma and alpha) assay to give a representative value of the alpha concentration exposed to by the cathode chip. This value corresponds to a predetermined Radon-222 level in the ambient environment.

After analysis, the chips are heated to a higher temperature and reactivated for further field usage.

7.0 QUALITY ASSURANCE

Installation/Removal of Chips

Quality assurance during installation and removal of the sensors on a monthly basis is maintained by:

1. Inspecting the sensors to ensure the chips are present and situated properly;
2. Inspecting the sensors for proper labelling;
3. Inspecting all electrical connections before and after chip changeout to ensure proper current flow;
4. Inspecting battery casing for battery leakage;

7.0 QUALITY ASSURANCE (continued)

5. Proper rotation of set A and set B chips;

6. Installation of site chips, for analysis, into site transport box immediately upon removal from monitor; and

7. Protection of chips from direct sunlight and heat buildup during rotation.

Access to the section containing the chips within the monitor is controlled by a lock to limit unauthorized access. Abnormal variations to normal operation conditions are noted in field logs by the technician(s) assigned. This includes any vandalism noted.

Humidity Control

Air passing into the unit, via convection, passes through a layer of silica gel for humidity control purposes. The silica gel quantity is approximately one point five (1.5) kilograms. The gel has an indicator coloration which changes from saphire blue to light sky blue to pink as it retains moisture. The gel is changed when greater than fifty (50) percent is sky blue by visual observation. Changeout occurs monthly at chip changeout, and any variation from this frequency is noted in the comment section of the field logs.

Voltage Potential

The PERM increases the collection efficiency for alpha particles by supplying a positive charge of 670 volts to a cathode on which the alpha sensitive chip is placed. The power is derived from two 510 volt batteries connected in series. The combined voltage is checked on a quarterly basis with a precision electrostatic high voltage voltmeter. Variations from desired voltage are noted on field logs. Variations greater than fifty (50) volts require replacement, as reasonably achievable, of the batteries. Batteries used are Eveready Photoflash No. 497. Surveys will be done in January, April, July and October of each year.

Sample Treatment

The TLD Chips are loaded at the site into an individual site container labeled for that site. The chips are checked during preparation for transshipment to the support laboratory. An analytical sheet goes with the chips detailing locations, dates, analytical request, and technician initials. The chips are sent via the postal transport service and are enclosed in a cardboard outer container which is sealed with packaging tape. The support laboratory is verbally notified of the shipment. A copy of the analytical sheet is returned to White Mesa noting arrival at the laboratory and maintained at White Mesa until the completed copy is returned from the support laboratory.

7.0 QUALITY ASSURANCE (continued)

Data Logging

Upon retrieval of the data analytical sheet from the support laboratory, the data is entered into a computer file for later retrieval.

Sampler Performance

Standby samplers are used on a periodic basis to check equipment and laboratory performance. Data is logged on field logs as a calibration check.

WHITE MESA PROCEDURES MANUAL

MONITORING GUIDELINE

ENVIRONMENTAL

AIR MONITORING - METEOROLOGICAL

1.0 EQUIPMENT

There is one meteorological station at White Mesa. The equipment utilized is summarized below:

1. Weathertronics Model 2030, Micro Response Anemometer,
2. Weathertronics Model 2020, Micro Response Wind Vane, and
3. Campbell Scientific Digital Recorder, Model CR5.

In addition, a substation is maintained at White Mesa and consists of a standard rain/snow gauge.

1. Standard Rain/Snow Gauge.

The meteorological station is equipped to operate on line or battery backup power, for power outages. The recordkeeping is handled in two methodologies, by digital cassette logging, and by paper tape printout. The logging equipment allows integration averaging over various time intervals from five minutes to twenty-four hours.

2.0 MONITORING METHODOLOGY

2.1 Substation

The substation is monitored at eight each morning by the crew who records twenty-four hour precipitation amounts. This information is utilized weekly by the environmental staff for precipitation storage calculations.

2.2 Meteorological Station

The meteorological station integrates data at a rate of one hundred times per minute and output mean values on an hourly interval basis. Data is outputted onto analog cassette tape and onto a digital paper tape providing two sources of data recovery.

2.0 MONITORING METHODOLOGY (continued)

2.2 Meteorological Station (continued)

The parameters monitored are:

1. Day,
2. Hour,
3. Minute,
4. Wind Mean Velocity, meters per second,
5. Total Wind Run, meters per second,
6. Wind direction, degrees from north, and
7. Stability Deviation, degrees.

3.0 CALIBRATION

3.1 Substation

The substation equipment requires no calibration as equipment meets NOAA specifications and NBS standards.

3.2 Meteorological Station

Calibaration of sensors mounted at the meteorological station is done on the following schedule:

1. Semi-annually -- Wind Direction
2. Annually -- Wind Velocity

Equipment is recalibrated following maintenance activities and/or above schedule.

3.2.1 Wind Direction

The sixth channel of the CR5 printer record contains wind direction information in degrees clockwise from true north. By aligning the arm of the wind vane to a known compass reading and obtaining the instantaneous readout on the printer, verification of wind direction can be achieved. Adjustment to true direction requires minute rotation of the potentiometer within the wind vane housing and must be accomplished with great care. Accuracy of wind direction should be less than two degree error.

3.0 CALIBRATION (continued)

3.2.2 Wind Velocity

The fourth channel of the CR5 printer record is wind velocity in meters per second while the fifth channel is total wind run in meters per second. The anemometer operates via a LED diode light emission being interrupted on varying frequencies by a rotating chopper attached to the cups is proportional to a frequency pulse, which is the signal being fed to the CR5 unit.

Calibration is performed by rotating the cup stem, using a constant rpm generator, and comparing frequency pulses at the signal end to the known frequency rate for that rpm. Differences greater than one percent require investigation and servicing of the unit.

4.0 RECORDKEEPING

4.1 Substation

The daily substation data is filed in the Environmental Files at White Mesa on a monthly summary sheet.

4.2 Meteorological Station

Data for wind speed, wind direction and stability classification is retained in computer storage.

5.0 MONITORING LOCATION AND FREQUENCY

Monitoring of meteorological conditions is on a continuous basis. Data collection and retrieval for the substation is daily. Digital tape retrieval of the meteorological data is monthly. Paper tape retrieval is on three week intervals.

Meteorological data is obtained by the sensors at a rate of one hundred times per minute, and the equipment is set up to output averaging data on one-hour intervals.

The site of the substation is behind the main administration building. The meteorological station is located to the northeast of the mill at air sampling station one (BHV-1).

The station has sensors mounted ten meters above the ground plane.

6.0 QUALITY ASSURANCE METHODOLOGY

6.1 Substation

Quality assurance of the substation consists of:

1. Weekly checks by environmental staff of crew function, and
2. Monthly checks of data transmittal.

6.2 Meteorological Stations

Quality assurance of the meteorological station is composed of:

1. Proper calibration of equipment,
2. Documentation of calibration,
3. Monthly documented inspections of equipment operation, and,
4. Maintenance of replacement standby equipment.

The formats covering calibration documentation and inspections are attached.

Wind direction sensors are checked semi-annually and wind anemometers are calibrated annually, or as needed.

Proper recording of data requires monthly cleaning of tape head mechanism and usage of correct tape for frequency response. The cassette tape to be used is:

TDK D-C90 minute tape

Normal Bias 120 EQS

METEOROLOGICAL STATION

CALIBRATION LOG

Site:______________ Year:____

WIND DIRECTION

Tech. Int.	Date of Calibration	Sensor Readout	Known RPM Direction	% Error
1.				
2.				
3.				

WIND VELOCITY

Tech. Int.	Date of Calibration	Sensor Readout	Known RPM Velocity	% Error
1.				
2.				
3.				

METEOROLOGICAL STATION

INSPECTION

YEAR__________ MONTH__________ TECH.__________

Checkoff List

Power to Unit__________ Power to Cassette Recorder__________

Backup Voltage Level__________

Date on Tape__________ Date of Check__________

Time on Tape__________ Time of Check__________

Printer Tape Condition__________

Tape Head Cleaned__________

Time Tape Installed__________

Time Tape Reversed__________

Time Tape Disconnected__________

Anemometer Operation__________

Wind Vane Operation__________

COMMENTS__________

WHITE MESA PROCEDURES MANUAL

MONITORING GUIDELINES

ENVIRONMENTAL

AIR QUALITY EMISSIONS

1.0 EQUIPMENT

Monitoring of air quality emissions, or stack sampling, is achieved by White Mesa Environmental personnel using Research Appliance Company equipment as discussed below:

1. RAC Model 201009 Model 2414 Stack Sampler Serial Number 2310;
2. Two each, RAC Model 201044 Modular Sample Case;
3. One each, RAC Model 201019 Umbilical Cord;
4. Three each, RAC Model 201013 - 100mm dia. Filter Holders;
5. One each, RAC Model 201005 Standard Pitobe 3 ft length, stainless steel./S-type probe;
6. Barometer;
7. Pyschronometer;
8. Satorius Model 2432 Balance; and
9. Triple Beam Balance.

2.0 MONITORING METHODOLOGY

The methods used for radionuclide monitoring of stack emissions are modified versions to EPA methods; STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES - Revision to Reference Method 1-8: Thursday, August 18, 1977, Part II, as attached. Modifications to standard procedures are limited to:

1. Sample Location: Due to existing stack designs;
2. Filter Size: Use of 100mm dia glassfiber filter rather than 64mm dia. glassfiber filter;
3. Usage of 0.1N nitric acid as impinger solution instead of distilled water, as uranium is more soluble in nitric acid; and,
4. Usage of 0.1N nitric acid as glassware wash medium.

2.0 MONITORING METHODOLOGY (continued)

Stack data formats and basic equations utilized in stack sampling are as attached. All calculations are made using either the operating nomograph or by programs written for a Texas Instruments 59 calculator.

Operations of equipment guideline also is attached.

2.0 MONITORING METHODOLOGY

(continued)

REFERENCE METHODS 1-8

federal register

THURSDAY, AUGUST 18, 1977

PART II

THE NATIONAL ARCHIVES OF THE UNITED STATES • 1934 •

ENVIRONMENTAL PROTECTION AGENCY

■

STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Revision to Reference Method 1-8

RF 814-1

Title 40—Protection of Environment

CHAPTER I—ENVIRONMENTAL PROTECTION AGENCY

[FRL 754-5]

PART 60—STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Revision to Reference Methods 1–8

AGENCY: Environmental Protection Agency.

ACTION: Final Rule.

SUMMARY: This rule revises Reference Methods 1 through 8, the detailed requirements used to measure emissions from affected facilities to determine whether they are in compliance with a standard of performance. The methods were originally promulgated December 23, 1971, and since that time several revisions became apparent which would clarify, correct and improve the methods. These revisions make the methods easier to use, and improve their accuracy and reliability.

EFFECTIVE DATE: September 19, 1977.

ADDRESSES: Copies of the comment letters are available for public inspection and copying at the U.S. Environmental Protection Agency, Public Information Reference Unit (EPA Library), Room 29[illegible] [illegible]1 M Street, S.W., Washington, D.C. 20460. A summary of the comments and EPA's responses may be obtained upon written request from the EPA Public Information Center (PM-215), 401 M Street, S.W., Washington, D.C. 20460 (specify "Public Comment Summary: Revisions to Reference Methods 1–8 in Appendix A of Standards of Performance for New Stationary Sources").

FOR FURTHER INFORMATION CONTACT:

Don R. Goodwin, Emission Standards and Engineering Division, Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone No. 919-541-5271.

SUPPLEMENTARY INFORMATION: The amendments were proposed on June 8, 1976 (40 FR 23060). A total of 55 comment letters were received during the comment period—34 from industry, 15 from governmental agencies, and 6 from other interested parties. They contained numerous suggestions which were incorporated in the final revisions.

Changes common to all eight of the reference methods are: (1) the clarification of procedures and equipment specifications resulting from the comments, (2) the addition of guidelines for alternative procedures and equipment to make prior approval of the Administrator unnecessary and (3) the addition of an introduction to each reference method discussing the general use of the method and delineating the procedure for using alternative methods and equipment.

Specific changes to the methods are:

METHOD 1

1. The provision for the use of more than two traverse diameters, when specified by the Administrator, has been deleted. If one traverse diameter is in a plane containing the greatest expected concentration variation, the intended purpose of the deleted paragraph will be fulfilled.

2. Based on recent data from Fluidyne (Particulate Sampling Strategies for Large Power Plants Including Nonuniform Flow, EPA-600/2-76-170, June 1976) and Entropy Environmentalists (Determination of the Optimum Number of Traverse Points: An Analysis of Method 1 Criteria (draft), Contract No. 68-01-3172), the number of traverse points for velocity measurements has been reduced and the 2:1 length to width ratio requirement for cross-sectional layout of rectangular ducts has been replaced by a "balanced matrix" scheme.

3. Guidelines for sampling in stacks containing cyclonic flow and stacks smaller than about 0.31 meter in diameter or 0.071 m^2 in cross-sectional area will be published at a later date.

4. Clarification has been made as to when a check for cyclonic flow is necessary; also, the suggested procedure for determination of unacceptable flow conditions has been revised.

METHOD 2

1. The calibration of certain pitot tubes has been made optional. Appropriate construction and application guidelines have been included.

2. A detailed calibration procedure for temperature gauges has been included.

3. A leak check procedure for pitot lines has been included.

METHOD 3

1. The applicability of the method has been confined to fossil-fuel combustion processes and to other processes where it has been determined that components other than O_2, CO_2, CO, and N_2 are not present in concentrations sufficient to affect the final results.

2. Based on recent research information (Particulate Sampling Strategies for Large Power Plants Including Nonuniform Flow, EPA-600/2-76-170, June 1976), the requirement for proportional sampling has been dropped and replaced with the requirement for constant rate sampling. Proportional and constant rate sampling have been found to give essentially the same result.

3. The "three consecutive" requirement has been replaced by "any three" for the determination of molecular weight, CO_2 and O_2.

4. The equation for excess air has been revised to account for the presence of CO.

5. A clearer distinction has been made between molecular weight determination and emission rate correction factor determination.

6. Single point, integrated sampling has been included.

METHOD 4

1. The sampling time of 1 hour has been changed to a total sampling time which will span the length of time the pollutant emission rate is being determined or such time as specified in an applicable subpart of the standards.

2. The requirement for proportional sampling has been dropped and replaced with the requirement for constant rate sampling.

3. The leak check before the test run has been made optional; the leak check after the run remains mandatory.

METHOD 5

1. The following alternatives have been included in the method:

a. The use of metal probe liners.

b. The use of other materials of construction for filter holders and probe liner parts.

c. The use of polyethylene wash bottles and sample storage containers.

d. The use of desiccants other than silica gel or calcium sulfate, when appropriate.

e. The use of stopcock grease other than silicone grease, when appropriate.

f. The drying of filters and probe-filter catches at elevated temperatures, when appropriate.

g. The combining of the filter and probe washes into one container.

2. The leak check prior to a test run has been made optional. The post-test leak check remains mandatory. A method for correcting sample volume for excessive leakage rates has been included.

3. Detailed leak check and calibration procedures for the metering system have been included.

METHOD 6

1. Possible interfering agents of the method have been delineated.

2. The options of: (a) using a Method 8 impinger system, or (b) determining SO_2 simultaneously with particulate matter, have been included in the method.

3. Based on recent research data, the requirement for proportional sampling has been dropped and replaced with the requirement for constant rate sampling.

4. Tests have shown that isopropanol obtained from commercial sources occasionally has peroxide impurities that will cause erroneously low SO_2 measurements. Therefore, a test for detecting peroxides in isopropanol has been included in the method.

5. The leak check before the test run has been made optional; the leak check after the run remains mandatory.

6. A detailed calibration procedure for the metering system has been included in the method.

METHOD 7

1. For variable wave length spectrophotometers, a scanning procedure for determining the point of maximum absorbance has been incorporated as an option.

METHOD 8

1. Known interfering compounds have been listed to avoid misapplication of the method.

2. The determination of filterable particulate matter (including acid mist) simultaneously with SO_2 and SO_3 has been allowed where applicable.

3. Since occasionally some commercially available quantities of isopropanol

have peroxide impurities that will cause erroneously high sulfuric acid mist measurements, a test for peroxides in isopropanol has been included in the method.

4. The gravimetric technique for moisture content (rather than volumetric) has been specified because a mixture of isopropyl alcohol and water will have a volume less than the sum of the volumes of its content.

5. A closer correspondence has been made between similar parts of Methods 8 and 5.

MISCELLANEOUS

Several commenters questioned the meaning of the term "subject to the approval of the Administrator" in relation to using alternate test methods and procedures. As defined in § 60.2 of subpart A, the "Administrator" includes any authorized representative of the Administrator of the Environmental Protection Agency. Authorized representatives are EPA officials in EPA Regional Offices or State, local, and regional governmental officials who have been delegated the responsibility of enforcing regulations under 40 CFR 60. These officials in consultation with other staff members familiar with technical aspects of source testing will render decisions regarding acceptable alternate test procedures.

In accordance with section 117 of the Act, publication of these methods was preceded by consultation with appropriate advisory committees, independent experts, and Federal departments and agencies.

(Secs. 111, 114 and 301(a) of the Clean Air Act, sec. 4(a) of Pub. L. No. 91-604, 84 Stat. 1683; sec. 4(a) of Pub. L. No. 91-604, 84 Stat. 1687; sec. 2 of Pub. L. No. 90-148, 81 Stat. 504 [42 U.S.C. 1857c-6, 1857c-9, 1857g(a)].)

NOTE.—The Environmental Protection Agency has determined that this document does not contain a major proposal requiring preparation of an Economic Impact Analysis under Executive Orders 11821 and 11949 and OMB Circular A-107.

Dated: August 10, 1977.

DOUGLAS M. COSTLE,
Administrator.

Part 60 of Chapter I of Title 40 of the Code of Federal Regulations is amended by revising Methods 1 through 8 of Appendix A—Reference Methods as follows:

APPENDIX A—REFERENCE METHODS

The reference methods in this appendix are referred to in § 60.8 (Performance Tests) and § 60.11 (Compliance With Standards and Maintenance Requirements) of 40 CFR Part 60, Subpart A (General Provisions). Specific uses of these reference methods are described in the standards of performance contained in the subparts, beginning with Subpart D.

Within each standard of performance, a section titled "Test Methods and Procedures" is provided to (1) identify the test methods applicable to the facility subject to the respective standard and (2) identify any special instructions or conditions to be followed when applying a method to the respective facility. Such instructions (for example, establish sampling rates, volumes, or temperatures) are to be used either in addition to, or as a substitute for procedures in a reference method. Similarly, for sources subject to emission monitoring requirements, specific instructions pertaining to any use of a reference method are provided in the subpart or in Appendix B.

Inclusion of methods in this appendix is not intended as an endorsement or denial of their applicability to sources that are not subject to standards of performance. The methods are potentially applicable to other sources; however, applicability should be confirmed by careful and appropriate evaluation of the conditions prevalent at such sources.

The approach followed in the formulation of the reference methods involves specifications for equipment, procedures, and performance. In concept, a performance specification approach would be preferable in all methods because this allows the greatest flexibility to the user. In practice, however, this approach is impractical in most cases because performance specifications cannot be established. Most of the methods described herein, therefore, involve specific equipment specifications and procedures, and only a few methods in this appendix rely on performance criteria.

Minor changes in the reference methods should not necessarily affect the validity of the results and it is recognized that alternative and equivalent methods exist. Section 60.8 provides authority for the Administrator to specify or approve (1) equivalent methods, (2) alternative methods, and (3) minor changes in the methodology of the reference methods. It should be clearly understood that unless otherwise identified all such methods and changes must have prior approval of the Administrator. An owner employing such methods or deviations from the reference methods without obtaining prior approval does so at the risk of subsequent disapproval and retesting with approved methods.

Within the reference methods, certain specific equipment or procedures are recognized as being acceptable or potentially acceptable and are specifically identified in the methods. The items identified as acceptable options may be used without approval but must be identified in the test report. The potentially approvable options are cited as "subject to the approval of the Administrator" or as "or equivalent." Such potentially approvable techniques or alternatives may be used at the discretion of the owner without prior approval. However, detailed descriptions for applying these potentially approvable techniques or alternatives are not provided in the reference methods. Also, the potentially approvable options are not necessarily acceptable in all applications. Therefore, an owner electing to use such potentially approvable techniques or alternatives is responsible for: (1) assuring that the techniques or alternatives are in fact applicable and are properly executed; (2) including a written description of the alternative method in the test report (the written method must be clear and must be capable of being performed without additional instruction, and the degree of detail should be similar to the detail contained in the reference methods); and (3) providing any rationale or supporting data necessary to show the validity of the alternative in the particular application. Failure to meet these requirements can result in the Administrator's disapproval of the alternative.

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:

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

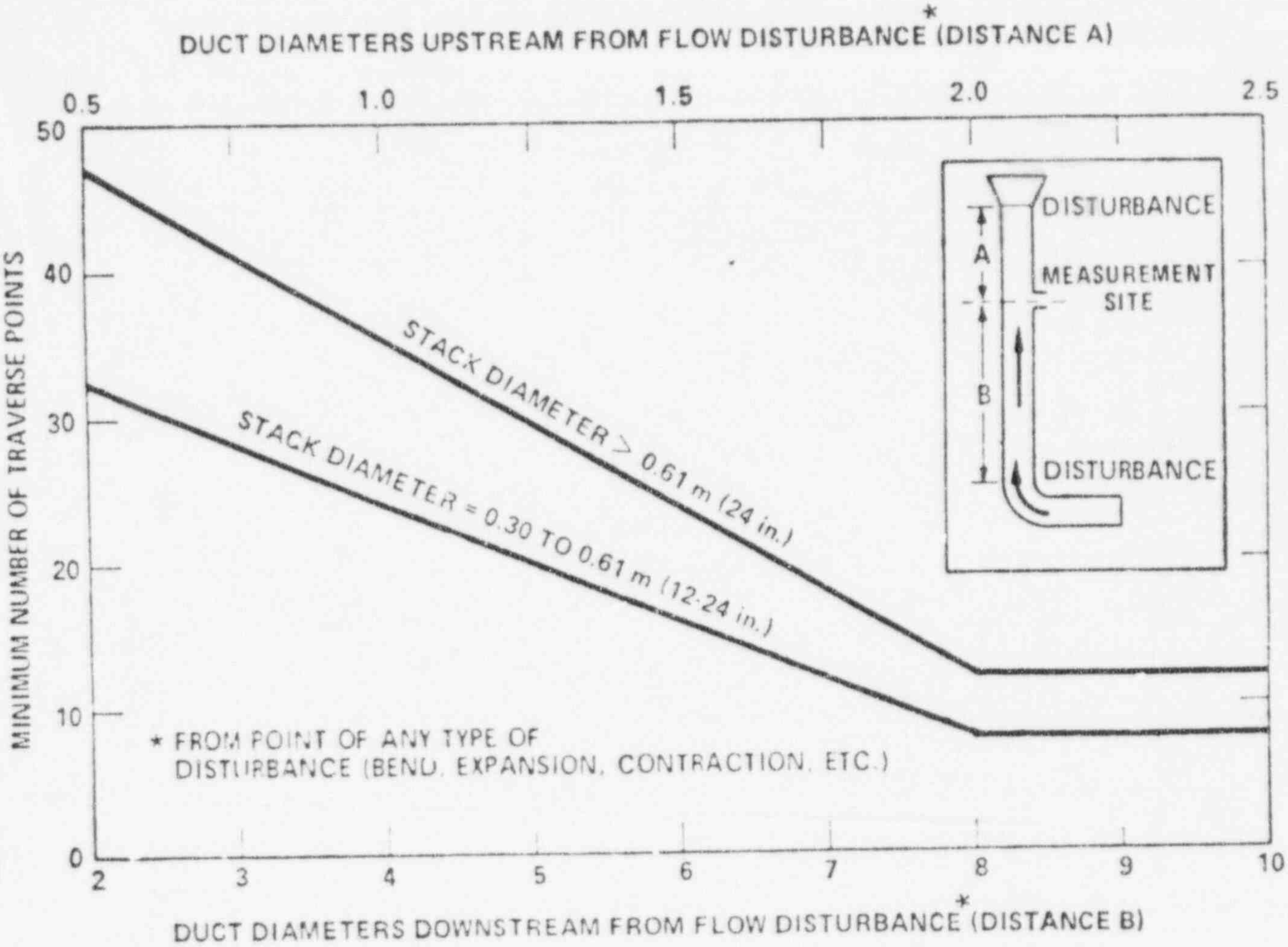


Figure 1-1. Minimum number of traverse points for particulate traverses.

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.); (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 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*

Number of traverse points	*Matrix layout*
9	3x3
12	4x3
16	4x4
20	5x4
25	5x5
30	6x5
36	6x6
42	7x6
49	7x7

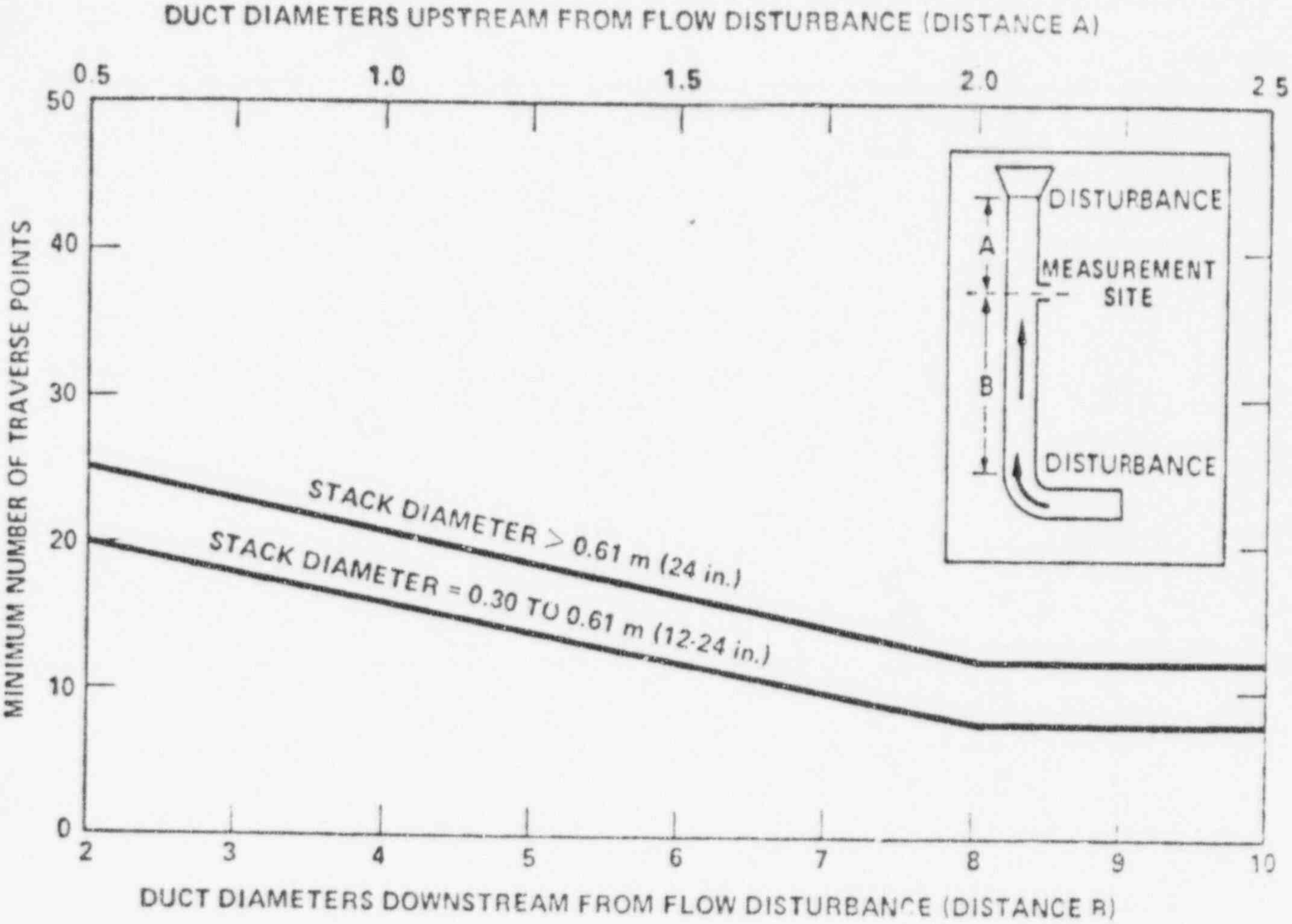


Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.

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 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.

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

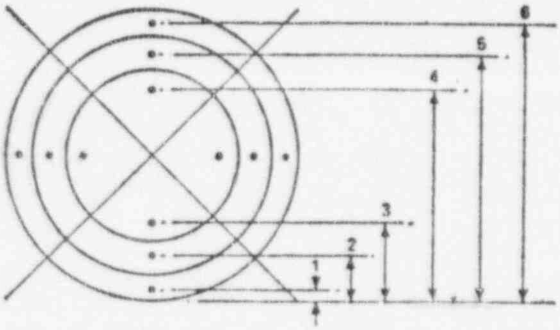


Figure 1-3. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points indicated.

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

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.

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.

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.

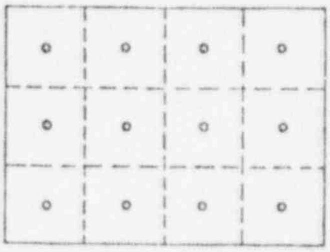


Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with a traverse point at centroid of each area.

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 ±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.

3. *Bibliography*

1. Determining Dust Concentration in a Gas Stream. ASME. Performance Test Code No. 27. New York. 1957.

2. Devorkin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District. Los Angeles, CA. November 1963.

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

4. Standard Method for Sampling Stacks for Particulate Matter. In: 1971 Book of ASTM Standards, Part 23. ASTM Designation D-2928-71. Philadelphia, Pa. 1971.

5. Hanson, H. A., et al. Particulate Sampling Strategies for Large Power Plants Including Nonuniform Flow. USEPA, ORD, ESRL, Research Triangle Park, N.C. EPA-600/2-76-170. June 1976.

6. Entropy Environmentalists, Inc. Determination of the Optimum Number of Sampling Points: An Analysis of Method 1 Criteria. Environmental Protection Agency. Research Triangle Park, N.C. EPA Contract No. 68-01-3172, Task 7.

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.

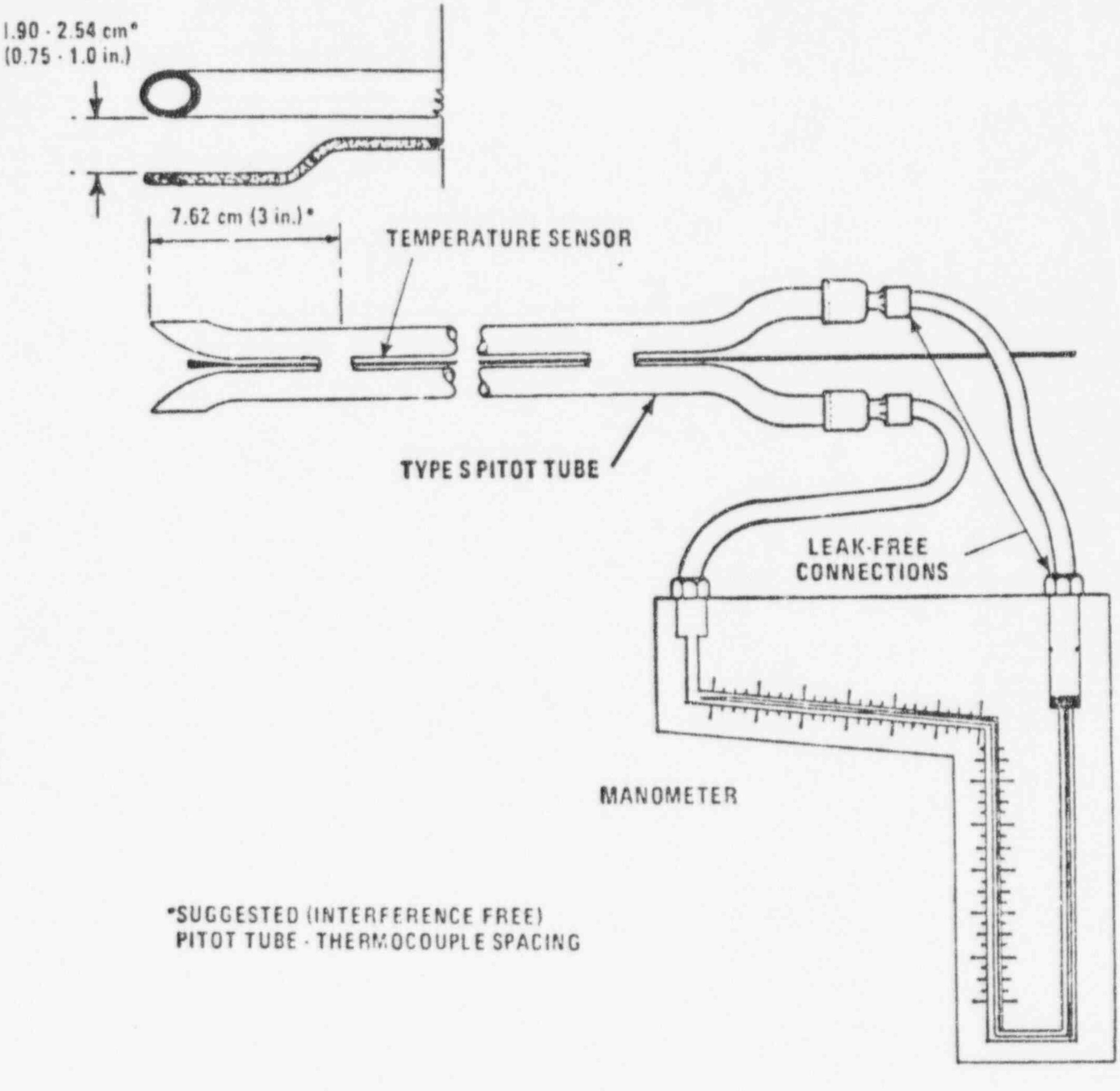


Figure 2-1. Type S pitot tube manometer assembly.

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 between 0.48 and 0.95 centimeters ($\frac{3}{16}$ and $\frac{3}{8}$ 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.

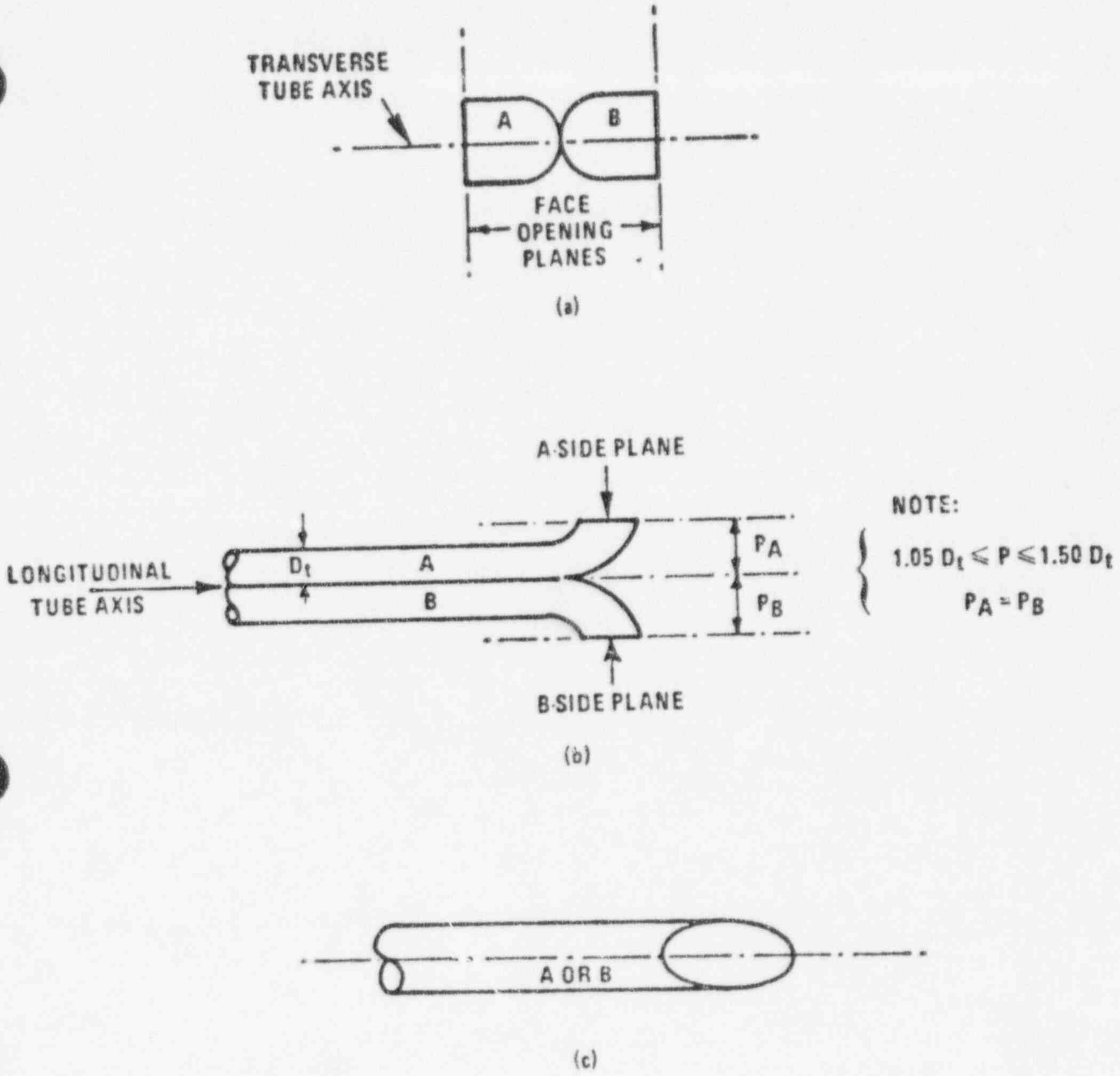


Figure 2-2. Properly constructed Type S pitot tube, shown in: (a) end view; face opening planes perpendicular to transverse axis; (b) top view; face opening planes parallel to longitudinal axis; (c) side view; both legs of equal length and centerlines coincident, when viewed from both sides. Baseline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

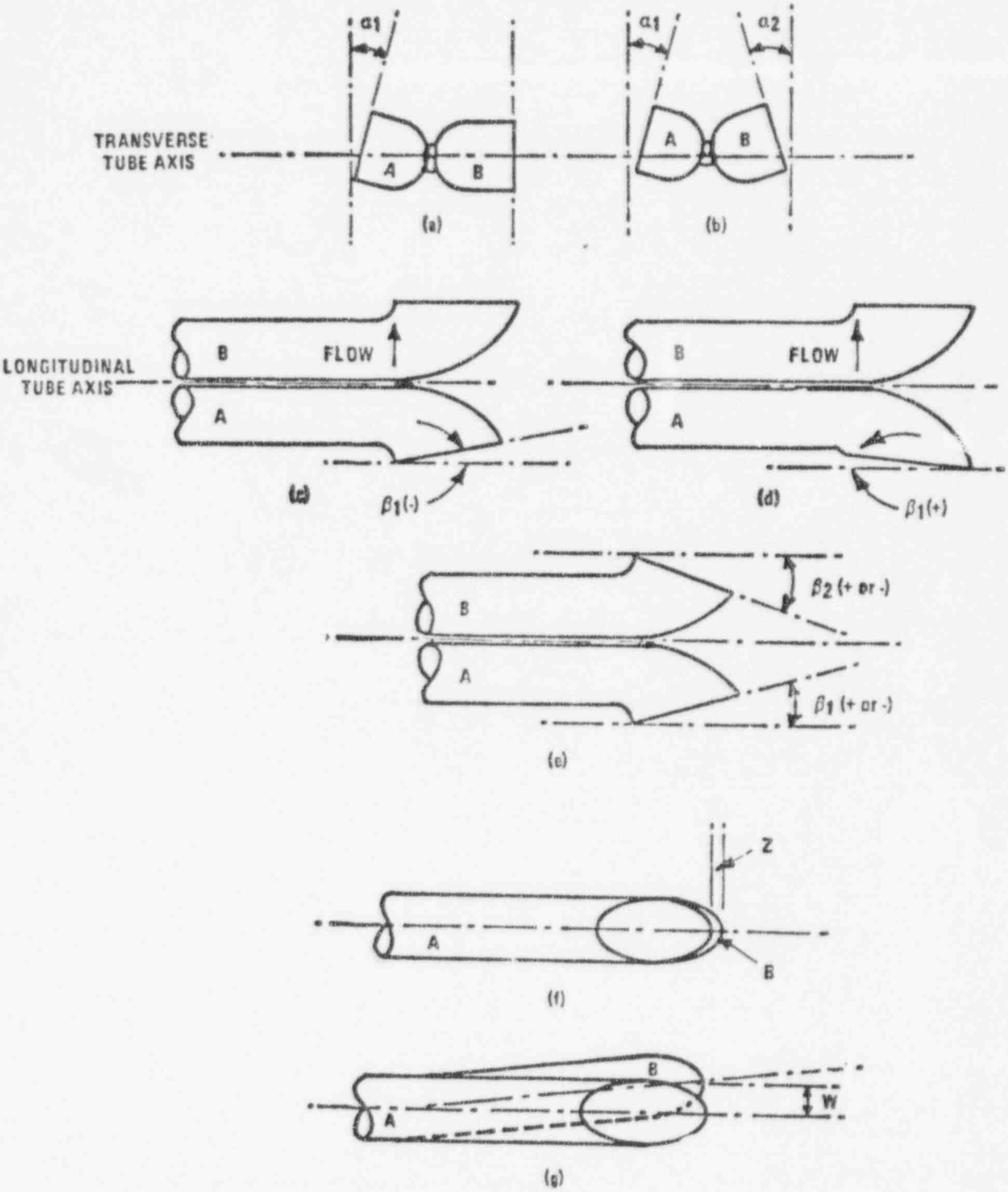


Figure 2-3. Types of face-opening misalignment that can result from field use or improper construction of Type S pitot tubes. These will not affect the baseline value of $\bar{C}p(s)$ so long as α_1 and $\alpha_2 < 10^{\circ}$, β_1 and $\beta_2 < 5^{\circ}$, $z < 0.32$ cm (1/8 in.) and $w < 0.08$ cm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

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 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±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).

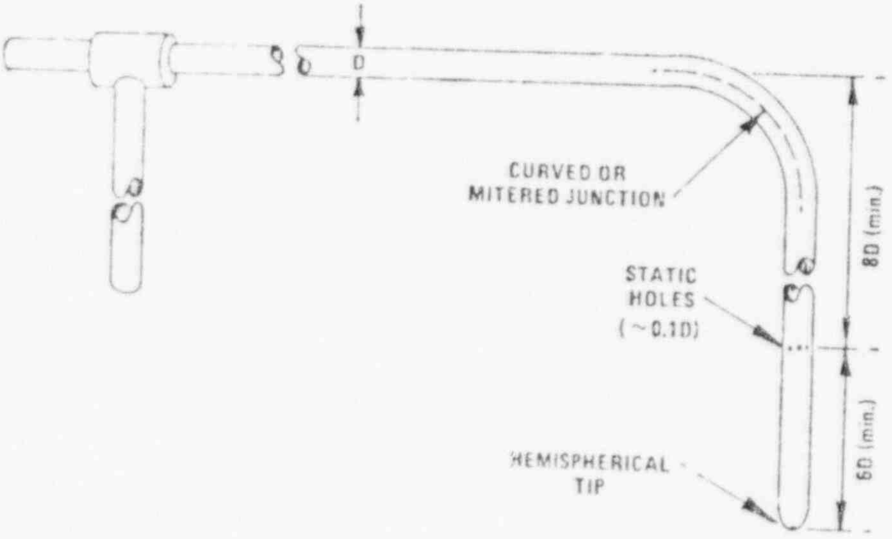


Figure 2-4. Standard pitot tube design specifications.

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.

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.

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

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

3.6 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.

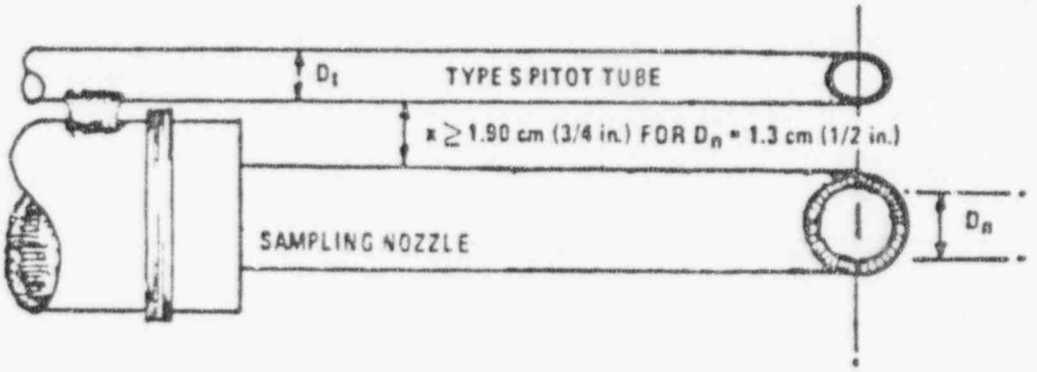
After verifying the face opening alignment, measure and record the following dimensions of the pitot tube: (a) the external tubing diameter (dimension D_t, Figure 2-2b); and (b) the base-to-opening plane distances (dimensions P_A and P_B, Figure 2-2b). If D_t is between 0.48 and 0.95 cm (3/16 and 3/8 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.

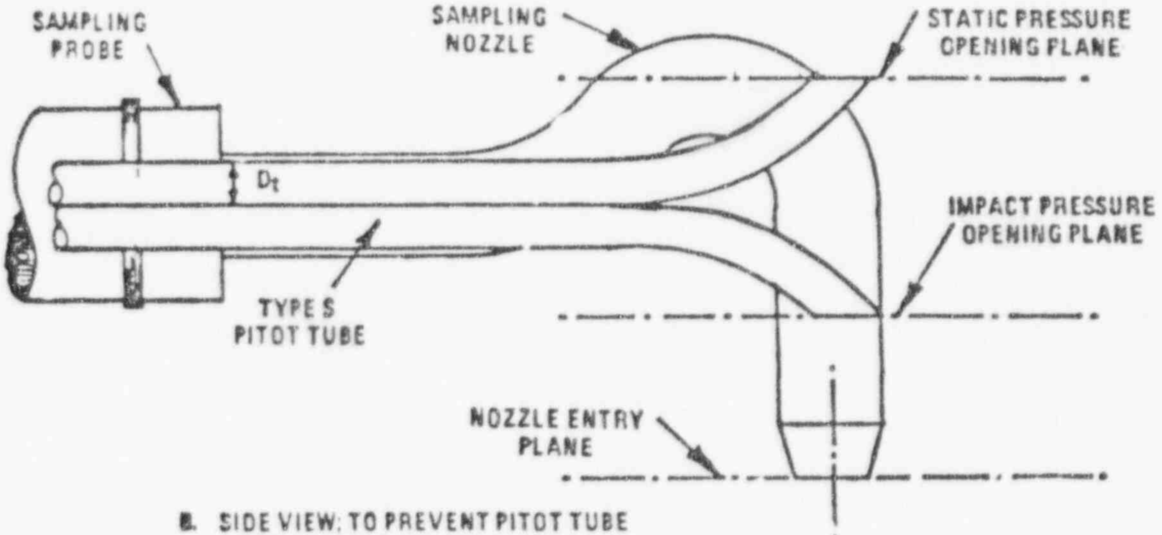
4.1.1 Type S Pitot Tube Assemblies. During sample and velocity traverses, the isolated Type S pitot tube is not always used; in many instances, the pitot tube is used in combination with other source-sampling components (thermocouple, sampling probe, nozzle) as part of an "assembly." The presence of 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:



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.).

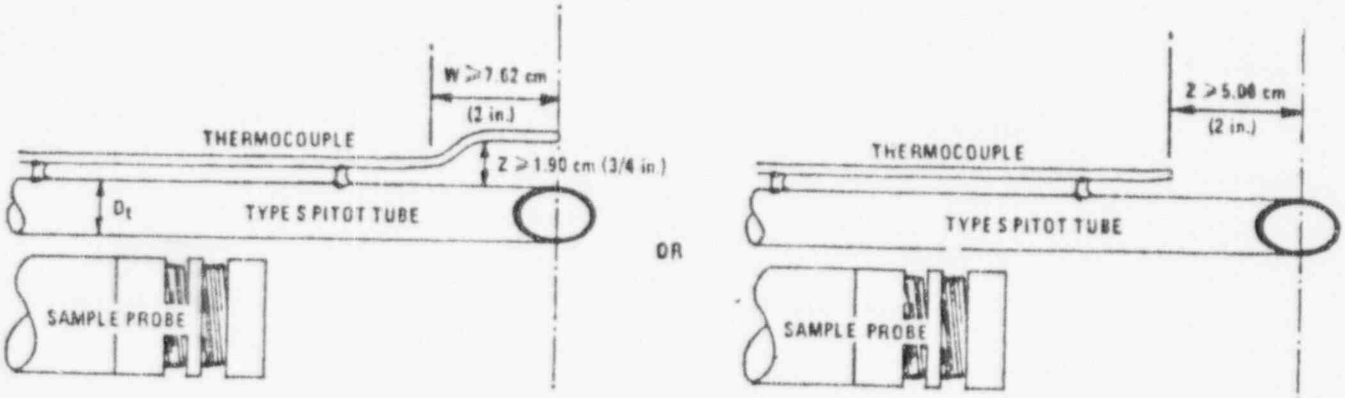


Figure 2-7. Proper thermocouple placement to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

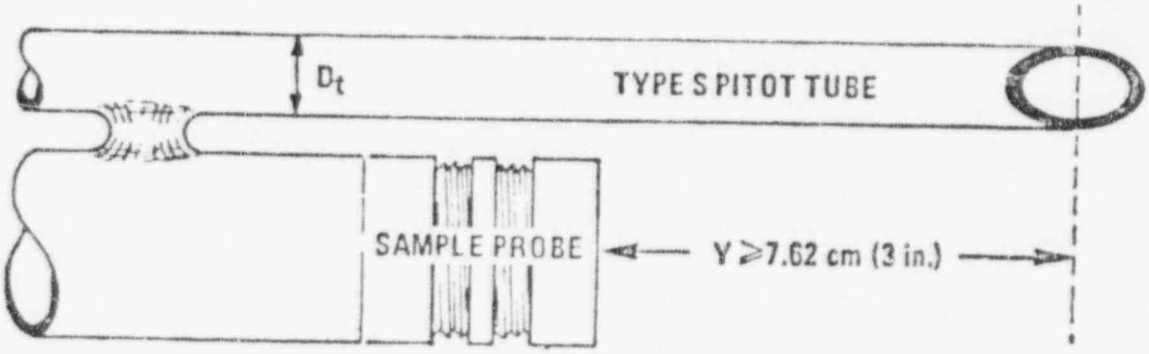


Figure 2-8. Minimum pitot-sample probe separation needed to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

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

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

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 (3,000 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 (3,000 ft/min) will generally be valid to within ±3 percent for the measurement of velocities above 305 m/min (1,000 ft/min) and to within ±5 to 6 percent for the measurement of velocities between 180 and 305 m/min (600 and 1,000 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 1,525 m/min (600 to 5,000 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:

ITOT TUBE IDENTIFICATION NUMBER: ______________ DATE: __________

LIBRATED 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)		

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

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

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 $\bar{C}_p$ (side A), the mean A-side coefficient, and $\bar{C}_p$ (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 $\bar{C}_p$ (side A), and the deviation of each B-side value of $C_{p(s)}$ from $\bar{C}_p$ (side B). Use the following equation:

$$\text{Deviation} = C_{p(s)} - \bar{C}_p(\text{A or B})$$

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:

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

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 $\bar{C}_p$ (A) and $\bar{C}_p$ (B) is 0.01 or less.

4.1.5 Special considerations.

4.1.5.1 Selection of calibration point.

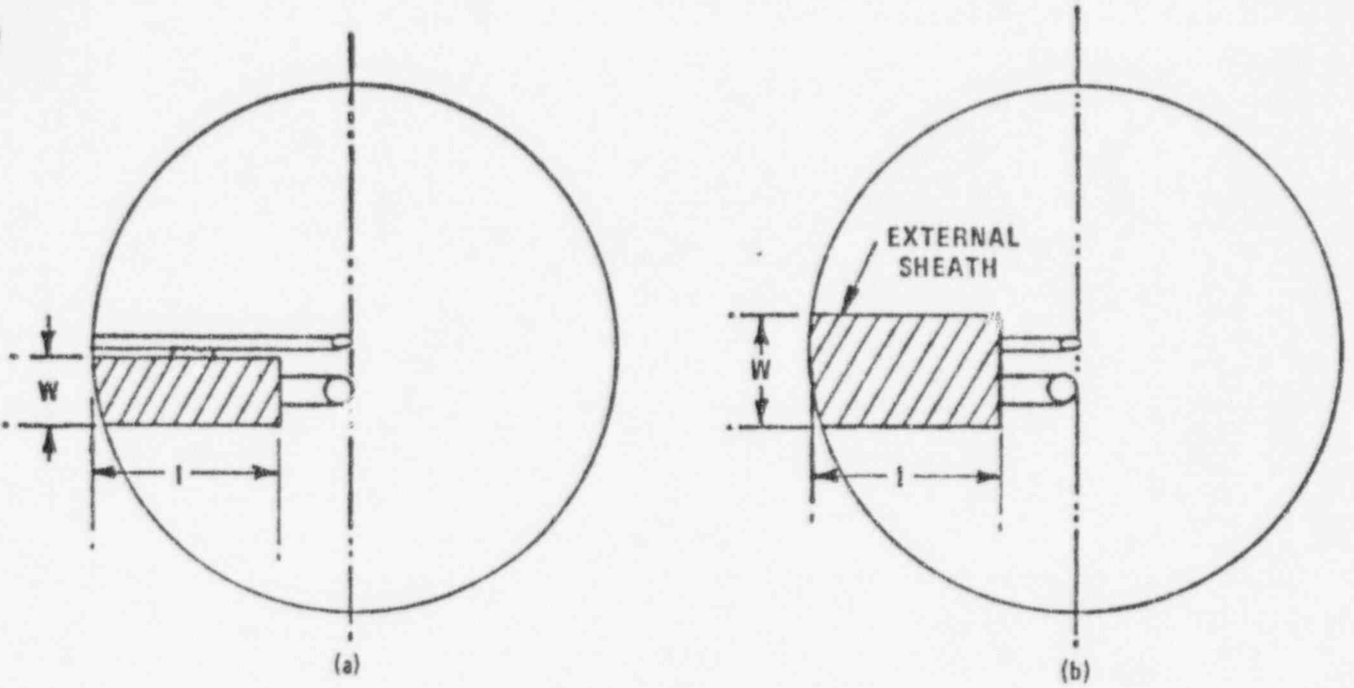
4.1.5.1.1 When an isolated Type S pitot tube is calibrated, select a calibration point at or near the center of the duct, and follow the procedures outlined in sections 4.1.3 and 4.1.4 above. The Type S pitot coefficients so obtained, i.e., $\bar{C}_p$ (side A) and $\bar{C}_p$ (side B), will be valid, 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 ¼ in.) are not ordinarily used for isokinetic sampling at velocities around 915 m/min (3,000 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, m² (ft²).

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,

$$34.97\ \frac{\text{m}}{\text{sec}}\left[\frac{(g/g\text{-mole})(\text{mm Hg})}{(°\text{K})(\text{mm H}_2\text{O})}\right]^{1/2}$$

for the metric system and

$$85.49\ \frac{\text{ft}}{\text{sec}}\left[\frac{(\text{lb/lb-mole})(\text{in. Hg})}{(°\text{R})(\text{in. H}_2\text{O})}\right]^{1/2}$$

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).

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

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).

$$= 273 + t_s \text{ for metric} \qquad \text{Equation 2-7}$$

$$= 460 + t_s \text{ for English} \qquad \text{Equation 2-8}$$

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 H₂O (in. H₂O).

3,600 = Conversion factor, sec/hr.

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

5.2 Average stack gas velocity.

$$v_s = K_p C_p (\sqrt{\Delta p})_{avg} \sqrt{\frac{T_{s(avg)}}{P_s M_s}}$$

Equation 2-9

5.3 Average stack gas dry volumetric flow rate.

$$Q_{sd} = 3{,}600(1 - B_{ws}) v_s A \left(\frac{T_{std}}{T_{s(avg)}}\right)\left(\frac{P_s}{P_{std}}\right)$$

Equation 2-10

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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 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.

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.

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

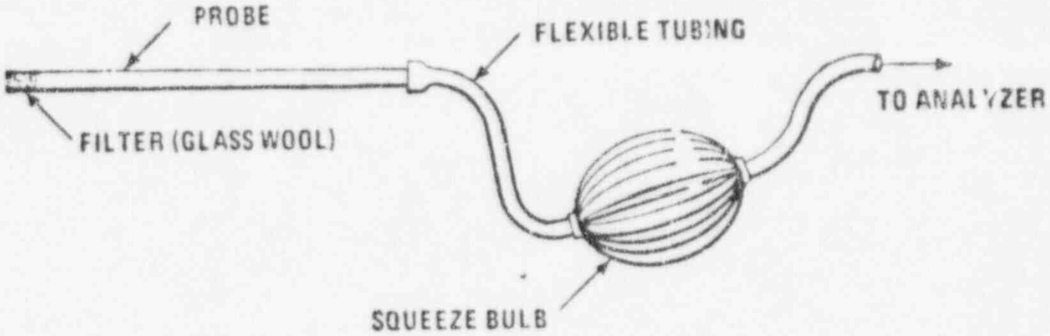

Figure 3-1. Grab sampling train.

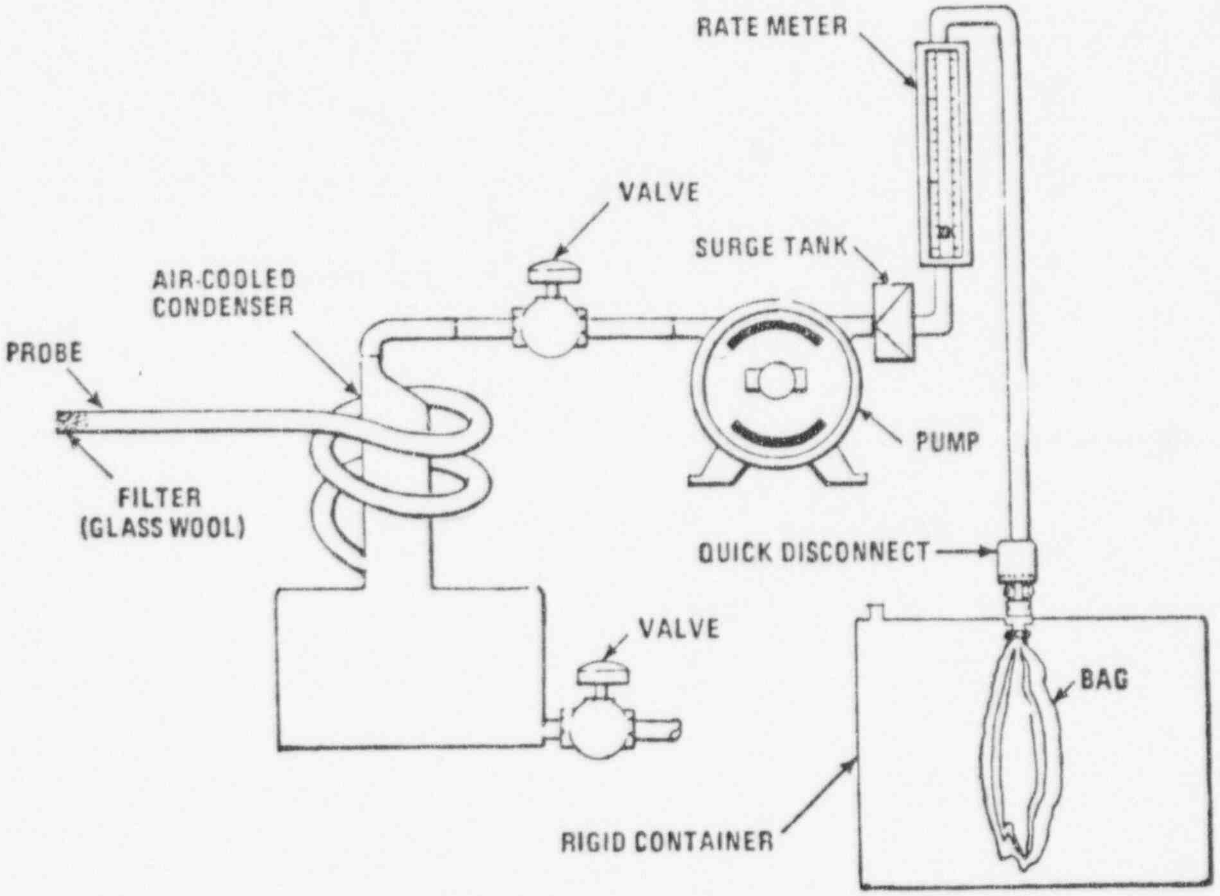

Figure 3-2. Integrated gas sampling train.

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 leaked-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 (0.1 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, a minimum of eight traverse points shall be used for circular stacks having diameters less then 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 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.

TIME	TRAVERSE PT.	Q 1pm	% DEV.^{a}
	AVERAGE		

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

Figure 3.3. Sampling rate data.

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 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 ±0.3 percent by volume when O_2 is less than 15.0 percent or ±0.2 percent by volume when O_2 is greater than 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 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 Method 1. 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.

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

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.

$$M_d=0.440(\%CO_2)+0.320(\%O_2)+0.280(\%N_2+\%CO)$$

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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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 ±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.

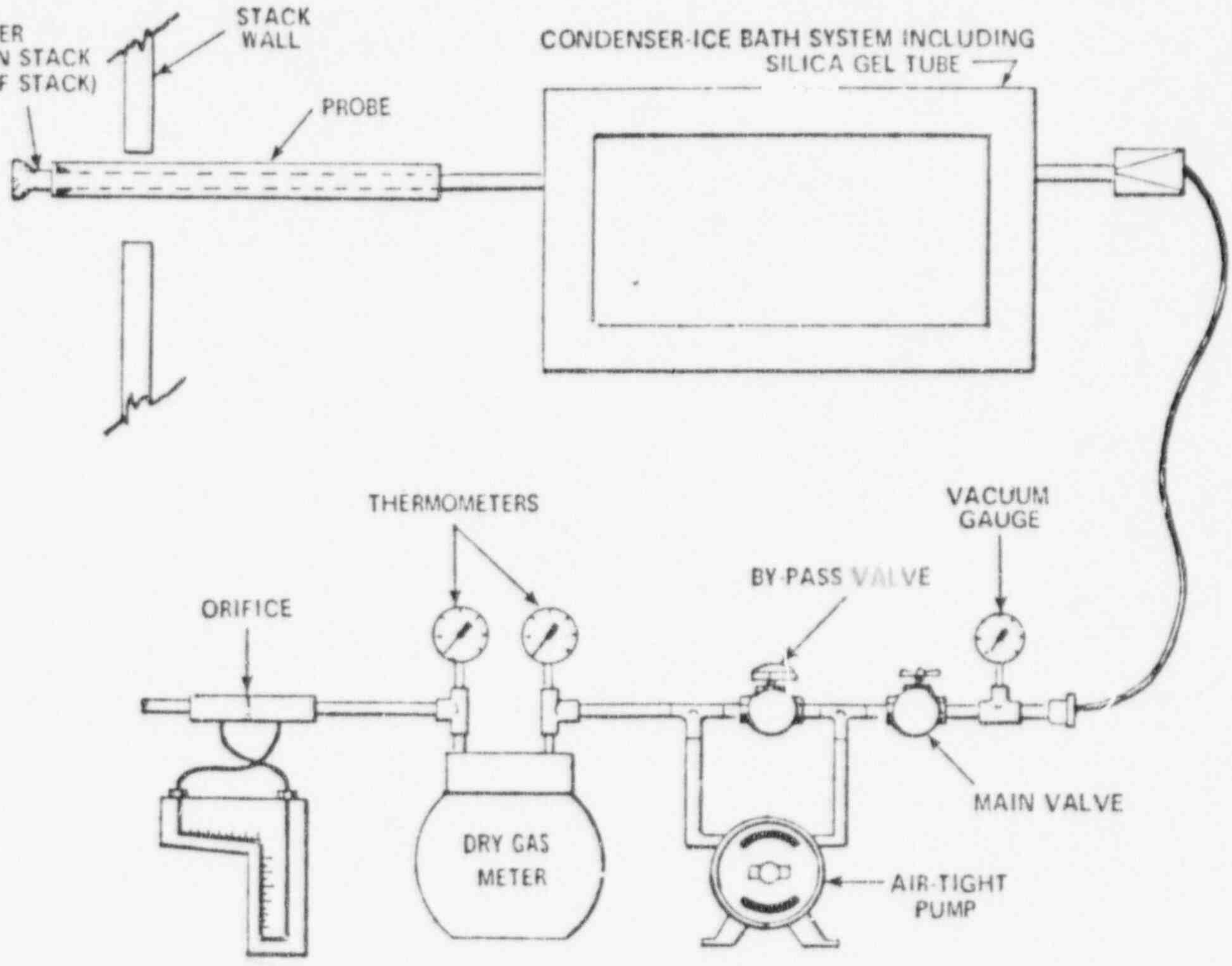

Figure 4-1. Moisture sampling train-reference method.

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 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 (½ inch) ID glass tube extending to about 1.3 cm (½ 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 travers 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 m³/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 m³/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 ($T_{m_{in}}$), °C (°F)	OUTLET ($T_{m_{out}}$), °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) (m³)/(g-mole) (°K) for metric units and 21.85 (in. Hg) (ft³)/(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.

$$V_{wc(std)} = \frac{(V_f - V_i)\rho_w R T_{std}}{P_{std} M_w} = K_1(V_f - V_i)$$

Equation 4-1

where:

K_1 = 0.001333 m³/ml for metric units
= 0.04707 ft³/ml for English units

2.3.3 Volume of water vapor collected in silica gel.

$$V_{wsg(std)} = \frac{(W_f - W_i) R T_{std}}{P_{std} M_w} = K_2(W_f - W_i)$$

Equation 4-2

where:

K_2 = 0.001335 m³/g for metric units
= 0.04715 ft³/g for English units

2.3.4 Sample gas volume.

$$V_{m(std)} = V_m Y \frac{(P_m)(T_{std})}{(P_{std})(T_m)} = K_3 Y \frac{V_m P_m}{T_m}$$

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.

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

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. Assemble the apparatus without the probe as shown in Figure 4-4. Leak check the train by placing a vacuum gauge at the inlet to the first impinger and drawing a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet of the rotameter, and then turning off the pump. The vacuum shall remain constant for at least one minute. Carefully release the vacuum gauge before unplugging the rotameter end.

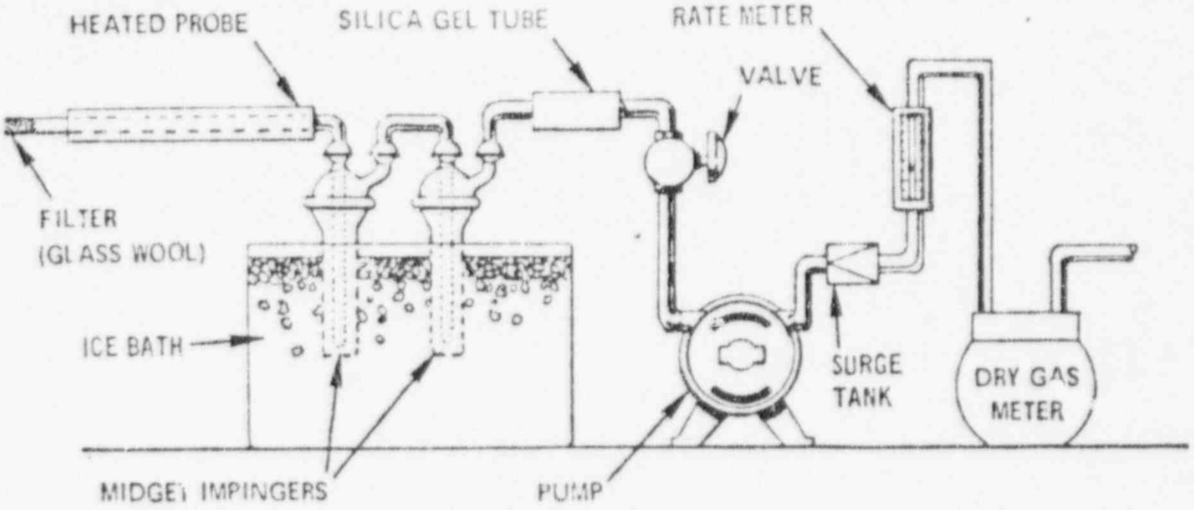

Figure 4-4. Moisture sampling train - approximation method.

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 ft³) 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) (m³)/(g-mole) (°K) for metric units and 21.85 (in. Hg) (ft³)/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).
ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).

3.3.2 Volume of water vapor collected.

$$V_{wc}=\frac{(V_f-V_i)\rho_w RT_{std}}{P_{std}M_w}$$
$$=K_1(V_f-V_i)$$

Equation 4-5

where:
K_1=0.001333 m³/ml for metric units
=0.04707 ft³/ml for English units.

3.3.3 Gas volume.

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

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.

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

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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2. Devorkin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District, Los Angeles, Calif. November, 1963.

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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 [illegible] 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 [illegible]). 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:

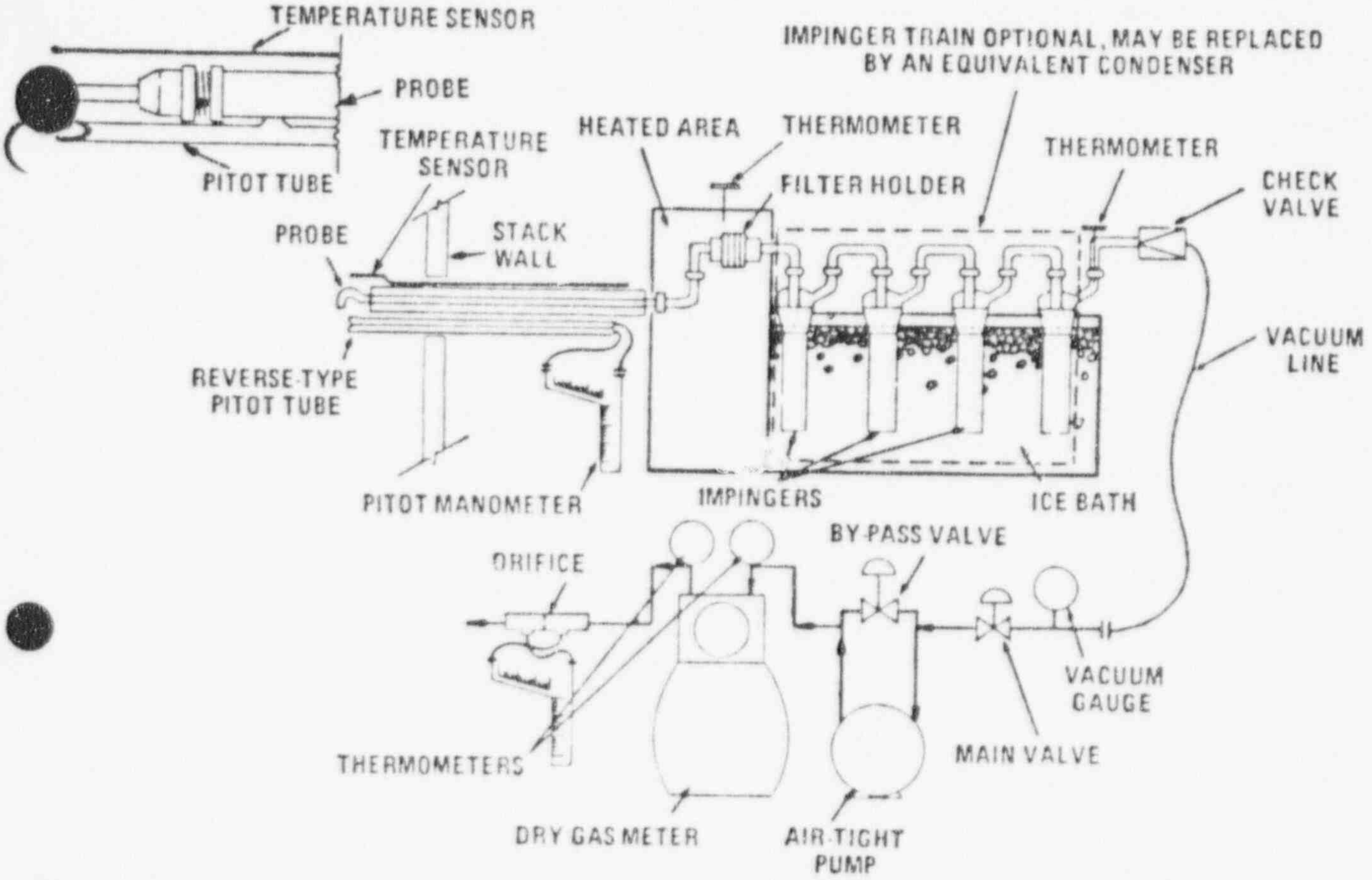

Figure 5 1. Particulate sampling train.

1.1 Probe Nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper shall be ≤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 (⅛ to ½ 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±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. (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.

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 1,650° 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 (1,508° F), and for quartz it is 1,500° C (2,732° 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,[2] 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.

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 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 1.3 cm (½ in.) ID glass tube extending to about 1.3 cm (½ 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 content.

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

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

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

10 Gas Density Determination Equipment. perature sensor and pressure gauge, as described 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 attached 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 esistant, 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 nsfer of silica gel to container; not necessary if silica 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 (≤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.

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 weighing.

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.

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±14° C (248±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 [illegible]ation in velocity head readings) necessitate addi[illegible]l adjustments in flow rate. Level and zero the [illegible]ometer. Because the manometer level and zero may [illegible]t due to vibrations and temperature changes, make [illegible]iodic 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 [illegible]cations within the same duct, or, in cases where equip[illegible]ent failure necessitates a change of trains. In all other [illegible]uations, the use of two or more trains will be subject to [illegible]e 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 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.

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).

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

Figure 5-3. Analytical data.

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 m³/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 m³/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.

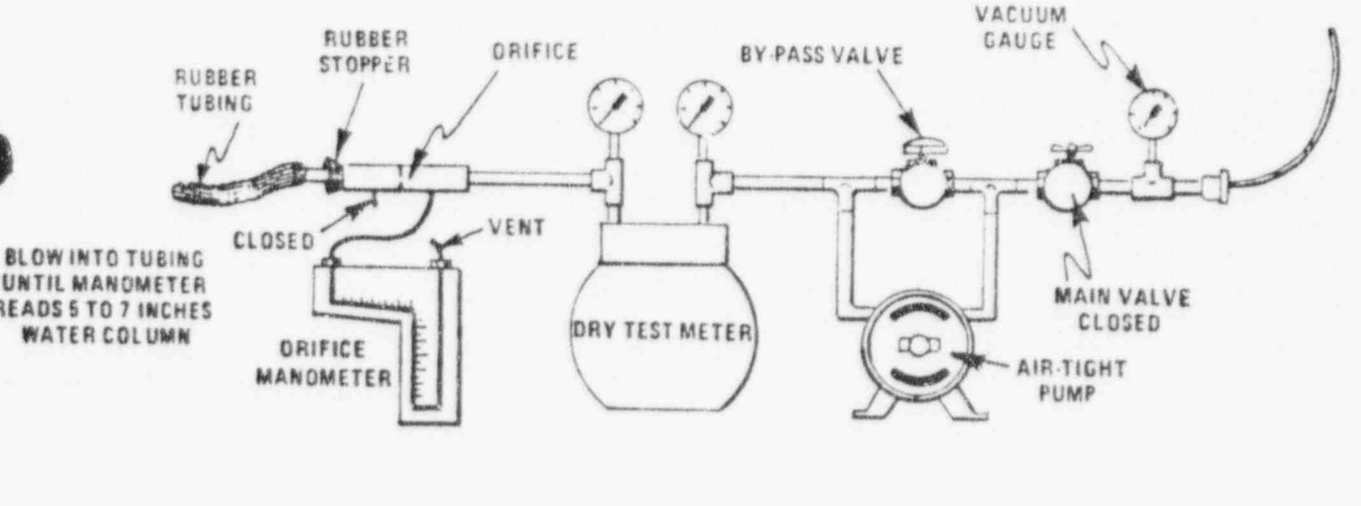

Figure 5-4. Leak check of meter box.

6.1 Nomenclature

A_n = Cross-sectional area of nozzle, m² (ft²).

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

C_a = Acetone blank residue concentrations, 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.

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 m³/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), m³/min (cfm).

L_p = Leakage rate observed during the post-test leak check, m³/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-m³/°K-g-mole (21.85 in. Hg-ft³/°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:
$m_1 = 0.3858$ °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:

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

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

6.4 Volume of water vapor.

$$V_{w(std)} = V_{lc}\left(\frac{\rho_w}{M_w}\right)\left(\frac{RT_{std}}{P_{std}}\right) = K_2V_{lc}$$

Equation 5-2

where:
$K_2 = 0.001333$ m³/ml for metric units
$= 0.04707$ ft³/ml for English units.

6.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

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 ± 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.

$$c_s = (0.001\ g/mg)\,(m_n/V_{m(std)})$$

Equation 5-6

6.10 Conversion Factors.

From	To	Multiply by
scf	m³	0.02832
g/ft³	gr/ft³	15.43
g/ft³	lb/ft³	2.205×10^{-3}
g/ft³	g/m³	35.31

6.11 Isokinetic Variation.

6.11.1 Calculation From Raw Data.

$$I = \frac{100\,T_s[K_3V_{lc} + (V_m/T_m)\,(P_{bar} + \Delta H/13.6)]}{60\,\theta v_s P_s A_n}$$

Equation 5-7

where:
$K_3 = 0.003454$ mm Hg-m³/ml-°K for metric units.
$= 0.002669$ in. Hg-ft³/ml-°R for English units.

6.11.2 Calculation From Intermediate Values.

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

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

Equation 5-8

where:
$K_4 = 4.320$ for metric units
$= 0.09450$ for English units.

6.12 Acceptable Results. If 90 percent ≤ I ≤ 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.

7. *Bibliography*

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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/m³ (2.12×10^{-7} lb/ft³). Although no upper limit has been established, tests have shown that concentrations as high as 80,000 mg/m³ 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/m³.

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 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*

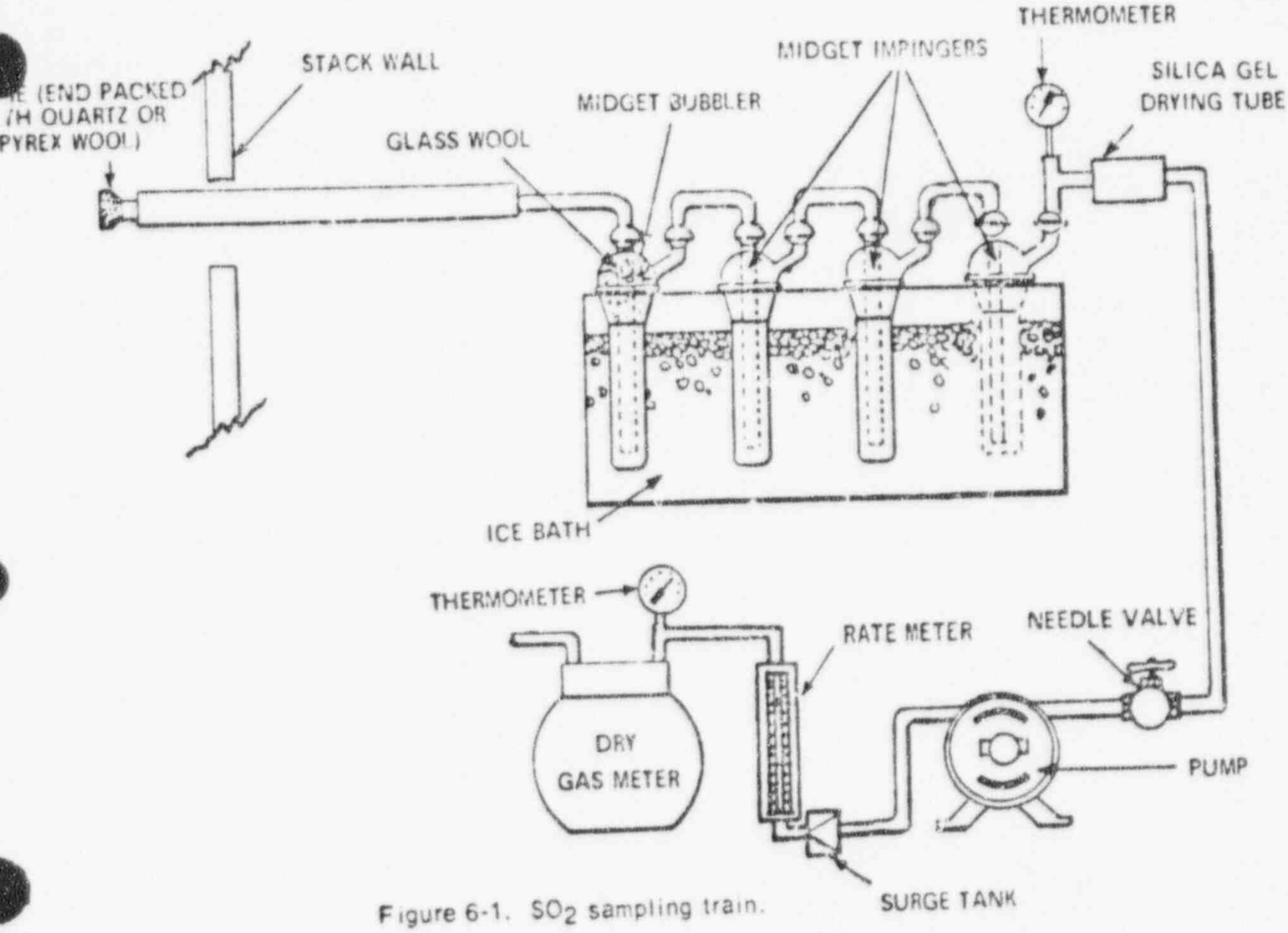

Figure 6-1. SO_2 sampling train.

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 siliac 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 Value. Needle value, 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 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. At least 760 mm Hg (30 in. Hg) gauge, 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. 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 ±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:

With the probe disconnected, place a vacuum gauge at the inlet to the bubbler 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 to prevent back flow of the impinger fluid.

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. 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 or 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.

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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[2] 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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[2] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

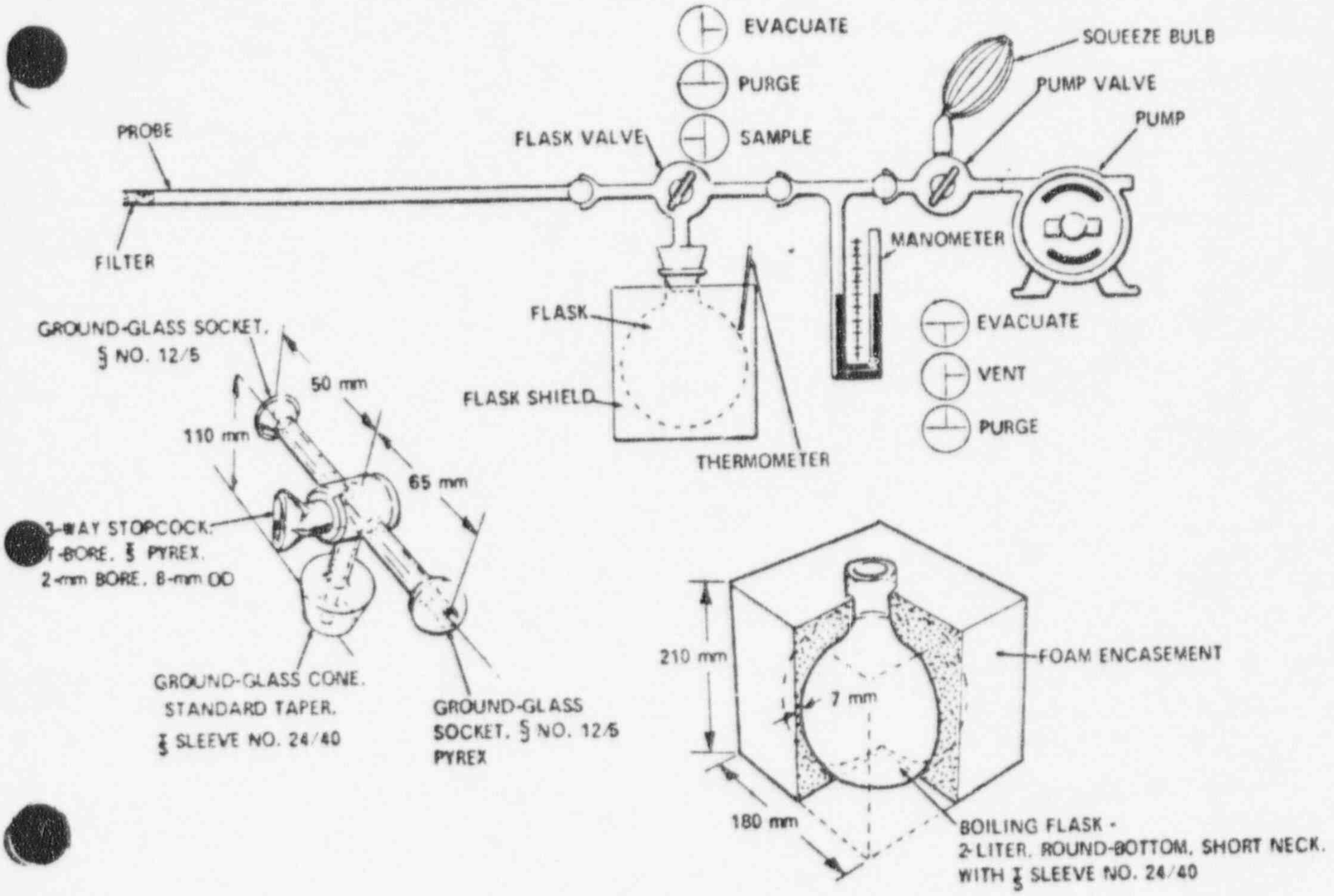

Figure 7-1. Sampling train, flask valve, and flask.

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 ±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), 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 (1N). 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 1,000-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 procelain 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. For both fixed and variable wavelength spectrophotometers, calibrate against standard certified wavelength of 410 nm, every 6 months. Alternatively, for variable wavelength spectrophotometers, scan the spectrum between 400 and 415 nm using a 200 μg NO_2 standard solution (see Section 5.2.2). 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 for both the standards and samples.

5.2.2 Determination of Spectrophotometer Calibration Factor K_c. Add 0.0, 1.0, 2.0, 3.0, and 4.0 ml of the KNO_3 working standard solution (1 ml = 100 μg NO_2) to a series of five porcelain evaporating dishes. To each, 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). 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:

$$K_c = 100\,\frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2}$$

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.

$$V_{sc} = \frac{T_{std}}{P_{std}}\,(V_f - V_a)\left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right]$$

$$= K_1(V_f - 25\text{ ml})\left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right]$$

Equation 7-2

where

$$K_1 = 0.3858\ \frac{°K}{\text{mm Hg}}\ \text{for metric units}$$

$$= 17.64\ \frac{°R}{\text{in. Hg}}\ \text{for English units}$$

6.3 Total μg NO_2 per sample.

$$m = 2\,K_c\,AF$$

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.

$$C = K_2\,\frac{m}{V_{sc}}$$

Equation 7-4

where

$$K_2 = 10^3\ \frac{\text{mg/m}^3}{\mu\text{g/ml}}\ \text{for metric units}$$

$$= 6.243 \times 10^{-5}\ \frac{\text{lb/scf}}{\mu\text{g/ml}}\ \text{for English units}$$

7. *Bibliography*

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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 (0.03×10^{-7} pounds/cubic foot) for sulfur trioxide and 1.2 mg/m³ (0.74×10^{-7} lb/ft³) 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 m³ (35.3 ft³) gas sample is about 12,500 mg/m³ (7.7×10^{-4} lb/ft³). 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, are required.

Filterable particulate matter may be determined along with SO_3 and SO_2 (subject to the approval of the Administrator); however, the procedure used for particulate matter must be consistent with the specifications and procedures given in Method 5.

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.

3.3.5 Sulfuric Acid Standard (0.0100 N). Purchase or standardize to ±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 m³/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 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.

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, m³/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, m³ (ft³)	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.

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 modification) 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 het 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

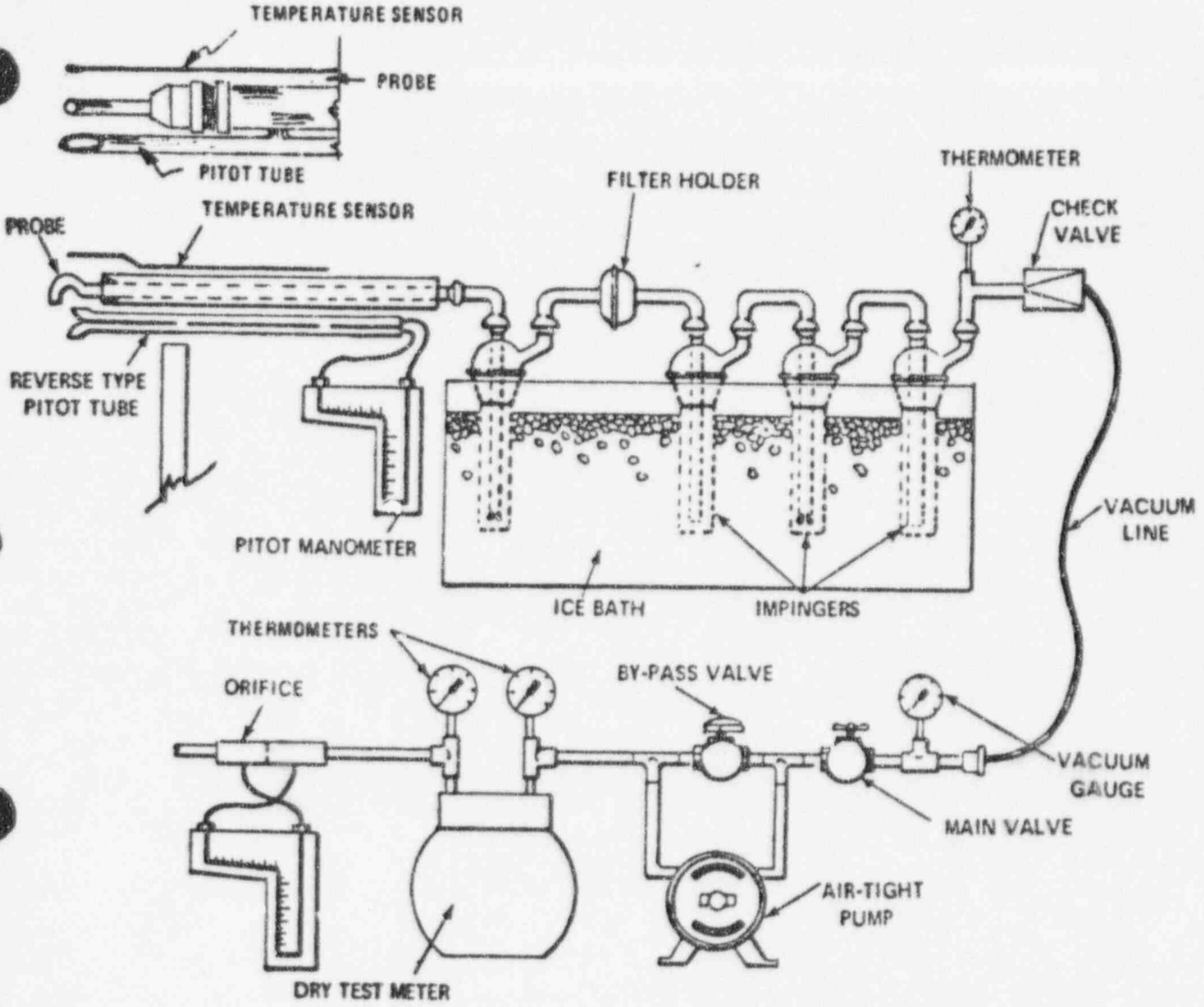

Figure 8-1. Sulfuric acid mist sampling train.

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).

2.7.2 Graduated Cylinders. 250 ml, 1 liter. (Volumetric flasks may also be used.)

3 Storage Bottles. Leak-free polyethylene bottles, 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 Burrette. 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. 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 ±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 m³/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 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.

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, m³/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, m³ (ft³)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
							INLET, °C (°F)	OUTLET, °C (°F)	
TOTAL									
AVERAGE							Avg	Avg	
							Avg		

Figure 8-2. Field data.

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 modification) 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 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, m² (ft²).
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 1,000 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.

$$I = \frac{100\, T_s [K_4 V_{lc} + (V_m/T_m) P_{bar} + \Delta H/13.6)]}{60\, \theta\, v_s P_s A_n}$$

Equation 8-4

where:
K_4 = 0.003464 mm Hg-m³/ml-°K for metric units.
= 0.002676 in. Hg-ft³/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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* * * * *

(Secs. 111, 114, 301(a), Clean Air Act, sec. 4(a) of Pub. L. 91-604, 84 Stat. 1683; sec. 4(a) of Pub. L. 91-604, 84 Stat. 1687; sec. 2 of Pub. L. 90-148, 81 Stat. 504 [42 U.S.C. 1857c-6, 1857c-9, 1857g(a)].)

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2.0 MONITORING METHODOLOGY
(continued)

STACK DATA FORMATS

VELOCITY TRAVERSE DATA

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, Cp= __________________

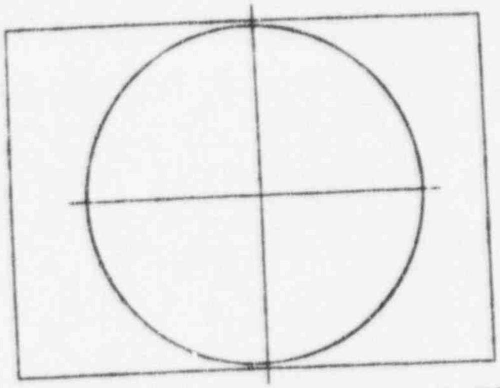
LAST DATE CALIBRATED ______________________

SCHEMATIC OF STACK CROSS SECTION

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

TRAVERSE POINT LOCATION

PLANT ____________________
DATE ____________________
SAMPLING LOCATION ____________________
INSIDE OF FAR WALL TO
OUTSIDE OF NIPPLE, (DISTANCE A) ____________________
INSIDE OF NEAR WALL TO
OUTSIDE OF NIPPLE, (DISTANCE B) ____________________
STACK I.D. (DISTANCE A - DISTANCE B) ____________________
NEAREST UPSTREAM DISTURBANCE ____________________
NEAREST DOWNSTREAM DISTURBANCE ____________________
CALCULATOR ____________________



SCHEMATIC OF SAMPLING LOCATIO

TRAVERSE POINT NUMBER	FRACTION OF STACK I.D.	STACK I.D.	PRODUCT OF COLUMNS 2 AND 3 (TO NEAREST 1/8 INCH)	DISTANCE B	TRAVERSE POINT LOCATIC FROM OUTSIDE OF NIPPL (SUM OF COLUMNS 4 & 5

SAMPLING TRAIN DATA AND CALCULATIONS

PROJECT ______________________________

TEST NO. __________ FILTER NO. __________

SAMPLING STATION __________ DATE __________

TIME	GAS METER READING CU. FT. V_M	 VAC. IN. HG P_M	 TEMP. °F T_M	SAMPLING RATE CFM	APPEARANCE OF PLUME	SAMPLING POINT

A. TOTAL WEIGHT COLLECTED, GRAMS __________

B. STACK GAS FLOW RATE, SCFM __________

C. CONDENSATE VOLUME, ML. __________

D. COND. VAPOR VOL., .00267 x C x (460 + T_M) / (29.9-P_M), CU. FT. __________

E. TOTAL SAMPLED VOL., V_M + D, CU. FT. __________

F. SAMPLED VOL., 520 x E x (29.9-P_M) / 29.9 x (460 + T_M), SCF __________

G. CONCENTRATION, 15.43 x A/F, GRAINS/SCF __________

H. MATERIAL FLOW RATE, 60 x G x B / 7000, LBS./HR. __________

1. SAMPLING NOZZLE DIAM. __________ 2. WEATHER CONDITIONS __________

3. REMARKS

NOMOGRAPH DATA

PLANT ______________________________

DATE ______________________________

SAMPLING LOCATION ____________________

CALIBRATED PRESSURE DIFFERENTIAL ACROSS ORIFICE, in.H_2O	$\Delta H_@$	
AVERAGE METER TEMPERATURE (AMBIENT + 20°F), °F	$T_{m_{avg.}}$	
PERCENT MOISTURE IN GAS STREAM BY VOLUME	B_{wo}	
BAROMETRIC PRESSURE AT METER, in.Hg	P_m	
STATIC PRESSURE IN STACK, in.Hg ($P_m \pm 0.073$ x STACK GAUGE PRESSURE in in.H_2O	P_s	
RATIO OF STATIC PRESSURE TO METER PRESSURE	P_s/P_m	
AVERAGE STACK TEMPERATURE, °F	$T_{s_{avg.}}$	
AVERAGE VELOCITY HEAD, in.H_2O	$\Delta P_{avg.}$	
MAXIMUM VELOCITY HEAD, in.H_2O	$\Delta P_{max.}$	
C FACTOR		
CALCULATED NOZZLE DIAMETER, in.		
ACTUAL NOZZLE DIAMETER, in.		
REFERENCE Δp, in.H_2O		

TO DETERMINE SAMPLING RATE WITHOUT NOMOGRAPH

$$S.R. = 0.33 \times V_s \times D^2 \times \frac{T_m}{T_s} \times \frac{P_s}{P_m} \times \left(1 - \frac{W.V.}{100}\right)$$

$S.R.$ = Isokinetic sampling rate ft^3/min.

V_s = Stack gas velocity, ft/sec.

D = Nozzle diameter in inches

T_s = Temperature in stack ^oR $(460 + {}^oF)$

T_m = Temperature at meter ^oR $(460 + {}^oF)$

P_s = Absolute pressure in stack $\left(P_b \pm \frac{P_{static}}{13.6}\right)$

P_b = Barometric pressure at sampling point (measured)

P_m = Pressure at meter $= P_b - P_{meter}$

$W.V.$ = Water vapor in stack gas % by vol.

$D = 1/8'' = 0.125$ in. $\quad D^2 = 0.0156 \quad .33D^2 = 0.00515$

$D = 1/4'' = 0.250$ in. $\quad D^2 = 0.0625\ in^2 \quad .33D^2 = 0.0206$

$D = 3/8'' = 0.375$ in. $\quad D^2 = 0.141\ in^2 \quad .33D^2 = 0.0465$

$D = 1/2'' = 0.500$ in. $\quad D^2 = 0.250\ in^2 \quad .33D^2 = 0.0825$

For 1/4" Nozzle

$$S.R. = V_s \times \left(0.0206 \times \frac{460 + T_m}{460 + T_s} \times \frac{P_b \pm \frac{P_{static}}{13.6}}{P_b \pm P_m} \times \frac{1 - W.V.}{100}\right)$$

$$0.0206 \times \frac{460 + T_m}{460 + T_s} \times \frac{P_b \pm \frac{P_{static}}{13.6}}{P_b \pm P_m} \times \frac{1 - W.V.}{100} = K$$

$$S.R. = K\, V_s$$

FIELD DATA

PLANT
DATE
SAMPLING LOCATION
SAMPLE TYPE
RUN NUMBER
OPERATOR
AMBIENT TEMPERATURE
BAROMETRIC PRESSURE
STATIC PRESSURE, (P_s)
FILTER NUMBER (s)

PROBE LENGTH AND TYPE
NOZZLE I.D.
ASSUMED MOISTURE, %
SAMPLE BOX NUMBER
METER BOX NUMBER
METER $\Delta H_@$
C FACTOR
PROBE HEATER SETTING
HEATER BOX SETTING
REFERENCE Δp

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY ______ MINUTES

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
					DESIRED	ACTUAL		INLET $(T_{m_{in}})$, °F	OUTLET $(T_{m_{out}})$, °F			

COMMENTS

PLANT ______________________
LOCATION ______________________
OPERATOR ______________________
DATE ______________________
RUN No. ______________________
SAMPLE BOX No. ______________________
METER BOX No. ______________________
METER Δ H@ ______________________

C FACTOR ______________________
AMBIENT TEMPERATURE ______________________
BAROMETERIC PRESSURE ______________________
ASSUMED MOISTURE, % ______________________
HEATER BOX SETTING ______________________
PROBE LENGTH, m. ______________________
NOZZLE DIAMETER, in. ______________________
PROBE HEATER SETTING ______________________

SCHEMATIC OF STACK CROSS SECTION

TRAVERSE POINT NUMBER	SAMPLING TIME (e) min.	STATIC PRESSURE (P_S)inHg	STACK TEMP. (T_S)*F	VELOCITY HEAD (ΔP_S)	ORIFICE PRESSURE DIFFERENTIAL (ΔH) In. H_2O	GAS SAMPLE VOLUME (Vm)ft.^{3}	GAS SAMPLE TEMPERATURE AT DRY GAS METER		SAMPLE BOX TEMPERATURE ^oF	TEMPERATURE OF GAS LEAVING LAST IMPINGER ^oF
							INLET $(Tm_{in})^oF$	OUTLET $(Tm_{out})^oF$		
TOTAL							avg.	avg.		
AVERAGE							avg.			

Figure 5-2. Particulate field data

NOMOGRAPH DATA

PLANT ______________________

DATE ______________________

SAMPLING LOCATION ______________________

CALIBRATED PRESSURE DIFFERENTIAL ACROSS ORIFICE, in. H_2O	$\Delta H_@$	
AVERAGE METER TEMPERATURE (AMBIENT + 20°F), °F	$T_{m_{avg.}}$	
PERCENT MOISTURE IN GAS STREAM BY VOLUME	B_{wo}	
BAROMETRIC PRESSURE AT METER, in. Hg	P_m	
STATIC PRESSURE IN STACK, in. Hg ($P_m \pm 0.073$ x STACK GAUGE PRESSURE in in. H_2O	P_s	
RATIO OF STATIC PRESSURE TO METER PRESSURE	P_s/P_m	
AVERAGE STACK TEMPERATURE, °F	$T_{s_{avg.}}$	
AVERAGE VELOCITY HEAD, in. H_2O	$\Delta P_{avg.}$	
MAXIMUM VELOCITY HEAD, in. H_2O	$\Delta P_{max.}$	
C FACTOR		
CALCULATED NOZZLE DIAMETER, in.		
ACTUAL NOZZLE DIAMETER, in.		
REFERENCE Δp, in. H_2O		

TO DETERMINE SAMPLING RATE WITHOUT NOMOGRAPH

$$S.R. = 0.33 \times V_s \times D^2 \times \frac{T_m}{T_s} \times \frac{P_s}{P_m} \times \left(1 - \frac{W.V.}{100}\right)$$

S.R. = Isokinetic sampling rate ft^3/min.

V_s = Stack gas velocity, ft/sec.

D = Nozzle diameter in inches

T_s = Temperature in stack ^oR $(460 + {^oF})$

T_m = Temperature at meter ^oR $(460 + {^oF})$

P_s = Absolute pressure in stack $\left(P_b \pm \frac{P_{static}}{13.6}\right)$

P_b = Barometric pressure at sampling point (measured)

P_m = Pressure at meter = $P_b - P_{meter}$

W.V. = Water vapor in stack gas % by vol.

D = 1/8" = 0.125 in. $D^2 = 0.0156$ $.33D^2 = 0.00515$

D = 1/4" = 0.250 in. $D^2 = 0.0625\ in^2$ $.33D^2 = 0.0206$

D = 3/8" = 0.375 in. $D^2 = 0.141\ in^2$ $.33D^2 = 0.0465$

D = 1/2" = 0.500 in. $D^2 = 0.250\ in^2$ $.33D^2 = 0.0825$

For 1/4" Nozzle

$$S.R. = V_s \times \left(0.0206 \times \frac{460 + T_m}{460 + T_s} \times \frac{P_b \pm \frac{P_{static}}{13.6}}{P_b \pm P_m} \times \frac{1 - W.V.}{100}\right)$$

$$0.0206 \times \frac{460 + T_m}{460 + T_s} \times \frac{P_b \pm \frac{P_{static}}{13.6}}{P_b \pm P_m} \times \frac{1 - W.V.}{100} = K$$

$$S.R. = K\ V_s$$

FIELD DATA

PLANT ____________________
DATE ____________________
SAMPLING LOCATION ____________________
SAMPLE TYPE ____________________
RUN NUMBER ____________________
OPERATOR ____________________
AMBIENT TEMPERATURE ____________________
BAROMETRIC PRESSURE ____________________
STATIC PRESSURE, (P_s) ____________________
FILTER NUMBER (s) ____________________

PROBE LENGTH AND TYPE ____________________
NOZZLE I.D. ____________________
ASSUMED MOISTURE, % ____________________
SAMPLE BOX NUMBER ____________________
METER BOX NUMBER ____________________
METER $\Delta H_@$ ____________________
C FACTOR ____________________
PROBE HEATER SETTING ____________________
HEATER BOX SETTING ____________________
REFERENCE Δ p ____________________

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY ________ MINUTES

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE °F
					DESIRED	ACTUAL		INLET ($T_{m_{in}}$), °F	OUTLET ($T_{m_{out}}$), °F			

COMMENTS

PLANT ______________________
LOCATION ______________________
OPERATOR ______________________
DATE ______________________
RUN No. ______________________
SAMPLE BOX No. ______________________
METER BOX No. ______________________
METER ΔH@ ______________________

C FACTOR ______________________
AMBIENT TEMPERATURE ______________________
BAROMETERIC PRESSURE ______________________
ASSUMED MOISTURE, % ______________________
HEATER BOX SETTING ______________________
PROBE LENGTH, m. ______________________
NOZZLE DIAMETER, in. ______________________
PROBE HEATER SETTING ______________________

SCHEMATIC OF STACK CROSS SECTION

TRAVERSE POINT NUMBER	SAMPLING TIME (e) min.	STATIC PRESSURE (P_S)inHg	STACK TEMP. (T_S)*F	VELOCITY HEAD (ΔP_S)	ORIFICE PRESSURE DIFFERENTIAL (ΔH) In. H_2O	GAS SAMPLE VOLUME (Vm)ft.^{3}	GAS SAMPLE TEMPERATURE AT DRY GAS METER		SAMPLE BOX TEMPERATURE ^oF	TEMPERATURE OF GAS LEAVING LAST IMPINGER ^oF
							INLET (Tm_{in})^oF	OUTLET (Tm_{out})^oF		
TOTAL							avg.	avg.		
AVERAGE							avg.			

Figure 5-2. Particulate field data

NOMOGRAPH DATA

PLANT ______________________

DATE ______________________

SAMPLING LOCATION ______________________

CALIBRATED PRESSURE DIFFERENTIAL ACROSS ORIFICE, in.H_2O	$\Delta H_@$	
AVERAGE METER TEMPERATURE (AMBIENT + 20°F), °F	$T_{m_{avg.}}$	
PERCENT MOISTURE IN GAS STREAM BY VOLUME	B_{wo}	
BAROMETRIC PRESSURE AT METER, in.Hg	P_m	
STATIC PRESSURE IN STACK, in.Hg ($P_m \pm 0.073$ x STACK GAUGE PRESSURE in in.H_2O	P_s	
RATIO OF STATIC PRESSURE TO METER PRESSURE	P_s/P_m	
AVERAGE STACK TEMPERATURE, °F	$T_{s_{avg.}}$	
AVERAGE VELOCITY HEAD, in.H_2O	$\Delta P_{avg.}$	
MAXIMUM VELOCITY HEAD, in.H_2O	$\Delta P_{max.}$	
C FACTOR		
CALCULATED NOZZLE DIAMETER, in.		
ACTUAL NOZZLE DIAMETER, in.		
REFERENCE Δp, in.H_2O		

TO DETERMINE SAMPLING RATE WITHOUT NOMOGRAPH

$$S.R. = 0.33 \times V_s \times D^2 \times \frac{T_m}{T_s} \times \frac{P_s}{P_m} \times \left(1 - \frac{W.V.}{100}\right)$$

$S.R.$ = Isokinetic sampling rate $ft^3/min.$

V_s = Stack gas velocity, ft/sec.

D = Nozzle diameter in inches

T_s = Temperature in stack ^{o}R $(460 + {}^{o}F)$

T_m = Temperature at meter ^{o}R $(460 + {}^{o}F)$

P_s = Absolute pressure in stack $\left(P_b \pm \frac{P_{static}}{13.6}\right)$

P_b = Barometric pressure at sampling point (measured)

P_m = Pressure at meter $= P_b - P_{meter}$

$W.V.$ = Water vapor in stack gas % by vol.

$D = 1/8" = 0.125$ in. $\quad D^2 = 0.0156 \quad .33D^2 = 0.00515$

$D = 1/4" = 0.250$ in. $\quad D^2 = 0.0625\ in^2 \quad .33D^2 = 0.0206$

$D = 3/8" = 0.375$ in. $\quad D^2 = 0.141\ in^2 \quad .33D^2 = 0.0465$

$D = 1/2" = 0.500$ in. $\quad D^2 = 0.250\ in^2 \quad .33D^2 = 0.0825$

For 1/4" Nozzle

$$S.R. = V_s \times \left(0.0206 \times \frac{460 + T_m}{460 + T_s} \times \frac{P_b \pm \frac{P_{static}}{13.6}}{P_b \pm P_m} \times \frac{1 - W.V.}{100}\right)$$

$$0.0206 \times \frac{460 + T_m}{460 + T_s} \times \frac{P_b \pm \frac{P_{static}}{13.6}}{P_b \pm P_m} \times \frac{1 - W.V.}{100} = K$$

$$S.R. = K\,V_s$$

PLANT ____________________

LOCATION ____________________

OPERATOR ____________________

DATE ____________________

RUN No. ____________________

SAMPLE BOX No. ____________________

METER BOX No. ____________________

METER ΔH@ ____________________

C FACTOR ____________________

AMBIENT TEMPERATURE ____________________

BAROMETERIC PRESSURE ____________________

ASSUMED MOISTURE, % ____________________

HEATER BOX SETTING ____________________

PROBE LENGTH, m. ____________________

NOZZLE DIAMETER, in. ____________________

PROBE HEATER SETTING ____________________

SCHEMATIC OF STACK CROSS SECTION

TRAVERSE POINT NUMBER	SAMPLING TIME (e) min.	STATIC PRESSURE (P_S) inHg	STACK TEMP. (T_S) *F	VELOCITY HEAD (ΔP_S)	ORIFICE PRESSURE DIFFERENTIAL (ΔH) In. H_2O	GAS SAMPLE VOLUME (Vm) $ft.^3$	GAS SAMPLE TEMPERATURE AT DRY GAS METER		SAMPLE BOX TEMPERATURE °F	TEMPERATURE OF GAS LEAVING LAST IMPINGER °F
							INLET (Tm_{in}) °F	OUTLET (Tm_{out}) °F		
TOTAL							avg.	avg.		
AVERAGE							avg.			

Figure 5-2. Particulate field data

TRAVE POINT NUMBER	AMPLING TIME, min	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (P_s), in. H_2O	ORIFICE SURE DIFFERENTIAL (H), in. H_2O)		STACK TEMPERATURE (T_s), °F	RY GAS METER TEMPERATURE		PUMP VACUUM in. Hg	SAMPLE BO TEMPERATURE, °F	IMPINGER TEMPERATURE °F
					DESIRED	ACTUAL		INLET ($T_{m_{in}}$), °F	OUTLET ($T_{m_{out}}$), °F			

DRY MOLECULAR WEIGHT DETERMINATION

PLANT__________________________________
DATE__________________________________
SAMPLING TIME (24-hr CLOCK)__________________
SAMPLING LOCATION________________________
SAMPLE TYPE (BAG, INTEGRATED, CONTINUOUS_________
ANALYTICAL METHOD________________________
AMBIENT TEMPERATURE______________________
OPERATOR______________________________

COMMENTS:

RUN / GAS	1		2		3		AVERAGE NET VOLUME	MULTIPLIER	MOLECULAR WEIGHT (STACK GAS (DRY BAS M_d, lb/lb-mole
	ACTUAL READING	NET	ACTUAL READING	NET	ACTUAL READING	NET			
CO_2								44/100	
O_2 (NET IS ACTUAL O_2 READING MINUS ACTUAL CO_2 READING)								32/100	
CO (NET IS ACTUAL CO READING MINUS ACTUAL O_2 READING)								28/100	
N_2(NET IS 100 MINUS ACTUAL CO READING)								28/100	
								TOTAL	

SAMPLIN MARY SHEET

PLANT ______________________________ LOCATION ______________________________

SAMPLED SOURCE ______________________________

RUN	DATE	N_p	P_m	P_b	V_m	T_m	$V_{M_{std}}$	V_W	$V_{W_{gas}}$	$\%_M$	M_d

RUN	MW_d	MW	P_{st}	P_s	C_p	$\sqrt{\Delta P_s \times (T_s + 460)}^c$	V_s	T_s	T_t	D_n	I%

$$V_{m_{std}} = \frac{17.7 \times V_m \left(P_b = \frac{P_m}{13.6}\right)}{(T_m + 460)} \qquad \% M = \frac{100 \times V_{w_{gas}}}{V_{m_{std}} + V_{w_{gas}}}$$

$$V_{w_{gas}} = 0.0474 \times V_w \qquad M_d = \frac{100 - \%M}{100}$$

$$MW_d = \left(\%CO_2 \times \frac{44}{100}\right) + \left(\%O_2 \times \frac{32}{100}\right) + (\%CO + \%N_2) \times \frac{28}{100}$$

$$MW = MW_d \times M_d + 18\,(1 - M_d)$$

$$P_s = P_b \pm P_{st}$$

$$V_s = 5128.8 \times Cp \times \sqrt{\Delta P_s \times (T_s + 460)} \left[\frac{1}{P_s \times MW}\right]^{1/2}$$

$$\%I = \frac{1{,}032 \times (T_s + 460) \times V_{m_{std}}}{V_s \times T_t \times P_s \times M_d \times (D_n)^2}$$

N_p	Total No. of Sampling Points
P_m	Average Orifice Pressure Drop, in. H_2O
P_b	Barometric Pressure, in. Hg Absolute
V_m	Volume of Dry Gas at Meter Conditions, DCF
T_m	Average Meter Temperature, °F
$V_{m_{std}}$	Volume of Dry Gas at STP, DSCF^a
V_w	Total H_2O Collected in Impingers and Silica Gel, ml
$V_{w_{gas}}$	Volume of Water Vapor Collected at STP, SCF^o
%M	% Moisture by Volume
M_d	Mole Fraction of Dry Gas
$\%CO_2$	Volume % Dry
$\%O_2$	Volume % Dry

%CO	Volume % Dry
$\%N_2$	Volume % Dry
MW_d	Molecular Weight of Stack Gas, Dry Basis
MW	Molecular Weight of Stack Gas, Wet Basis
P_{st}	Static Pressure of Stack Gas, in. Hg
P_s	Stack Gas Pressure, in. Hg Absolute
C_p	Pitot Tube Coefficient
V_s	Stack Gas Velocity at Stack Conditions, fpm
T_s	Average Stack Temperature °F
T_t	Net Time of Test, Min.
Dn	Sampling Nozzle Diameter, in.
%I	Percent Isokinetic

[a] Dry standard cubic feet at 70°F, 29.92 in. Hg.

[b] Standard conditions at 70°F, 29.92 in. Hg.

[c] $\sqrt{\Delta P_s \times (T_s + 460)}$ is determined by averaging the square root of the product of the velocity head (Ps) and the absolute stack temperature from each sampling point.

PLANT________________________

DATE_________________________

RUN NO.______________________

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			

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

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

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

Figure 5-3. Analytical data.

SIGNIFICANCE OF ERRORS IN STACK SAMPLING MEASUREMENTS

Measurement	Maximum Error, %
Stack Temperature, t_s	1.4
Meter Temperature, t_m	1.0
Stack Gage Pressure, P_{gs}	0.42
Meter Gage Pressure, P_{gm}	0.42
Atmospheric Pressure, P_{atm}	0.21
Dry Molecular Weight, M_d	0.42
Moisture Content, $d\ B_{ws}$ (absolute)	1.1
$d\ B_{ws}/1 - B_{ws}$	1.0
Pressure Head, Δp	10.0
Orifice Pressure Differential, Δm	5.0
Pitot Tube Coefficient, C_p	2.4
Orifice Meter Coefficient, K_m	1.5
Diameter of Probe Nozzle, D_n	0.80

2.0 MONITORING METHODOLOGY

(continued)

STACK SAMPLING FORMULAS

FORMULAS USED

FOR STACK SAMPLING

EQUATION 1-1

$$\text{equivalent diameter} = 2\,\frac{((\text{length})\ (\text{width}))}{(\text{length} + \text{width})}$$

EQUATION 2-1 - Pitot Tube Coefficient

$$C_{P_{test}} = C_{P_{std}} \sqrt{\frac{\Delta P_{std}}{\Delta P_{test}}}$$

where:

$C_{P_{test}}$ = Pitot tube coefficient of Type S Pitot Tube

$C_{P_{std}}$ = Pitot tube coefficient of standard type pitot tube (if unknown, use 0.99).

ΔP_{std} = Velocity head measured by standard type pitot tube.

ΔP_{test} = Velocity head measured by Type S pitot tube.

EQUATION 2-2 - Gas Stack Velocity

$$(V_s)\text{avg.} = K_P C_P\ (\sqrt{\Delta P})\ \text{avg.} \sqrt{\frac{(T_s)\ \text{avg.}}{P_s\ M_s}}$$

where:

(V_s)avg. = Stack gas velocity, feet per second (F.P.S.).

K_P = 85.48 $\frac{\text{ft.}}{\text{sec.}} \left(\frac{\text{lb.}}{\text{lb. mole-}^{\circ}\text{R}} \right)^{1/2}$ when these units are used.

C_P = Pitot tube coefficient dimensionless.

(T_s)avg. = Average absolute stack gas temperature, °R.

$(\sqrt{\Delta P})$avg.= Average velocity head of stack gas, inches H_2O (See Fig. 2-2).

P_s = Absolute stack gas pressure, inches Hg.

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

$M_d\ (1-B_{wo}) + 18B_{wo}$

M_d = Dry molecular weight of stack gas (from Method 3).

B_{wo} = Proportion by volume of water vapor in the gas stream (from Method 4).

EQUATION 2-3 - Stack Gas Volumetric Flow Rate

$$Q_s = 3600\ (1-B_{wo})\ V_s A\ \left(\frac{T_{std}}{(T_s)avg.}\right)\left(\frac{P_s}{P_{std}}\right)$$

where:

Q_s = Volumetric flow rate, dry basis, standard conditions, $ft^3/hr.$

A = Cross-sectional area of stack, $ft.^2$

T_{std} = Absolute temperature at standard conditions, 530°R.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

EQUATION 3-1 - Percent Excess Air

%EA=

$$\frac{(\%\ O_2) - 0.5\ (\%\ CO)}{0.264\ (\%\ N_2) - (\%\ O_2) + 0.5\ (\%\ CO)} \times 100$$

where:

% EA = Percent excess air.

$\%\ O_2$ = Percent oxygen by volume, dry basis.

$\%\ N_2$ = Percent nitrogen by volume, dry basis.

% CO = Percent carbon monoxide by volume, dry basis.

0.264 = Ratio of oxygen to nitrogen in air by volume.

EQUATION 3-2 Dry Molecular Weight

$$Md = 0.44\ (\%\ CO_2) + 0.32\ (\%\ O_2) + 0.28\ (\%\ N_2 + \%\ CO)$$

where:

Md = Dry molecular weight, lb./lb-mole.

$\%CO_2$ = Percent carbon dioxide by volume, dry basis.

$\%O_2$ = Percent oxygen by volume, dry basis.

$\%N_2$ = Percent nitrogen by volume, dry basis.

0.44 = Molecular weight of carbon dioxide divided by 100.

0.32 = Molecular weight of oxygen divided by 100.

0.28 = Molecular weight of nitrogen and CO divided by 100.

EQUATION 4-1 - Volume of Water Vapor Collected

$$V_{wc} = \frac{(V_f - V_i)\ pH_2O\ RT_{std}}{P_{std}M_{H_2O}} = 0.0474\ \frac{ft.^3}{ml.}\ (V_f - V_i)$$

where:

Vwc = Volume of water vapor collected (standard conditions), cu. ft.

Vf = Final volume of impinger contents, ml.

Vi = Initial volume of impinger contents, ml.

R = Ideal gas constant, 21.83 inches Hg - cu. ft./lb. mole-$^{\circ}$R.

pH_2O = Density of water, 1 g./ml.

Tstd = Absolute temperature at standard conditions, 530° R.

Pstd = Absolute pressure at standard conditions, 29.92 inches Hg.

MH_2O = Molecular weight of water, 18 lb./lb.-mole.

EQUATION 4-2 -Dry Gas Volume

$$V_{mc} = V_m\left(\frac{P_m}{P_{std}}\right)\left(\frac{T_{std}}{T_m}\right) = 17.71\ \frac{^{\circ}R}{in.\ Hg}\left(\frac{V_m\ P_m}{T_m}\right)$$

where:

V_{mc} = Dry gas volume through meter at standard conditions, cu. ft.

V_m = Dry gas volume measured by meter, cu. ft.

P_m = Barometric pressure at the dry gas meter, inches Hg.

P_{std} = Pressure at standard conditions, 29.92 inches Hg.

T_{std} = Absolute temperature at standard conditions, 530° R.

T_m = Absolute temperature at meter ($^{\circ}$F + 460), $^{\circ}$R.

Moisture content.

EQUATION 4-3 - Proportion By Volume of Water Vapor.

$$B_{wo} = \frac{V_{wc}}{V_{wc} + V_{mc}} + B_{wm} = \frac{V_{wc}}{V_{wc} + V_{mc}} + (0.025)$$

where:

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

V_{wc} = Volume of water vapor collected (standard conditions), cu. ft.

V_{mc} = Dry gas volume through meter (standard conditions), cu. ft.

B_{wm} = Approximate volumetric proportion of water vapor in the gas stream leaving the impingers, 0.025.

Moisture Determination (Hygrometer)

To determine the water vapor content of the gas with the Hygrometer - wet and dry bulb apparatus, a probe is inserted into the duct and a source of aspiration used to pull a gas sample into the probe past the dry and then the wet thermometers. A mercury manometer is used to measure the vacuum in the system, which in this application will be the static pressure.

To maintain optimum conditions in the apparatus the length of hose between the probe and the apparatus should be varied so that the dry bulb reading is 180^{o}F or more.

The flow rate through the apparatus should be maintained at 16 FPS, for a $\frac{1}{2}$" ID tube this would require 1.33 CFM.

Terms:

P_{va} - Water vapor pressure in Hygrometer (In. Hg.)

P_{vd} - Water vapor pressure in duct (In. Hg.)

P_{v} (wb) - Saturated water vapor pressure at t_w (In. Hg.)

P_d - Pressure in duct (static) at sampling point (In. Hg.)

P_b - Barometric pressure (In. Hg.)

P_a - Static pressure in Hygrometer (In. Hg.)

t_d - Temperature dry bulb (°F)

t_w - Temperature wet bulb (°F)

P_{va} may be obtained from a standard psychrometric chart or calculated from the following formula.

$$P_{va} = P_{v\,(wb)} - \left[\frac{(P_b - P_a - P_{v\,(wb)})\ (t_d - t_w)}{2800 - 1.3t_w} \right]$$

$$P_{vd} = \frac{P_b - P_d}{P_b - P_a} \ X\ P_{va}$$

% H_2O (volume percent moisture in gas)

$$= \frac{P_{vd}}{P_b - P_d} \ X\ 100$$

TABLE OF VAPOR PRESSURES OF WATER

In Inches of Mercury

Temp. eg. F.	0	1	2	3	4	5	6	7	8	9
-20	.0126	.0119	.0112	.0106	.0100	.0095	.0089	.0084	.0080	.0075
-10	.0222	.0209	.0199	.0187	.0176	.0168	.0158	.0150	.0142	.0134
-	.0376	.0359	.0339	.0324	.0306	.0289	.0275	.0259	.0247	.0233
0	.0376	.0398	.0417	.0441	.0463	.0489	.0517	.0541	.0571	.0598
10	.0631	.0660	.0696	.0728	.0768	.0810	.0846	.0892	.0932	.0982
20	.1025	.1080	.1127	.1186	.1248	.1302	.1370	.1429	.1502	.1567
30	.1647	.1716	.1803	.1878	.1955	.2035	.2118	.2203	.2292	.2383
40	.2478	.2576	.2677	.2732	.2891	.3004	.3120	.3240	.3364	.3493
50	.3626	.3764	.3906	.4052	.4203	.4359	.4520	.4686	.4858	.5035
60	.5218	.5407	.5601	.5802	.6009	.6222	.6442	.6669	.6903	.7144
70	.7392	.7648	.7912	.8183	.8462	.8750	.9046	.9352	.9666	.9989
80	1.032	1.066	1.102	1.138	1.175	1.213	1.253	1.293	1.335	1.378
90	1.422	1.467	1.513	1.561	1.610	1.660	1.712	1.765	1.819	1.875
100	1.932	1.992	2.052	2.114	2.178	2.243	2.310	2.379	2.449	2.521
110	2.596	2.672	2.749	2.829	2.911	2.995	3.081	3.169	3.259	3.351
120	3.446	3.543	3.642	3.744	3.848	3.954	4.063	4.174	4.289	4.406
130	4.525	4.647	4.772	4.900	5.031	5.165	5.302	5.442	5.585	5.732
140	5.881	6.034	6.190	6.350	6.513	6.680	6.850	7.024	7.202	7.384
150	7.569	7.759	7.952	8.150	8.351	8.557	8.767	8.981	9.200	9.424
160	9.652	9.885	10.12	10.36	10.61	10.86	11.12	11.38	11.65	11.92
170	12.20	12.48	12.77	13.07	13.37	13.67	13.98	14.30	14.62	14.96
180	15.29	15.63	15.98	16.34	16.70	17.07	17.44	17.82	18.21	18.61
190	19.01	19.42	19.84	20.27	20.70	21.14	21.59	22.05	22.52	22.99
200	23.47	23.96	24.46	24.97	25.48	26.00	26.53	27.07	27.62	28.18
210	28.75	29.33	29.92	30.52	31.13	31.75	32.38	33.02	33.67	34.33
220	35.00	35.68	36.37	37.07	37.78	38.50	39.24	39.99	40.75	41.52
230	42.31	43.11	43.92	44.74	45.57	46.41	47.27	48.14	49.03	49.93
240	50.84	51.76	52.70	53.65	54.62	55.60	56.60	57.61	58.63	59.67
250	60.72	61.79	62.88	63.98	65.10	66.23	67.38	68.54	69.72	70.92
260	72.13	74.36	74.61	75.88	77.16	78.46	79.78	81.11	82.46	83.83
270	85.22	86.63	88.06	89.51	90.97	92.45	93.96	95.49	97.03	98.61
280	100.2	101.8	103.4	105.0	106.7	108.4	110.1	111.8	113.6	115.4
290	117.2	119.0	120.8	122.7	124.6	126.5	128.4	130.4	132.4	134.4
300	136.4	138.5	140.6	142.7	144.8	147.0	149.2	151.4	153.6	155.9
310	158.2	160.5	162.8	165.2	167.6	170.0	172.5	175.0	177.5	180.0
320	182.6	185.2	187.8	190.4	193.1	195.8	198.5	201.3	204.1	206.9
330	209.8	212.7	215.6	218.6	221.6	224.6	227.7	230.8	233.9	237.1
340	240.3	243.5	246.8	250.1	253.4	256.7	260.1	263.6	267.1	270.6
350	274.1	277.7	281.3	284.9	288.6	292.3	296.1	299.9	303.8	307.7
360	311.6	315.5	319.5	323.5	327.6	331.7	335.9	340.1	344.4	348.7
370	353.0	357.4	361.8	366.2	370.7	375.2	379.8	384.4	389.1	393.8
380	398.6	403.4	408.2	413.1	418.1	423.1	428.1	433.1	438.2	443.4
390	448.6	453.9	459.2	464.6	470.0	475.5	481.0	486.6	492.2	497.9
400	503.6	509.3	515.1	521.0	526.9	532.9	538.9	545.0	551.1	557.3

EQUATION 5-1 - Volume of Sample Gas

$$V_{m_{std}} = V_m \left(\frac{T_{std}}{T_m}\right) \left(\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right) =$$

$$\left(17.71 \frac{^{\circ}R}{\text{in. Hg}}\right) V_m \left(\frac{P_{bar} + \frac{\Delta H}{13.6}}{T_m}\right)$$

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_{std} = Absolute temperature at standard conditions, 530°R.

T_m = Average dry gas meter temperature, °R.

P_{bar} = Barometric pressure at the orifice meter, inches Hg.

ΔH = Average pressure drop across the orifice meter, inches H_2O.

13.6 = Specific gravity of mercury.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

EQUATION - 5-2 - Volume of Water Vapor

$$V_{w_{std}} = V_{1_c} \left(\frac{\rho H_2O}{M_{H2}}\right) \left(\frac{RT_{std}}{P_{std}}\right) = \left(0.0474 \frac{\text{cu. ft.}}{\text{ml.}}\right) V_{1_c}$$

where:

$V_{w_{std}}$ = Volume of water vapor in the gas sample (standard conditions) cu. ft.

V_{1_c} = Total volume of liquid collected in impingers and silica gel (fig. 5-2 ml.

ρH_2O = Density of water, 1 g./ml.

MH_2O = Molecular weight of water, 18 lb/lb mole.

R = Ideal gas constant, 21.83 in Hg-cu. ft./lb. mole-°R.

T_{std} = Absolute temperature at standard conditions, 530°R.

P_{std} = Absolute pressure at standard conditions, 29.92 in Hg.

EQUATION 5-3 - Moisture Content

$$B_{wo} = \frac{V_{w_{std}}}{V_{m_{std}} + V_{w_{std}}}$$

where:

B_{wo} = Proportion by volume of water in the gas stream, dimensionless.

$V_{w_{std}}$ = Volume of water in the gas sample (standard conditions), cu. ft.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions) cu. ft.

EQUATION 5-4 - Concentration in gr./s.c.f.

$$C'_s = \left(0.0154 \frac{gr.}{mg.} \right) \left(\frac{M_n}{V_{m_{std}}} \right)$$

where:

C'_s = Concentration of particulate matter in stack gas, gr./s.c.f., dry basis.

M_n = Total amount of particulate matter collected, mg.

$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

EQUATION 5-5 - Concentration in lb./cu. ft.

$$C = \frac{\left(\frac{1 \quad \text{lb.}}{453{,}600 \text{ mg.}}\right) M_n}{V_{m_{std}}} = 2.205 \times 10^{-6} \frac{M_n}{V_{m_{std}}}$$

where:

C_s = Concentration of particulate matter in stack gas, lb./s.c.f., dry basis

453,600 = Mg/lb.

M_n = Total amount of particulate matter collected, mg.

$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

EQUATION 5-6 - Isokinetic Variation

$$I = \frac{T_s \left[\frac{V_{1_c} (\rho H_2O) R}{M_{H_2O}} + \frac{V_m}{T_m} \left(P_{bar} + \frac{H}{13.6}\right)\right] \times 100}{\theta V_s P_s A_n}$$

$$= \frac{T_s \left(1.667 \frac{\text{min.}}{\text{sec.}}\right) \left[\left(0.00267 \frac{\text{in.Hg. cu. ft.}}{\text{ml.-}^{\circ}\text{R.}}\right) V_s + \frac{V_m}{T_m} \left(P_{bar} + \frac{\Delta H}{13.6}\right)\right]}{0 V_s P_s A_n}$$

where:

I = Percent of isokinetic sampling.

V_{1_c} = Total volume of liquid collected in impingers and silica gel, ml.

ρH_2O = Density of water, 1 g/ml.

R = Ideal gas constant, 21.83 inches Hg-cu. ft./lb. mole-°R.

M_{H_2O} = Molecular weight of water, 18 lb./lb.-mole.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_m = Absolute average dry gas meter temperature (see page 40), °R.

P_{bar} = Barometric pressure at sampling site, inches Hg.

ΔH = Average pressure drop across the orifice (see page 40), inches H_2O.

T_s = Absolute average stack gas temperature (see page 40), °R.

θ = Total sampling time, min.

V_s = Stack gas velocity calculated by Method 2, Equation 2-2, ft./sec.

P_s = Absolute stack gas pressure, inches Hg.

A_n = Cross-sectional area of nozzle, sq. ft.

EQUATION 6-1 Dry Gas Volume

$$V_{m_{std}} = V_m \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}}{P_{std}}\right) = 17.71 \frac{^{\circ}R}{\text{in.Hg.}} \left(\frac{V_m P_{bar}}{T_m}\right)$$

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_{std} = Absolute temperature at standard conditions , cu. ft.

T_m = Average dry gas meter temperature, $^{\circ}$R.

P_{bar} = Barometric pressure at the orifice meter, in. Hg.

P_{std} = Absolute pressure at standard conditions, 29.92 in. Hg.

EQUATION 6-2 - Sulfur Dioxide Concentration

$$C_{so_2} = \left(7.05 \times 10^{-5} \; \frac{\text{lb.-l.}}{\text{g.-ml.}}\right) \frac{(V_t - V_{tb})\,N\,\left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}}$$

where:

Cso_2 = Concentration of sulfur dioxide at standard conditions, dry basis, lb./cu. ft.

$7.05X10^{-5}$ = Conversion factor including the number of grams per gram equivalent of sulfur dioxide (32 g./g.-eq.), 453.6 g./lb., and 1,000 ml./l, lb.-l./g.-ml.

V_t = Volume of barium perchlorate titrant used for the sample, ml.

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

N = Normality of barium perchlorate titrant, g.-eq./l.

V_{soln} = Total solution volume of sulfur dioxide, ml.

V_a = Volume of sample aliquot titrated, ml.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), see Equation 6-1, cu. ft.

EQUATION 7-1 - Sample Volume

$$V_{sc} = \frac{T_{std}\,(V_{f}-V_{a})}{P_{std}} \left(\frac{P_f}{T_f}\;\frac{P_i}{T_i}\right) = \left(17.71\ \frac{^{\circ}R}{\text{in.Hg.}}\right)(V_t - 25\text{ml.})\left(\frac{P_f}{T_f}\;\frac{P_i}{T_i}\right)$$

where:

V_{sc} = Sample volume at standard conditions (dry basis), ml.

T_{std} = Absolute temperature at standard condtions, 530°R.

P_{std} = Pressure at standard condtions, 29.92 in. Hg.

V_f = Volume of flask and valve, ml.

V_a = Volume of absorbing solution, 25 ml.

P_f = Final absolute pressure of flask, in. Hg.

P_i = Initial absolute pressure of flask, in. Hg.

T_f = Final absolute temperature of flask, °R.

T_i = Initial absolute temperature of flask, °R.

EQUATION 7-2 - Sample Concentration

$$C = \left(\frac{m}{V_{sc}}\right)\left(\frac{1\text{ lb./cu. ft.}}{1.6\text{X}10^{4}\text{ ug./ml.}}\right) = \left(6.2\text{X}10^{-5}\ \frac{\text{lb./s.c.f.}}{\mu\text{g./ml.}}\right)\left(\frac{m}{V_{sc}}\right)$$

where:

C = Concentration of NO_x as NO_2 (dry basis), lb./s.c.f.

m = Mass of NO_2 in gas sample, μg.

V_{sc} = Sample volume at standard conditions (dry basis), ml.

EQUATION 8-1 - Dry Gas Volume

$$V_{m_{std}} = V_m \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar} + \Delta H/13.6}{P_{std}}\right) = \left(17.71\ \frac{^{\circ}R}{\text{in.Hg.}}\right)(V_m)\left(\frac{P_{bar} + \Delta H/13.6}{T_m}\right)$$

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_{std} = Absolute temperature at standard conditions, 530°R.

T_m = Average dry gas meter temperature, °R.

P_{bar} = Barometric pressure at the orifice meter, in. Hg.

ΔH = Pressure drop across the orifice meter, in. H_2O.

13.6 = Specific gravity of mercury.

P_{std} = Absolute pressure at standard conditions, 29.92 in. Hg.

EQUATION 8-2 - Sulfuric Acid Concentration

$$C_{H_2SO_4} = \left(1.08\ X10^{-4}\ \frac{lb.-l.}{g.-ml.}\right) \frac{(V_t - V_{tb})(\underline{N})\left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}}$$

where:

$C_{H_2SO_4}$ = Concentration of sulfuric acid at standard conditions dry basis, lb./ cu. ft.

$1.08.10^{-4}$ = Conversion factor including the number of grams per gram equivalent of sulfuric acid (49 g./g.-eq.), 453.6 g./lb., and 1,000 ml./l., lb.-l.g.-ml.

V_t = Volume of barium perchlorate titrant used for the sample, ml.

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

$\underline{N}$ = Normality of barium perchlorate titrant, g.-eq./l.

V_{soln} = Total solution volume of sulfuric acid (first impinger and filter), ml.

V_a = Volume of sample aliquot titrated, ml.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.. see Equation 8-1.

EQUATION 8-3 - Sulfur Dioxide Concentration

$$C_{so_2} = \left(7.05\ X\ 10^{-5}\ \frac{lb.-l.}{b.-ml.}\right) \frac{(V_t - V_{tb})(\underline{N})\left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}}$$

where:

C_{so_2} = Concentration of sulfur dioxide at standard conditions, dry basis, lb./cu. ft.

$7.05X10^{-5}$ = Conversion factor including the number of grams per gram equivalent of sulfur dioxide (ew g./g.-eq.) 453.6 g./lb., and 1,000 ml./l., lb.-l/g.-ml.

V_t = Volume of barium perchlorate titrant used for the sample, ml.

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

$\underline{N}$ = Normality of barium perchlorate titrant, g.-eq./l.

V_{soln} = Total solution volume of sulfur dioxide (second and third impingers), ml.

V_a = Volume of sample aliquot titrated, ml.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft., see Equation 8-1.

TO DETERMINE SAMPLING RATE WITHOUT NOMOGRAPH

$$S.R. = 0.33 \times V_s \times D^2 \times \frac{T_m}{T_s} \times \frac{P_s}{P_m} \times \left(1- \frac{W.V.}{100}\right)$$

S.R. = Isokinetic sampling rate ft^3/min.

V_s = Stack gas velocity, ft/sec.

D = Nozzle diameter in inches

T_s = Temperature in stack °R (460 + °F)

T_m = Temperature at meter °R (460 + °F)

P_s = Absolute pressure in stack $\left(P_b \pm \frac{P_{static}}{13.6}\right)$

P_b = Barometric pressure at sampling point (measured)

P_m = Pressure at meter = $P_b - P_{meter}$

W.V. = Water vapor in stack gas % by vol.

D = 1/8" = 0.125 in. D^2 = 0.0156 $.33D^2$ = 0.00515

D = 1/4" = 0.250 in. D^2 = 0.0625 in^2 $.33D^2$ = 0.0206

D = 3/8" = 0.375 in. D^2 = 0.141 in^2 $.33D^2$ = 0.0465

D = 1/2" = 0.500 in. D^2 = 0.250 in^2 $.33D^2$ = 0.0825

For 1/4" Nozzle

$$S.R. = V_s \times \left(0.0206 \times \frac{460 + T_m}{460 + T_s} \times \frac{P_b \pm \frac{P_{static}}{13.6}}{P_b \pm P_m} \times \frac{1- W.V.}{100}\right)$$

$$0.0206 \times \frac{460 + T_m}{460 + T_s} \times \frac{P_b \pm \frac{P_{static}}{13.6}}{P_b \pm P_m} \times \frac{1- W.V.}{100} = K$$

$$S.R. = K\ V_s$$

8-2-74 - PMG

2.0 MONITORING METHODOLOGY

(continued)

DESCRIPTION, OPERATING INSTRUCTIONS, AND PROCEDURES

Figures

Appendix

Supplemental

NOTE: Supplemental instruction supplied with manual apply only to units supplied with either/or of the above options.

INSTRUCTIONS

1. Preliminary Inspection: Upon unpacking, please inspect all parts for damage in shipment. If damage has occurred, please notify carrier immediately. Save all packing material for their inspection.

2. Factory Guarantee: You are protected by a one year factory guarantee against defective material or workmanship, subject to inspection and replacement F.O.B. factory. If the unit appears defective, whether new or old, it should be returned to the factory for inspection. This is less expensive than unauthorized field repairs, which may void the guarantee or render minor parts replacement impossible due to damage to major parts.

3. Introduction: The R.A.C. Train Stacksamplr was developed by the Emissions Testing Branch of the Office of Air Programs, E.P.A. It is specified in Volume 42, Number 160 of the Federal Register under the topic "Standards of Performance for New Stationary Sources," "Method 5 Particulate Sampling," and with slight modification "Method 8 Determination of SO_2, SO_3, and Sulfuric Acid Mist," "Method 102 Determination of Particulate and Gaseous Mercury Emissions," "Method 104 Determination of Beryllium Emissions."

The sampling train has four major components: (1) The pitobe assembly includes the probe nozzle, a heated, glass lined (optional) probe, and an "S" type pitot tube for monitoring gas velocity during sampling. (2) The sample case has a heated portion for the filter, cyclone and flask, and an ice bath portion for the impingers. (3) The umbilical cord connects the sample box with the meter box. It is a development of the aquanaut program called "Life Line." (4) The meter case consists of a dual inclined vertical manometer, an orifice meter, vacuum pump, dry gas meter, thermometers and electrical controls for sampling.

4. <u>Assembling the Unit</u>: After the unit has been completely unpacked and checked for breakage and exclusion of parts (use the included packing slip for this purpose) a trial assembly should be completed in the following manner. Not all the procedures presented here need to be completed with a new unit. However, they do help familiarize oneself with the unit and they comprise the necessary maintenance which must be performed as the stacksamplr ages.

<u>Meter Control Case</u>: #201009 - A schematic of the meter case plumbing is shown in Figure 2. The components and a brief description of their function are given below.

The pitot tube lines of the umbilical cord connect through a quick-disconnect fitting to an inclined/vertical manometer in the meter case and the umbilical cord sample line connects through a quick disconnect to the vacuum pump.

The pump intake vacuum, and indirectly the pressure drop across the collection apparatus, is monitored with a vacuum gauge just after the quick disconnect. The pressure drop across the sample apparatus is the gauge vacuum less the stack draft.

A bypass valve is in parallel with the vacuum pump to give fine flow control and to permit recirculation of gases at low sample rate so that the pump motor is not overloaded. Downstream of the pump and bypass valve are a thermometer, dry gas meter, another thermometer, and a calibrated orifice with an inclined manometer in parallel. The calibrated orifice and inclined manometer read in inches of water and are used to indicate the instantaneous sampling rate. The dry gas meter gives an integrated gas-sample volume. The average of the two temperatures on each side of the dry gas meter gives the temperature at which the volume is sampled. The meter pressure is the atmospheric pressure.

The electrical circuitry for the meter case is shown schematically in Figure 3. The electrical controls are shown in Figure 1. The meter case has a pilot light to indicate when the meter case is energized. Five toggle switches are located on the front of the meter case. They are as follows:

1. Vacuum pump and a pilot light.
2. Fan in the sample case and a pilot light on the meter case.
3. Heating element in the sample case and a pilot light on the meter case, all in series with the toggle switch for the sample case fan. This prevents the heating element from operating without the fan; however, the fan can operate without the heating element.
4. Probe heating element controlled by a variable transformer and a pilot light.
5. Orifice meter, manometer, by-pass valve assembly, and pitot light.

Power for the sample case fan, heating element, and the probe heating element along with a common ground are connected to a four-pronged female amphenol in the meter case for ready connection to the umbilical cord.

The meter control case is shipped completely assembled, however, there are several items which should be checked. Unlock the door, open it, and lift it up off the pin type hinges. Remove the cabinet by removing screws around the bottom and the two screws in the upper right and left corners of the front panel and lift the cabinet off. The chassis and front panel will stay intact. Check the general condition of the unit paying particular attention to the tubing and electrical connections.

Check the pump oil, fill to mark if low, note the cabinet has a door on the side so that the oil may be checked regularly, and changed if dirty, without removing the entire cabinet. The door also allows access to the pump filter, which should be cleaned regularly.

Check the dry-gas meter thermometers against a mercury-inglass thermometer. They should read room temperature.

Make sure the quick disconnects are clean. A drop of penetrating oil helps keep them in good working order.

After the manometer is opened, attach the manometer lines to the dual inclined vertical manometer. They are numbered to correspond to the manometer ports. Specific instructions for this procedure are found on the front of the manometer. <u>CAUTION</u>: Always close the ports before transporting the meter control case, this simple procedure will save a large clean-up job and/or costly repair.

Manometer fluids are color-coded: red is used for the pitot tube manometer and yellow is for the orifice manometer for ease of reading in the field.

After making sure the manometer ports are open and the manometer lines connected, level the manometer and check the fluid level. The manometer can be filled with fluid by removing the screw on the left side. When the manometer is zeroed, the fluid-level plunger should have about 1/4-to-1/2-inch travel inward.

Blow through the pitot tube quick connects to check for obstructions. The pitot tube manometer should respond.

After sampling, always check the pitot and orifice manometer lines. They should be free of fluid. Check for leaks, especially around the fluid-level plunger and drain screws. Replace the fluid level plunger or O-rings, when this becomes necessary. Wipe the dual manometer clean. If the dual manometer is unusually dirty, clean as recommended by the manufacturer. Replace the red manometer fluid, if it is faded.

Plug the meter box power cord into a 110-volt outlet. The power indicator light should go on and a click should be heard. The click is the sound of the solenoid valves moving into the position. If the clicking sound does not occur, check the fuse power source, plug, power cord and connections, bulb and solenoid valve.

The valve switch operates the solenoid valve assembly, which contains three 2-way solenoid valves. Two are usually closed and one is open. When the valve switch is turned off, the two open valves close and the flow into the manometer is blocked and the closed solenoid valve opens to allow the pressure to equalize. This allows the manometer to be zeroed while the pump is running.

The solenoid assembly operates only on the orifice manometer. To zero the pitot tube manometer, the pitot tube lines can be disconnected at the quick connects on the meter case.

Turn the coarse-adjust valve to the OFF position, for a short period only and fully open the fine-adjust valve. Turn on all switches to check the lights. The heater light should not go on until the fan switch is also on. If any of the lights do not function, check for defective parts, including switches, light fuses and wiring.

Turn off all switches except the pump switch. Turn on the valve switch and carefully adjust the coarse-adjust and fine-adjust valves. The orifice meter manometer should respond to valve controls. If no movement is observed, check the solenoid valves or the orifice meter system.

Check the Amphenol outlet with a voltmeter or check light by connecting the leads to the different terminals. When connected across the variable transformer, the voltage meter or check light should respond.

Check for leakage by inserting a plugged male quick connect into the sample port. Make sure that the valve switch is off and then activate the pump. Turn the coarse-adjust valve to the ON position and close fully the fine-adjust valve. The vacuum gauge should read about 27 inches of mercury for a barometric pressure of 30 inches of mercury. Check the leakage rate using the reading on the dry gas meter and a watch. If leakage exceed 0.02 cubic foot per minute, find and correct the leak or leaks. Parts to check are the pump, vacuum gauge, metering valves and pipes.

Umbilical Cord: The umbilical cord connects the last modified Greenburg-Smith impinger, the pitot tube, and the heating element at the sampling case to the meter case which is located in a convenient place within 300 feet of the sample port. The umbilical cord consists of five parts:

1. A 3/8 inch vacuum hose with a quick disconnect to the sample head and a quick disconnect connection to the meter case.

2. Two 1/4 inch lines made of tygon tubing with quick disconnects on both ends of the tubing (connects pitot tube to the meter case manometer).

3. Five multistrand insulated conductors with Amphenol connector on both ends(connects sample case circuit to meter case power supply).

4. Ground wire.

5. Sample Head: 996109 - The socket connection of the sample head connects the last impinger to the umbilical cord. It consists of a thermometer, check valve, stainless steel tubing, and a quick-disconnect connection.

The umbilical cord is checked out in the following manner:

Wipe the vacuum line and pitot line (1-2) quick connects clean. A drop of penetrating oil on each helps keep them in good working condition.

Check the dial thermometer on the sample head. The temperature should check against room temperature when a mercury-in-glass thermometer is used as the standard.

Check the electrical lines for continuity using an ohmmeter or a battery-light system. Make sure that the leads are connected to the proper prongs. If there is no continuity in any of the lines, check the Amphenol connections.

Disconnect the joint at the check valve and add a drop of lubricating oil on the valve.

Test the vacuum line for leaks by blocking the inlet of the sample head with a plugged ball joint. Connect the line to the meter case vacuum. If leakage is noted, check all connections first and then, if necessary, check the tubing. If the cause is not readily identified, slightly pressurize the line and check for leaks using soapy water.

Connect one end of the pitot-tube line to a 36-inch U-tube mercury manometer or a similar device. Pull a vacuum of at least 10 inches, seal the tubing at the pump end, and check for leaks by noting the mercury manometer. Do the same for the other pitot-tube line and the vacuum line.

<u>Sample Case</u>: Figure 9 and 9A - The ball joint from the pitobe connects directly to a glass cyclone located in the sampling box, which is designed to remove large particles. The cyclone is followed by a glass filter holder with coarse-porosity fritted glass filter support. Both the cyclone and filter are positioned in the heated portion of the sampling box, which is usually maintained at 250°F. The filter is usually glass fiber of 0.3 micron porosity. Catalog #994553, 2.5", #994554, 3", #994555, 4".

A separate ice bath impinger case is attached to the heated sample case with vertical moving slides. The outlet of the filter in the heated sample case attaches to four Greenburg-Smith impingers in the ice bath case. Only the second impinger has the original tip, the other three have been made without the tip in order to decrease the pressure drop through them. Other glass configurations are available to comply with other test methods.

The front panel assembly houses the heater, fan and motor, adjustable thermostat, and electrical circuitry. A hinged door with two quarter turn fasteners permits easy access to the cyclone and filter assembly.

The sample case is checked in the following manner:

Using an Amphenol adapter, plug into a 110-volt source. The fan and heater should go on. Failure to operate may be caused by a faulty electrical source, a poor connection, a defective fan, or a defective heating element. Check the thermostat operation by monitoring the dial thermometer in the heated section of the case. The temperature will generally fluctuate 20 to 30° about the average after it stabilizes. The usual setting is 250°F, but the thermostat can be adjusted to the setting desired.

Check the probe heater socket with a voltmeter or with a 110-volt check light. Its output voltage is determined by the probe voltage control setting on the meter control case panel. This socket should only be used for the probe as other electrical loads might damage the voltage control.

For recording purposes, the cases should be numbered. At least three sets of the impinger case assemblies, including all the glassware should be on hand for a stack test and preferably more, depending on the number of runs required in a day. The availability of several sets increases efficiency during the test since the sample train can be cleaned while the second run is being made or the trains can be cleaned after all runs have been completed.

<u>Glassware</u>: See Figure 2 - Wash the cyclone, flask, filter holder, frit, glass connectors, and impingers with glass cleaning detergent. After sampling, the hard-to-clean parts are cleaned with dichromate sulfuric acid cleaning solution. Rinse all parts with distilled, deionized water, and then acetone; air dry. Reassemble the impingers using a thin coat of acetone-insoluble silicone stopcock grease on the upper half of the taper joints. After sampling the stopcock grease should be wiped off before washing. When the glassware is stored before sampling, cover exposed openings to avoid contamination.

Assemble the glassware placing the cyclone, filter, and flask in the heated sample case. The side inlet of the cyclone faces the probe outlet, the flask attaches to the bottom of the cyclone and the right angle bend of the filter holder attaches to the top of the cyclone. The impingers are then placed in the ice bath case, standard Greenburg-Smith plated impinger is second if the set up is for particulate sampling. A glass right angle bend connects the filter to the first impinger and glass "U's" connect the impingers in series. Make sure the elbow and "U's" are positioned so that a small exit, either ball or socket is always attached to a larger entrance, either ball or socket, this orientation prevents the sample from hanging up if ball and sockets are not properly aligned.

Probe: See Figure 4 - The following procedure is not absolutely necessary with a new probe, however, the procedure is strongly recommended since it allows familiarization with the new unit. This procedure should be followed after the probe has been used.

To disassemble the pitobe, first remove the Swagelok union except for the welded nut. The nozzle is usually removed in the field and carried separately. Check for broken glass chips in pitobe with optional glass liner.

Remove the glass liner from the sheath and check visually for broken or fractured sections. The most common area of broken or fractured sections is within 1-1/2 inches from the front and approximately 3 inches from the rear. Some fractures on the back end are hard to see because of the tape and rubber stopper. One way of checking is to gently twist the glass probe and listen for grating sounds. Inspect the electrical system of the probe for visible shorts or burned spots shown by uniform darkening of the high-temperature insulation tape, and the power cord connection. Then plug the power cord into a variable transformer and set the power rating at about 55 volts. The probe should become warm to the touch over its entire length in a few minutes. If the probe does not heat, check the variable transformer for proper voltage and for loose connections in the plug and Nichrome wire. Shorts are indicated by partial heating in the rear portion of the probe. Breaks in the Nichrome wire can be checked with an ohmmeter or a battery-light system.

After sampling, if the probe is in good order, per the above procedure, proceed with the cleaning process. Wipe the grease from the ball joint. Clean the probe internally by brushing*, first using tap water, then distilled, deionized water followed by acetone. Rinse the probe with acetone and allow it to dry in the air. Inspect visually for cleanliness and repeat the procedure if necessary.

Remove the quick connects from the pitot tube and wipe them clean. A drop of penetrating oil helps keep them in good working condition.

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*RAC Probe brush available (201062)

Use compressed air to blow out the pitot tube. Both the pitot tube and sheath should be wiped or washed clean with water and air dried. Inspect the pitot tube openings for damage and misalignment, and if necessary, repair and recalibrate the tube.

After the parts are cleaned and inspected, assemble the pitobe components except the nozzle. Insert the liner into the sheath, making sure that the rubber stopper seats properly onto the sheath. The front end of the glass liner should be about flush with the front end of the welded union nut. This prevents the union from pushing against the probe and chipping the end or unseating the rubber stopper. Replace the Swagelok union. The union should be only finger tight. The use of a wrench can cause cracks within the first 1/2 inch of the probe.

Cover the open ends of the probe with serum caps or similar covers to avoid contamination.

The graphs in Figure 5 may be used to determine the probe heat setting required. If the probes are not constructed as outlined or a calibration of the probes is required, similar graphs can be constructed by using a heat source and measuring the inlet and outlet temperatures at several variable transformer settings while passing air through at about 0.75 cubic foot per minute.

Inspect the edge of the nozzles for damage. If damaged, repair and recalibrate. Clean the nozzle by scrubbing and rinse with distilled, deionized water, and then with acetone; air dry. Cover the open ends with serum caps or similar covers to avoid contamination or damage to the knife edge.

Monorail: The Monorail is composed of the following parts, please locate them with the assistance of Figs. 7 and 8.

(1.) Monorail; 5 ft. length for 3 ft. probe, 7 ft. length for 5 ft. probe, and 12 ft. length for 10 ft. probe.

(2.) Pulley assembly (on pulley bar)

(3.) End plates (2)

(4.) Position nuts with springs (2)

(5.) Position bolts (2)

(6.) 1/4 - 20 X 1 in. with nut and washer (1)

(7.) Slotted steel angles (2) 6 ft. lengths

(8.) Slotted steel angle (1) 2 ft. length

(9.) 5/16 X 5/8 in. with nuts (7)

<u>End Plate Assembly</u>: To one end plate, as orientated in Fig. 2, apply the following procedures, (see Figs. 7 and 8).

A. Insert the position bolt through the opening in the tab which is perpendicular to the "T" shaped end plate so that the head of the bolt is outside the "T".

B. Screw position nut (spring toward inside of "T") on bolt so that it catches only the first few threads.

C. Slide Monorail, open end down, over the tab on the end plate and under the position nut so that the grooves in the nut are aligned with the edges of the Monorail and are holding the Monorail as shown in Fig. 7.

D. Tighten position bolt and Monorail will be held firmly to end plate. Make sure end plate is butted to Monorail.

E. Slide pulley assembly, preassembled to pulley bar, into the Monorail as shown in Fig. 8.

F. Repeat steps A through D with second end plate. This will hold pulley assembly captive on Monorail.

Mounting Monorail: Figure 6 - There are many ways of attaching the Monorail to the stack and of supporting the other end. These procedures depend on the type of stack and the location of the sampling port with reference to a roof top or cat-walk. Outlined below are several general procedures which should be followed when mounting the unit.

A. Either end plate may be attached to the stack, however, the direction in which the prongs on the pulley bar are pointed, to the left or right with respect to the sides of the Monorail, will determine the orientation of the sample case. When looking down the length of the Monorail toward the stack the prongs of the pulley bar should point to your left. Thus when the sample case is hung on the pulley bar with the Monorail orientated in this manner the probe will be pointed in the appropriate direction.

B. Mount appropriate end plate to the stack. Consult Fig. 7 for hole sizes and locations.

C. The end plate not attached to the stack may be supported in various ways. The two 6 ft. slotted angles are generally attached to the end plate and these legs spread to form an "A", the 2 ft. section being used as the cross member of the "A" to add rigidity. The 6 ft. sections may be attached to the end plate anywhere along their length depending on the desired height of the Monorail. Use the 7 - 5/16 X 5/8 in. bolts with nuts to secure the slotted angles in the desired position.

Mounting the Sample Case: It is suggested that the sample case be mounted before water and ice are added to the condensing section to eliminate unnecessary strain.

A. Lift the sample box to the height of the pulley bar and slip the prongs through the corresponding holes in the T-bar on the Monorail cabinet support.

B. Push in on the mounting handle until it mates with the captive nut on the pulley bar and tighten.

The sample case can be removed from the Monorail by simply reversing the two above steps.

Field Operation: Presurvey:

Sampling sites on the stack should be selected so that there is a uniformity of gas mixture and the stack gases should be as free as possible from turbulence. When selecting a site the usual rule is for measurements to be made, eight diameters downstream from a disturbance and three diameters upstream. Disturbances are usually caused by inlets, outlets, bends or constrictions in a stack. This rule cannot always be adhered to and in such cases compromises must be made. In doing this, flow measurements and sampling techniques must be carefully applied to minimize errors.

Because sampling approaches vary, the description of the operation of the isokinetic sampling equipment will be confined to the use of the nomographs, assembly of the components, and adjustment of the flow rate to isokinetic conditions once the probe is positioned at the desired sampling point.

Nomographs:

Slide rule nomograph Side 1 and 2 (Standard) #201014
Consult the instruction booklet titled "Programmed Stack Calculator Instruction Book" which accompanied your slide rule nomograph. Instruction booklet can be found in the leather carrying case that is provided for your nomograph.

E.P.A. type nomograph (Optional) #997600
Consult the instruction sheet which accompanied your E.P.A. nomograph.

Pitobe Assembly:

Remove the cover from the nozzle side of the stainless steel union on the probe sheath.

Remove the cover from the shank end of the selected nozzle (obtained from the nomograph), insert the nozzle in the probe sheath union, and finger tighten the union. Avoid the use of wrenches as this would bind the ferrules to the nozzle shank and could cause the glass probe to crack. Keep the nozzle tip and the ball joint on the glass probe covered until the assembly of the equipment is complete and sampling is about to begin. 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. This distance was determined when the stack was divided into sampling areas.

Attach a temperature probe to the metal sheath of the sampling probe so that the sensor does not touch any metal. Its position should be in accordance with Figure 10 to avoid interference with the gas flow. Probes with pyrometers in place can be supplied by Research Appliance Company.

Sample Box Assembly:

Place the tared filter in the filter holder and record the filter number on a data sheet. Tighten the clamps around the filter holder to prevent leakage around the rubber gasket.

Assemble the collection system, as shown in Figure 2, or as dictated by the requirements of the test, using a very light coat of silicone grease (acetone non-reactive) on the lower half of all the male ball joints.

Leak Test:

Place the meter case in a convenient position up to 300 feet from the test port and connect the vacuum line of the umbilical cord to the inlet of the meter case and the sample head. Place a ball point stopper in the inlet to the cyclone to check for possible leakage. Turn the coarse-adjust valve on the meter box to the OFF position and open the fine-adjust valve slowly until fully opened. Partially close the fine-adjust valve until the vacuum gauge read 10 inches of mercury; then check the dry gas meter for flow. If the flow through the dry gas meter exceed 0.02 cubic foot per minute at 10 inches of mercury gauge pressure, the leak or leaks must be found and corrected.

Final Sampling Train Assembly:

Remove the cover from the ball joint on the probe, lightly grease the lower half of the ball joint with silicone grease, and connect the probe. Make sure that the rubber stopper is snug in the opening in the sample case. Secure the probe by tightening the probe holding clamp.

Make all necessary electrical and pitot-tube line connections. Set the variable transformer on the meter case so that the desired probe temperature is obtained (use Figure 5 as a guide). Set the Thermostat on the sample box at 250°F or at the desired temperature. Turn on the heater, blower, and probe switches on the meter case. Allow the sample case and probe to heat for at least 15 minutes before starting the test and make periodic checks and adjustments to assure the desired sample case temperature.

Fill the impinger section of the sample case with ice and a little water. Add ice periodically during testing to maintain the temperature of the gas leaving the last impinger at less than 70°F.

Sample Run:

Record all necessary initial data such as that shown on the sample data sheets in Appendix A, including the initial dry gas meter reading. Turn off the coarse-adjust valve on the meter case, fully open the fine-adjust valve. Remove the cover from the nozzle tip and place the pitobe at the first sampling point. Record the clock time, read ΔP on the pitot tube manometer, and determine ΔH from the nomograph as shown previously.

Turn the pump on and set ΔH on the meter box first by adjusting the coarse-adjust valve and then the fine-adjust valve.

The sample run plan should consider the number of traverse points and the sampling time at each point. A good rule of thumb to follow is to collect a weight of particulate matter equal to about 20 per cent of the filter weight. The upper limit is usually set by the pressure drop across the filter and the amount of condensate the impingers can hold. Generally the length of sampling time at each traverse point is 10 minutes.

During the sample traverse the pitobe is moved from point to point without turning the pump off except when changing ports. The ΔP should be monitored and adjustments made on the orifice meter with the aid of the nomograph when necessary. Besides the regular time interval recordings, a set of readings should be recorded when the ΔP changes by more than 20 per cent.

Shutdown:

When testing has been completed, take a final set of readings, turn off the adjusting valves and then the vacuum pump. Turn off the heater, blower, and probe heat switches and remove the probe from the sampling port and read the dry gas meter. Cover the nozzle tip as soon as it is cool enough in order to avoid contamination or loss of sample. Disconnect the probe from the cyclone inlet and cover both the end of the probe and the inlet to the cyclone. Disconnect the umbilical cord from the sample case and cover the last impinger outlet. The probe and sample case are then ready for cleanup and the analytical procedure.

FIGURES ONE THROUGH TEN

MODEL 2414

STAKSAMPLR WITH MODULAR SAMPLE CASE

RESEARCH APPLIANCE COMPANY
P.O. Box 265
Cambridge, Maryland 21613

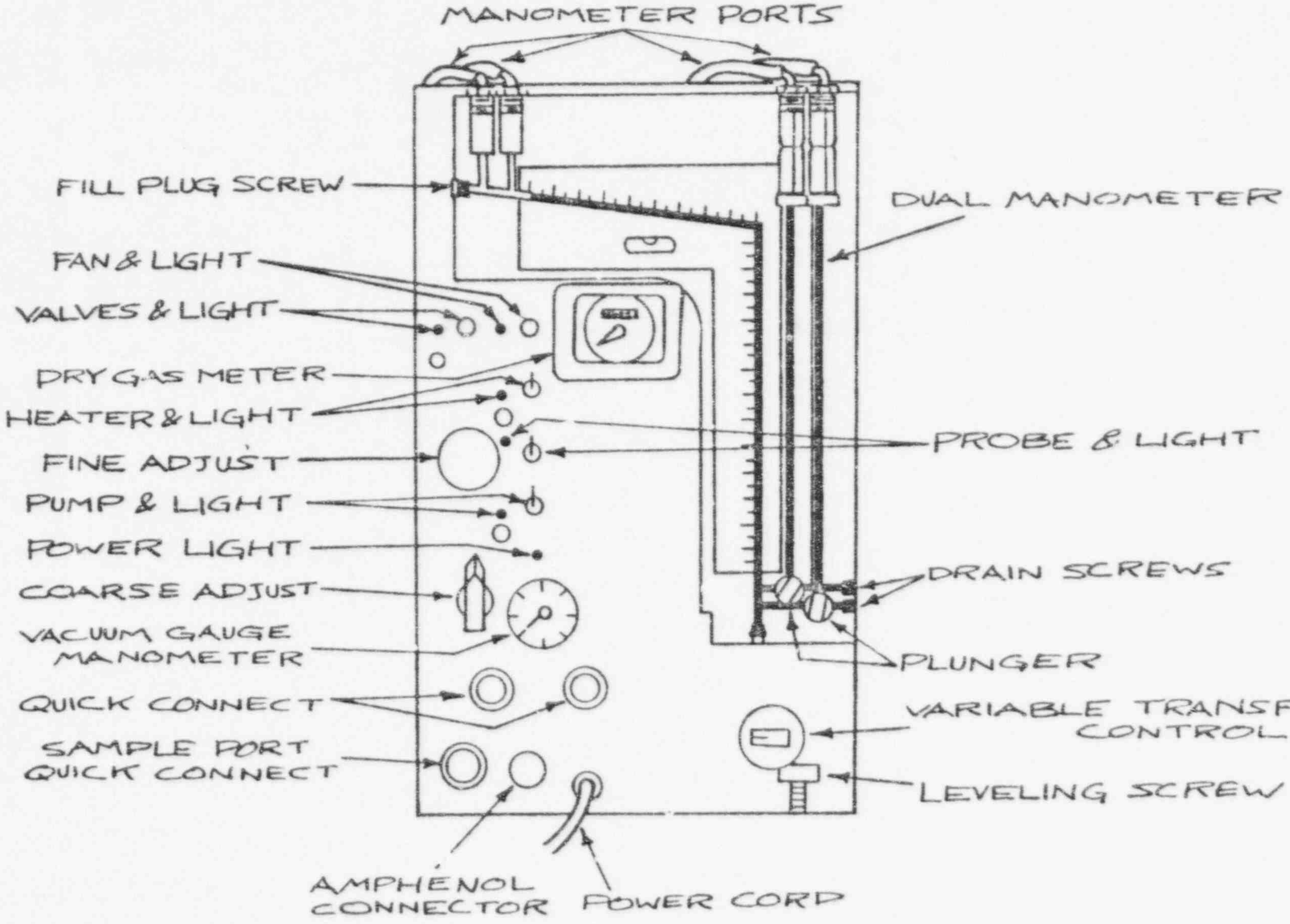

MANOMETER PORTS
FILL PLUG SCREW
DUAL MANOMETER
FAN & LIGHT
VALVES & LIGHT
DRY GAS METER
HEATER & LIGHT
PROBE & LIGHT
FINE ADJUST
PUMP & LIGHT
POWER LIGHT
DRAIN SCREWS
COARSE ADJUST
VACUUM GAUGE MANOMETER
PLUNGER
QUICK CONNECT
VARIABLE TRANSFORMER CONTROL
SAMPLE PORT QUICK CONNECT
LEVELING SCREW
AMPHENOL CONNECTOR
POWER CORD

FIGURE 1

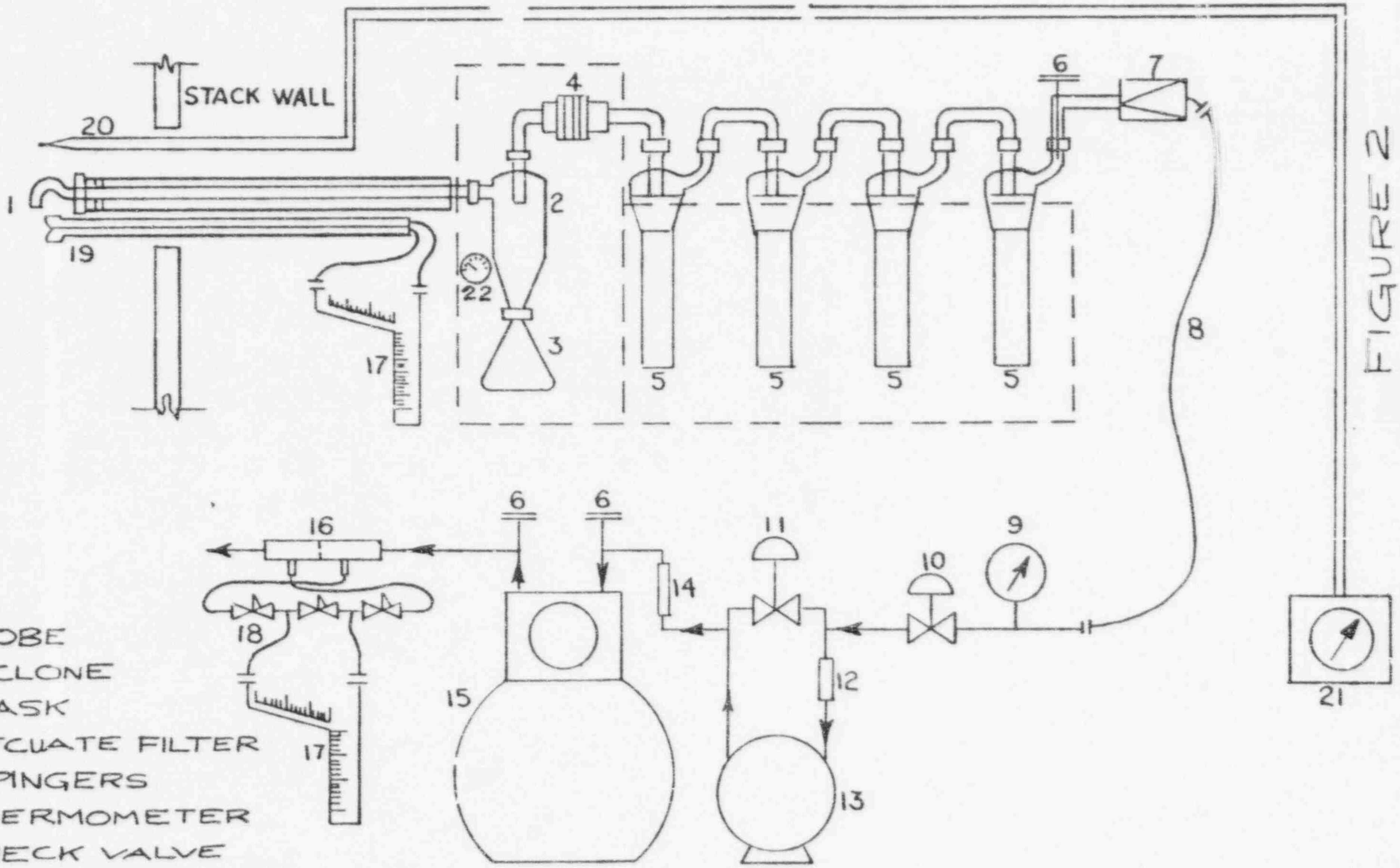


FIGURE 2

1. PROBE
2. CYCLONE
3. FLASK
PATCUATE FILTER
5. IMPINGERS
6. THERMOMETER
CHECK VALVE
8. UMBILICAL CORD
VACUUM GAGE
0. COURSE FLOW ADJUST VALVE
FINE FLOW ADJUST VALVE
2. OILER
3. VACUUM PUMP
14. FILTER
15. DRY GAS METER
16. ORIFICE TUBE
17. INCLINE MANOMETER
18. SOLENOID VALVES
19. PITOT
20. THERMOCOUPLE
21. PYROMETER
22. THERMOMETER

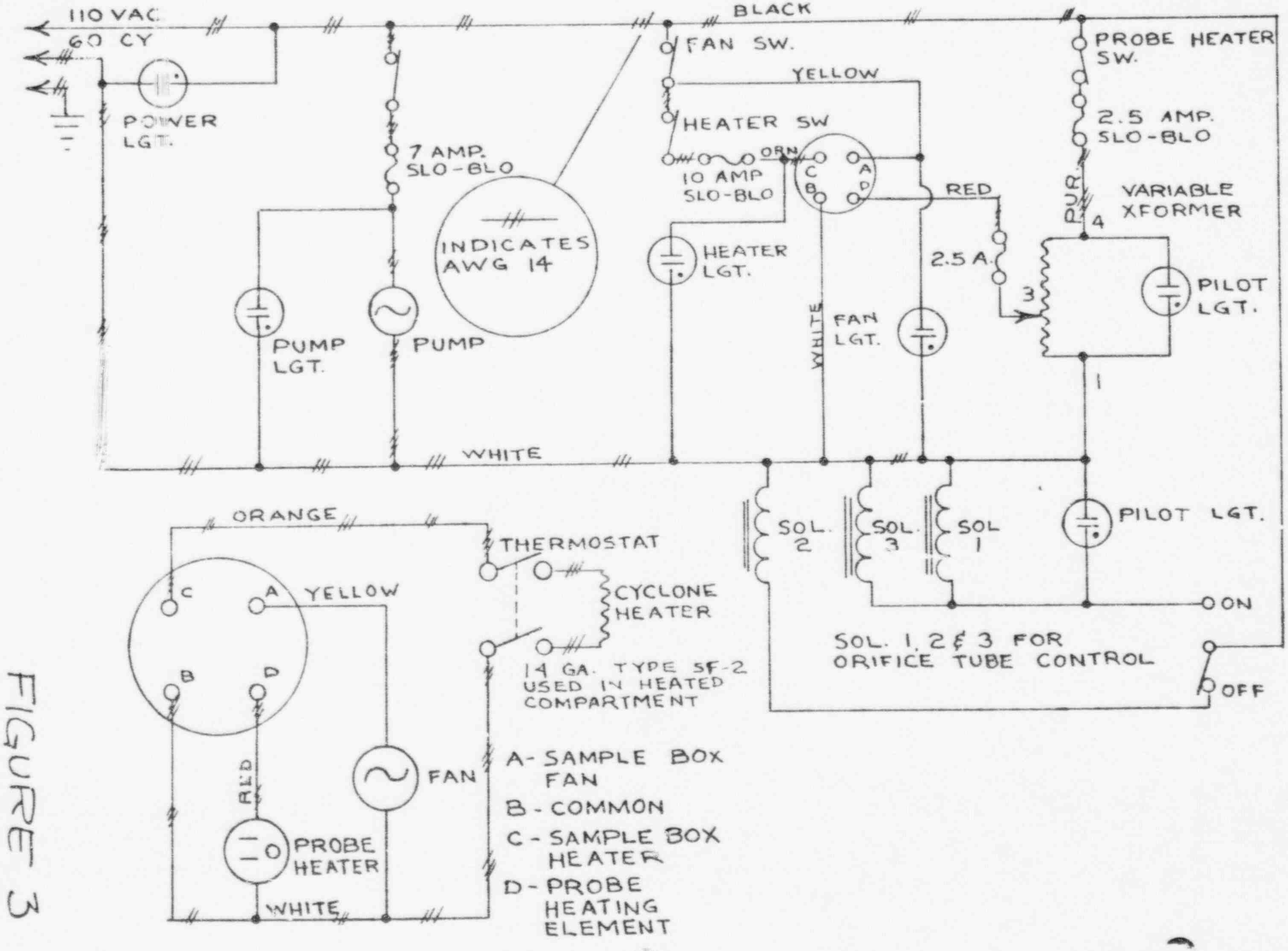

110 VAC
60 CY
POWER LGT.
7 AMP. SLO-BLO
PUMP LGT.
PUMP
INDICATES AWG 14
BLACK
FAN SW.
YELLOW
HEATER SW
10 AMP SLO-BLO
ORN
C
B
A
D
HEATER LGT.
WHITE
FAN LGT.
RED
2.5 A.
PROBE HEATER SW.
2.5 AMP. SLO-BLO
PUR.
VARIABLE XFORMER
4
3
1
PILOT LGT.
WHITE
SOL. 2
SOL. 3
SOL 1
PILOT LGT.
ON
OFF
SOL. 1, 2 & 3 FOR ORIFICE TUBE CONTROL
ORANGE
THERMOSTAT
CYCLONE HEATER
14 GA. TYPE SF-2 USED IN HEATED COMPARTMENT
C
A
YELLOW
B
D
RED
FAN
PROBE HEATER
WHITE
A - SAMPLE BOX FAN
B - COMMON
C - SAMPLE BOX HEATER
D - PROBE HEATING ELEMENT

FIGURE 3

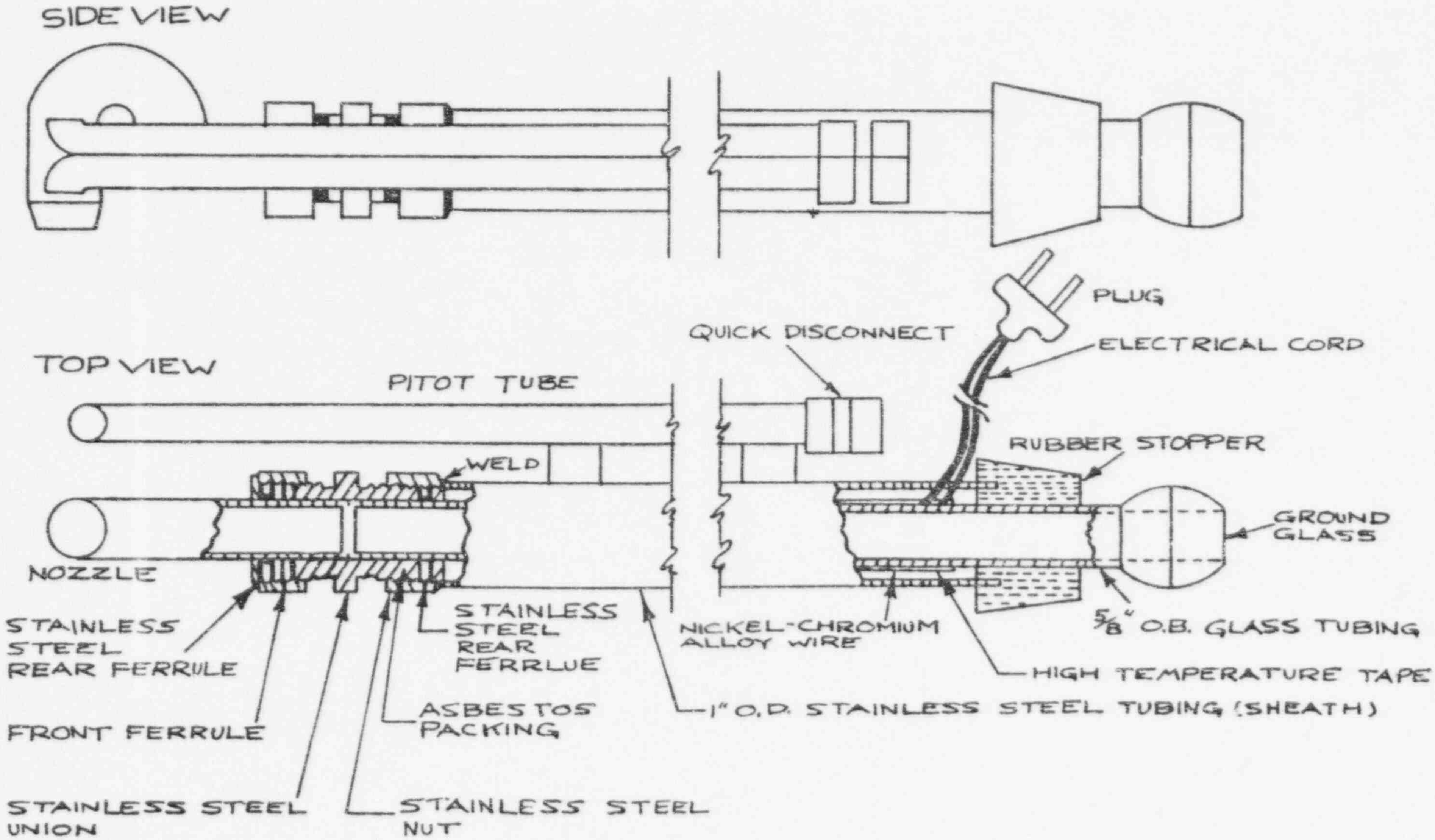
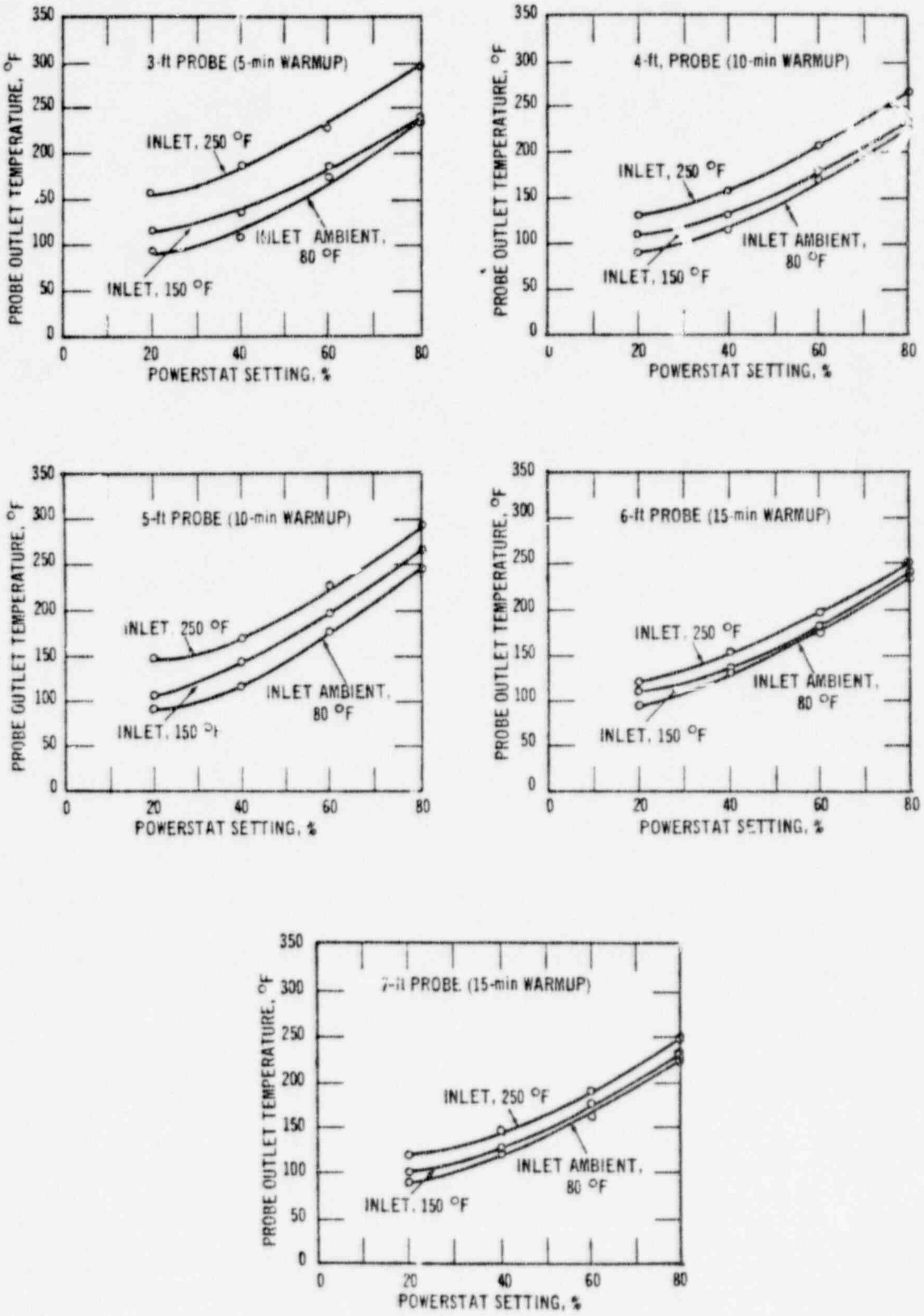


FIGURE 4

NOTE:

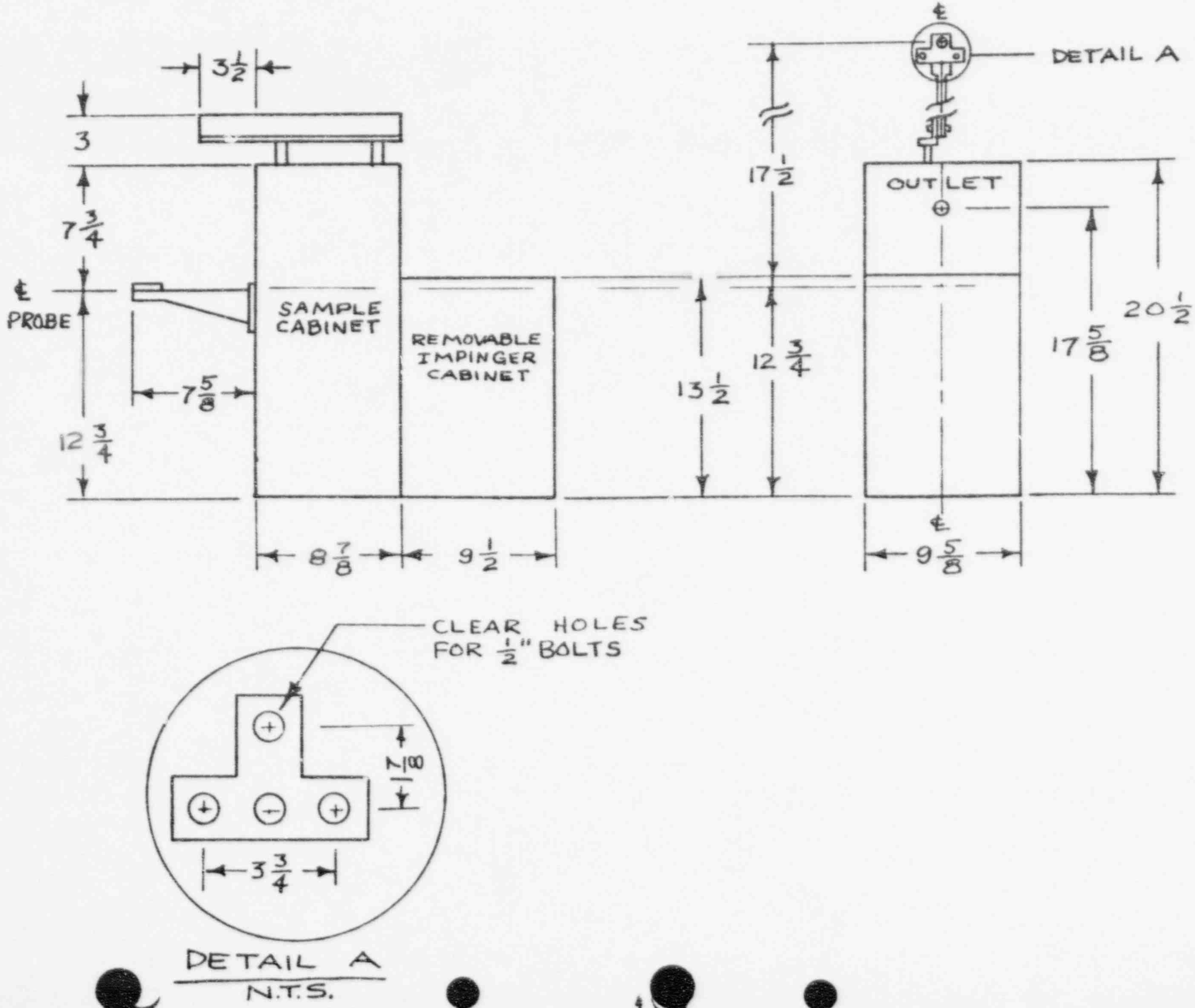
1. PITOBE LENGTH CAN BE ANY SUITABLE SIZE.

2. NOZZLE DIAMETER AND CONFIGURATION CAN BE A VARIETY OF SIZES, BUT IT MUST HAVE A 5/8" SHANK.



NOTE: Flow rate held constant at 0.75; 50% change in flow rate has little effect on probe temperature.

FIGURE 5


3 1/2
3
7 3/4
℄ PROBE
SAMPLE CABINET
REMOVABLE IMPINGER CABINET
7 5/8
12 3/4
8 7/8
9 1/2
13 1/2
12 3/4
17 1/2
℄
DETAIL A
OUTLET
17 5/8
20 1/2
℄
9 5/8
CLEAR HOLES FOR 1/2" BOLTS
7/8
3 3/4
DETAIL A
N.T.S.

FIGUER 6

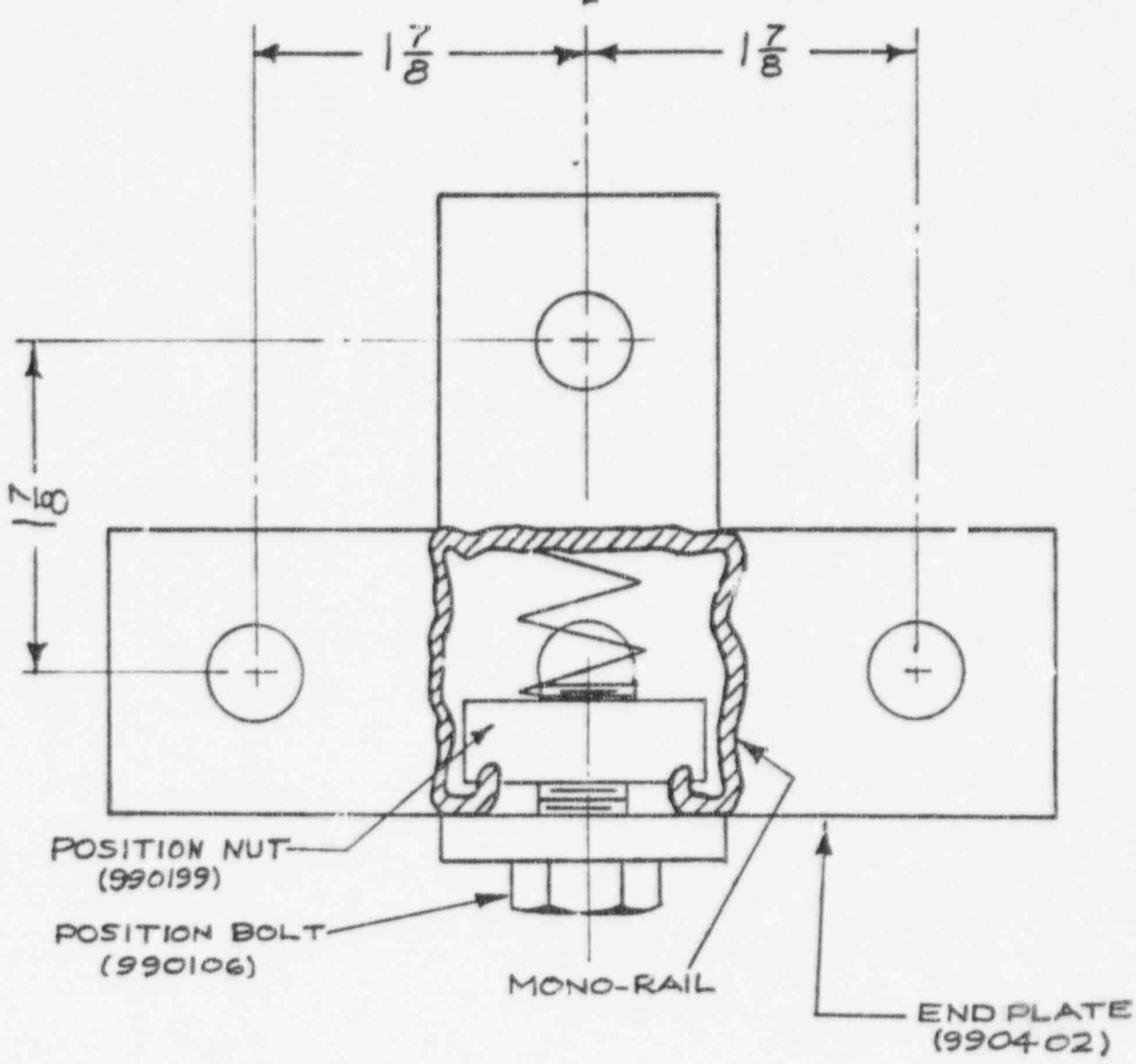

℄
1 7/8
1 7/8
1 7/8
POSITION NUT (990199)
POSITION BOLT (990106)
MONO-RAIL
END PLATE (990402)

FIGURE 7

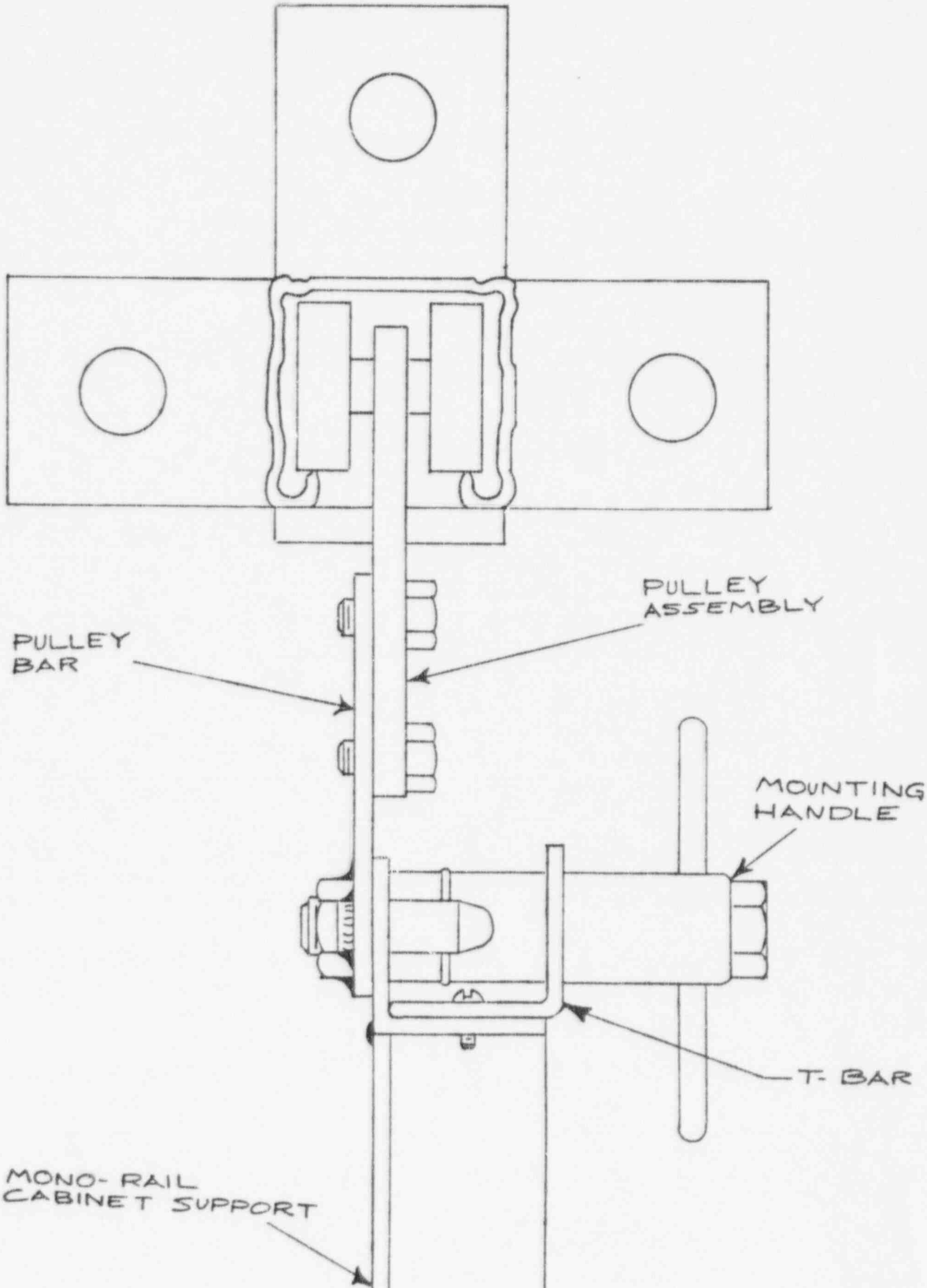

PULLEY ASSEMBLY
PULLEY BAR
MOUNTING HANDLE
T- BAR
MONO- RAIL CABINET SUPPORT

FIGURE 8

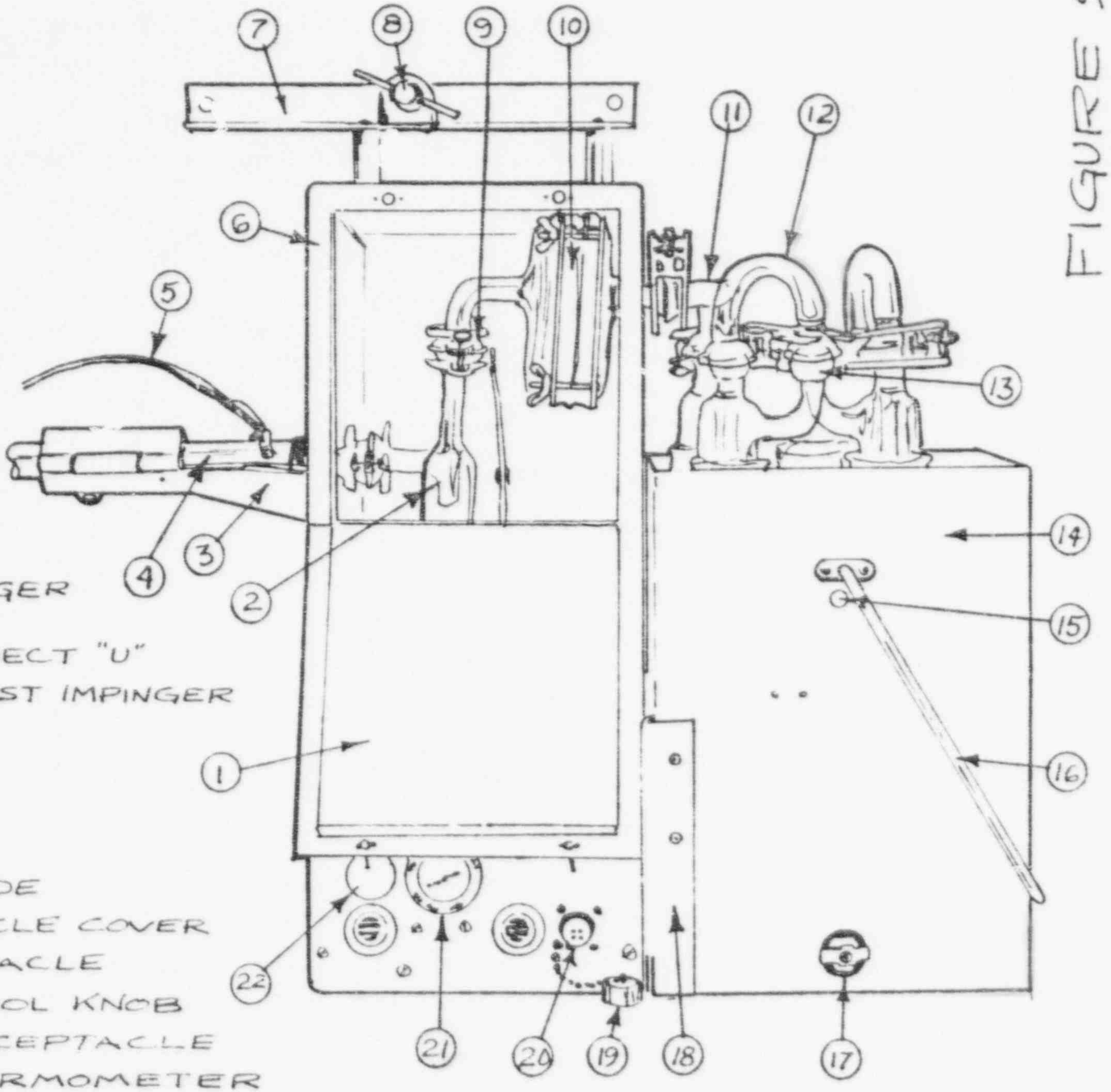
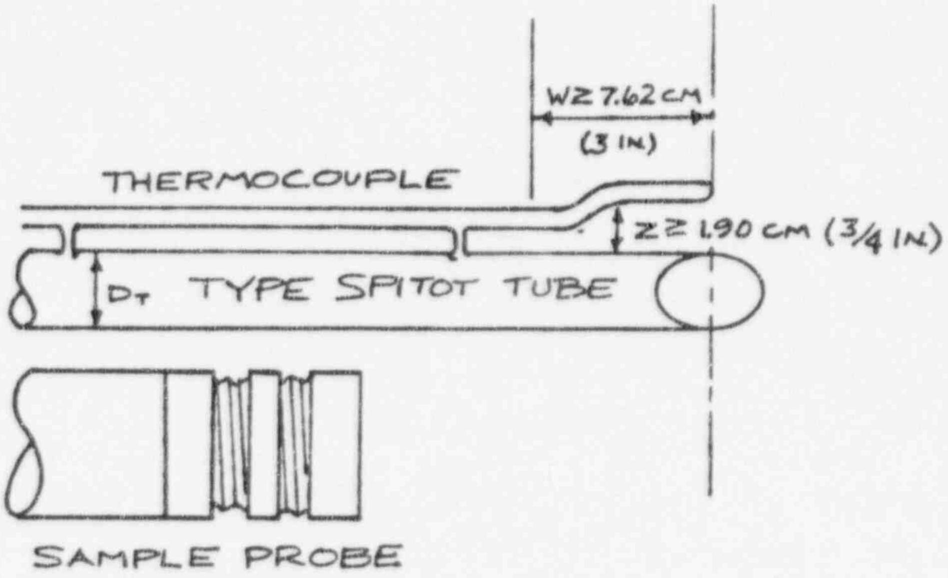
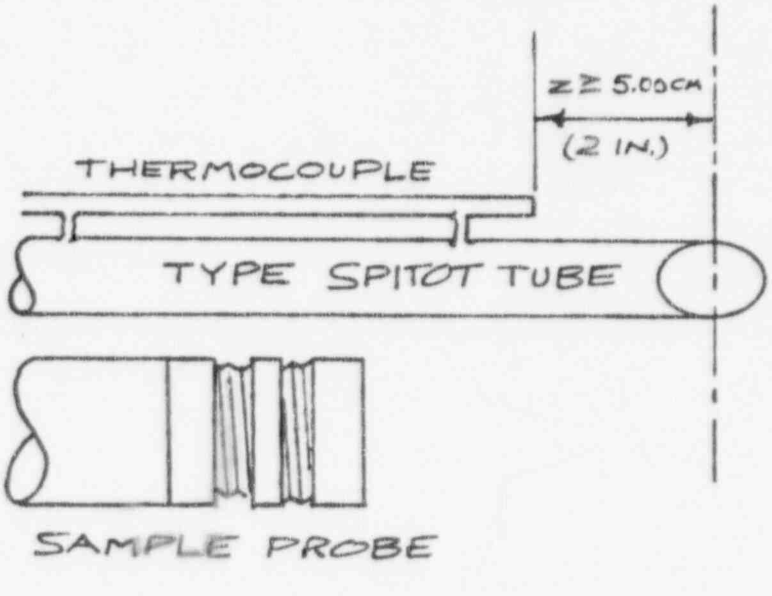


FIGURE 9

1. DOOR
2. CYCLONE
3. PROBE SUPPORT
4. PROBE
5. PROBE HEATER CORD
6. MODULAR SAMPLE CASE
7. MONORAIL ANGLE
8. MONORAIL HANDLE ASSEMBLY
9. BALL JOINT CLAMP
10. FILTER ASSEMBLY
11. FILTER TO FIRST IMPINGER ELBOW
12. IMPINGER INTERCONNECT "U"
13. BALL JOINT OUTLET, LAST IMPINGER
14. IMPINGER CASE
15. OVERFLOW HOLE
16. CARRYING HANDLE
17. DRAIN COCK
18. IMPINGER CASE SLIDE
19. AMPHENOL RECEPTACLE COVER
20. AMPHENOL RECEPTACLE
21. THERMOSTAT CONTROL KNOB
22. PROBE HEATER RECEPTACLE
23. HOT SECTION THERMOMETER



OR



PROPE THEROCUPLE PLACEMENT TO PREVENT INTERFERENCE; D_T BETWEEN 0.48 AND 0.95 CM (3/16 AND 3/8 IN.).

FIGURE 10

3.0 CALIBRATION

Calibration of stack sampling equipment as done at White Mesa, on a periodic basis, is as per the attached forms. During full operation, the equipment is checked on a semi-annual basis, and during partial operating periods, calibration frequency is annually. During non-operational periods, calibration of the equipment is not performed.

CALIBRATION PROCEDURES

ORIFICE AND METER

Initial Check: A preliminary check is made of the following for proper operation before trial test: pump, pump oil, quick connects, valves, manometers, dry test meter, thermometers, lights, electrical check amphenol, variac, vacuum gauge, leakage at 27".

COVER OFF

PLUG IN POWER:	Bottom light on, solenoid click.
PUMP OIL:	Minimum level.
PUMP:	Provides sufficient suction when in operation.
QUICK CONNECTS:	Proper locking operation.
VALVES:	Proper opening and closing.
MANOMETERS:	Fluid level and zeroing.
DRY TEST METER:	Thermometers (inlet, outlet) do not hang up.
THERMOMETERS:	Calibrate to a known temperature.
LIGHTS:	Turn fan, heater, probe on; turn fan off and heater light also goes out. Turn fan on and heater off. Lights should indicate position.
ELECTRICAL CHECK AMPHENOL:	Continuity check.
VARIAC:	Connect neon bulb electrically to amphenol and turn variac up beyond 45 watching neon increase in brightness.
VACUUM GAGE:	Turn selector valve (course) to "on" position, fine adj. to fully open; insert plug (plug input line); turn fine adj. until gage reads 27" and hold for 15-30 seconds, observe dry meter for movement, check for leaks.

Introduction: You are now ready to follow calibration procedures. $\Delta H@$ and Y can be obtained simultaneously. After warmup and wetting, set wet meter at zero, start stop watch when dry meter reading is at a convenient position and record all information required on the following page. When 10 CF_w have gone through the meter stop the stop-watch and note dry meter for CF_d. Perform all calculations, and if readings are not within tolerance make adjustments as outlined.

TERMINOLOGY:

CFM	Cubic foot/minute
$\Delta H@$	Orifice pressure differential in inches of water that gives 0.75 CFM of air at 70°F and 29.92 in. Hg.
Y	Dry meter differential (in. H_2O)
P_b	Barometric pressure (in. Hg.)
CF_w	Cubic feet (wet test meter)
CF_d	Cubic feet (dry test meter)
T_w	Temperature (wet test meter)
IT_d	Input temperature (dry) °F
OT_d	Output Temperature (dry) °F
T_d avg.	$(IT_d + OT_d)/2$ °F
T	Time (minutes)
WTM	Wet test meter

Procedure: With the meter and pump enclosed and a wet test meter attached (1 cubic foot per revolution), allow the equipment to warm up and dry parts of WTM to become wetted (approximately 1/2 hour). Level the box by turning the adjusting screw in the lower right front corner. Then zero the manometer by placing the manometer switch in the up position and adjusting fluid level if necessary. After the warmup period run a trial test at an orifice manometer setting of 2 in. H_2O and CF_w of 10 to obtain $\Delta H@$ and Y. See Fig. 1 for test set-up.

Step One:

ΔH@ is calculated by using the formula:

$$\Delta H@ = \frac{0.0634\,(\text{manometer orifice})}{P_b\,(OT_d + 460)} \left[\frac{(T_w + 460)t}{CF_w}\right]^2$$

A different factor is used for the manometer orifice for each successive ΔH set on the orifice manometer in accordance with the following table:

Set ΔH	Manometer Orifice Factor
.5" H_2O	.01585
1	.0317
2	.0634
4	.1268
6	.1902
8	.2536

If the value is not within 1.6 to 2.1 (1.84 nom. desired) the orifice can be drilled in increments of 64ths to lower the value, or if the value is below 1.6, then the orifice should be replaced and the procedure repeated.

Step Two:

Y is calculated by using the formula:

$$Y = \frac{CF_w\,P_b\,(T_{d\ avg.} + 460)}{CF_d\,(P_b + 0.147)\ (T_w + 460)}$$

and is obtained during the trial run along with ΔH@.

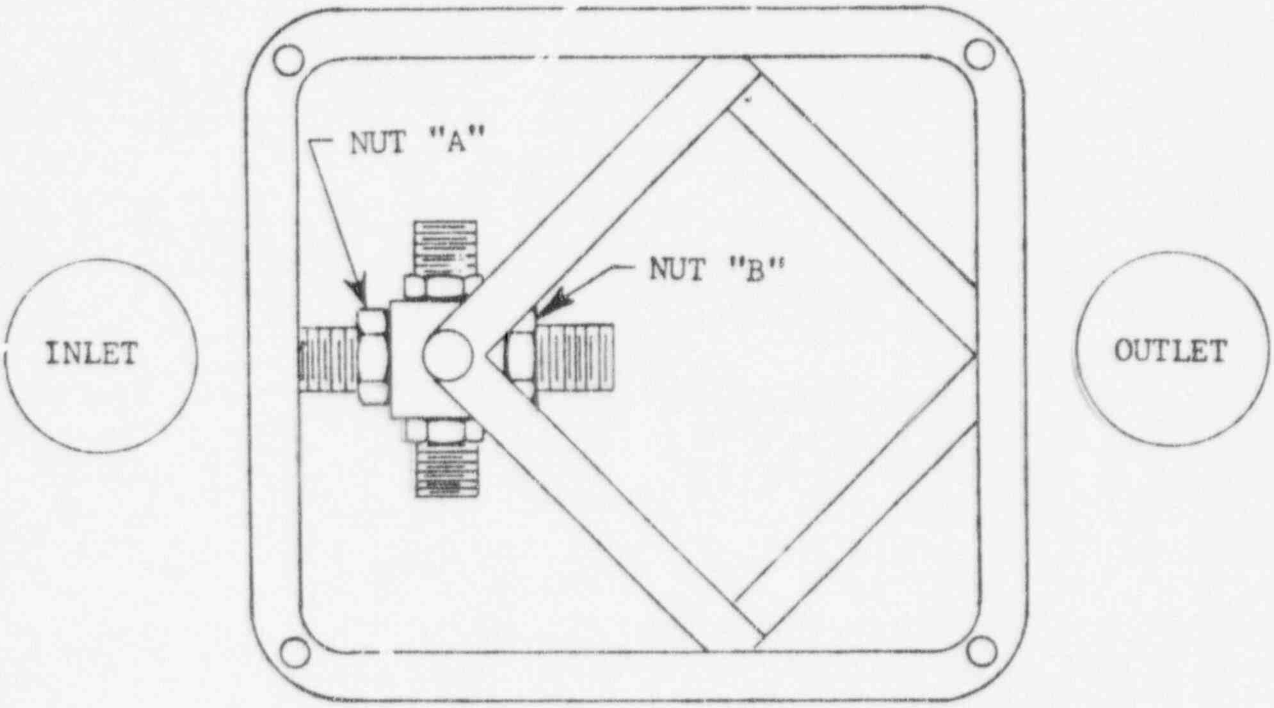
The factor 0.147 is replaced in the formula as the ΔH is changed. The following table lists the factors to be used with the various ΔH settings.

Set ΔH	Formula Factor
.5" H_2O	.0368
1	.0737
2	0.147
4	0.294
6	0.431
8	0.588

If the value is not within 0.99 and 1.01 (1.00 nom. desired) then adjustments must be made to the meter. If the unit is still under warranty return to the factory, adjustments in the field will void the warranty. To adjust the meter, remove the four (4) screws on the top of the meter, carefully remove the lid, and exposing the lever mechanism make sure that the adjusting nuts "A" and "B" are in position, see Fig. 2. To adjust the meter, "A" and "B" are turned clockwise to decrease the value of Y and counterclockwise to increase the value of Y. After making the adjustment replace the lid and screws and run another trial test.

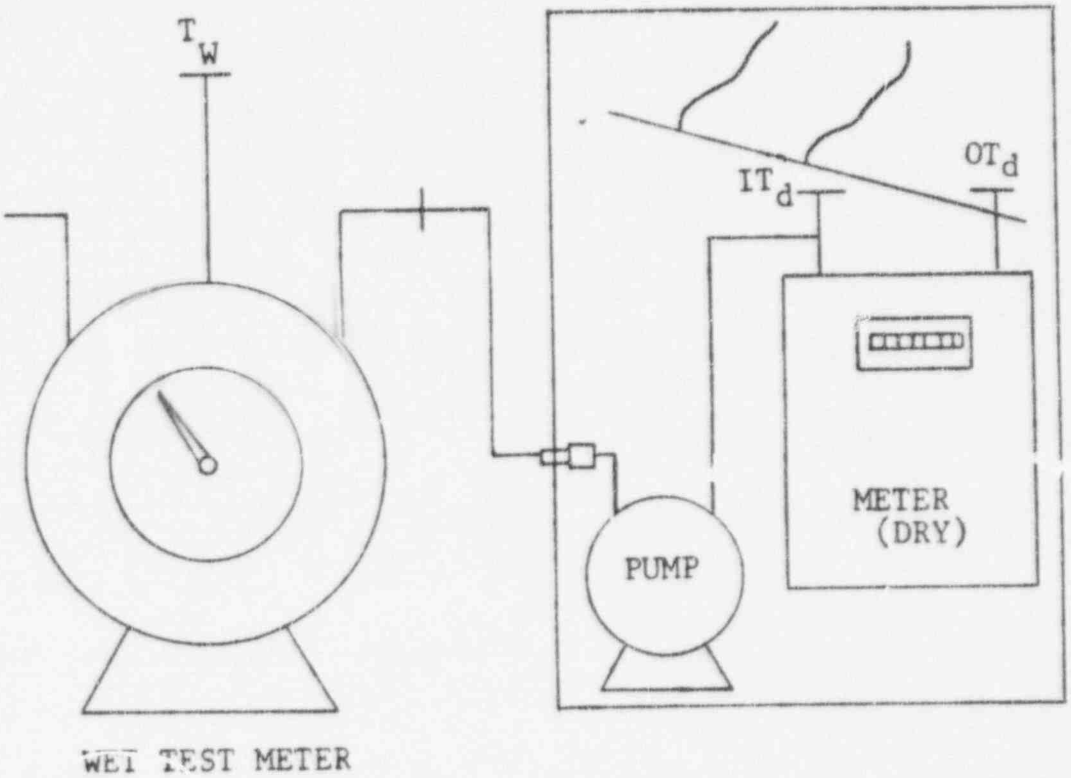
RW/1-7-75

TOP OF DRY METER



WHEN REMOVING LID FOR ADJUSTMENT USE CAUTION IN REMOVING GASKET SO IT DOES NOT BREAK. WHEN ADJUSTING NUTS HAVE STRING ATTACHED TO WRENCHES.

TEST SET UP SCHEMATIC



WHEN PERFORMING VACUUM CHECK PUT PLUG (QUICK-CONNECT) ON INPUT SIDE OF PUMP

FIG. 1

4.0 RECORD KEEPING

Record maintenance as per the formats shown in Section 2.0, are maintained in the WHITE MESA AMBIENT ENVIRONMENTAL notebook maintained in the Environmental Office. A summary log is maintained of each monitoring run results and the same for a history log of that particular stack's performance. This data is also on the computer system.

Data located on a run summary log includes:

1. Stack and Run ID:
2. Date and Sampler;
3. Sampled Air Volume at standard conditions;
4. Sampled Water Volume at standard conditions;
5. Moisture Content; Volume Basis;
6. Stack Gas Molecular Weight (wet basis);
7. Stack Gas Velocity;
8. Stack Gas Volumetric Flow Rate, dry basis at standard conditions;
9. Particulate Concentration;
10. Percent IsoKinetics; and
11. Emission Rates for Particulates, Uranium-Nat., Thorium-230, Radium-226 and Lead-210.

The history log maintains a record summary for each stack of the following data:

1. Data Sampled;
2. Stack Gas Volumetric Flow Rate, dry basis at standard condition;
3. Percent IsoKinetic;
4. Emission Rates for Particulates, Uranium-Nat., Thorium-230, Radium-226 and Lead-210.

Data is calculated to give emission rates in pounds per hour for particulates and picoCuries per hour for radionuclides.

5.0 MONITORING LOCATION AND FREQUENCY

During non-operational periods, monitoring is not done.

5.1 Yellow Cake Stacks

The exhaust stack for the drying and packaging equipment associated with the yellow cake calciner is sampled on a quarterly basis during operations. The sample ports are located on the roof of the main mill building.

Samples are analyzed for: Natural Uranium, Lead-210, Radium-226, and Thorium-230 on a semi-annual basis and for Natural Uranium on a quarterly basis.

5.2 Crusher Stacks

The crusher stacks are located at:

1. Ore Buying Station (3) and,
2. Ore Hopper Feed (1)

These stacks are accessible from stack platforms and are sampled on a semi-annual basis during operations. Samples are analyzed for: Natural Uranium, Lead-210, Radium-226, and Thorium-230.

6.0 ANALYSIS PROCEDURE

The lab analytical procedures utilized are as presented in the Representative Analytical Laboratory Procedure Manual.

Field sample handling consists of sampled air volume moisture determination, sampled gas volume, percent isoKinetic, stack air emission rate, particulate weight sampled, particulate emission rates determinations, measuring collected condensate volumes, rinsing glassware, and collection of condensate and rinse solutions for analysis.

All glassware is pre-rinsed with 0.1N nitric acid, and all solutions used in impinger traps contain 0.1N nitric acid.

After sampling, the glassware and stainless steel units are rinsed with 0.1 nitric acid, with the rinse added to the condensate solutions. The glassware is then rinsed with isopropyl alcohol and stored.

Samples submitted for analysis for each run are:

1. Glassfiber filters, net gain weight determined at White Mesa;
2. Condensate and rinse solutions (volumes of each determined at White Mesa);

6.0 ANALYSIS PROCEDURE (continued)

3. Glassfiber filter blank; and
4. 0.1N Nitric Acid blank.

Results received from the analytical laboratory are in picoCuries per filter and/or picoCuries per milliliter of sample. With knowledge of volumes of condensate and rinse used, along with blank values, a tabulation is made to determine net isotope gain for the run in picoCuries. This value divided by the known sampled volume (dscfm) gives the emission concentration in activity per standard air volume. This value, in turn, multiplied by the known standard air volume stack emissions rate gives activity emission rates for that particular sampling period.

7.0 QUALITY ASSURANCE METHODOLOGY

7.1 Equipment Operation

Prior to performing an emission point sampling run, the sampling equipment is subjected to a dry run test to determine leakages or equipment malfunction. Calibration of equipment is checked on a periodic basis. Probe tips are protected by a protective cap while not in use to protect accuracy determinations. During transport of equipment, all openings are sealed to prevent contamination. Calculations utilized during runs to maintain isoKinetic conditions are reviewed and dry run tested prior to the actual run. All containers and probes are washed prior to each usage.

Malfunction of sampling equipment, excessive malfunctions of normal operations being monitored, or percent isokinetic sampling rates greater than plus or minus ten percent error indicate mandatory voiding of the run or data involved.

7.2 Operations

Operations such as the calciner or crushers that appear, in the judgement of the sampler, to be functioning in a manner not consistent with normal operations shall void the sampling run.

7.3 Chemical Sample Control

Analyses on each period's sample shall include blanks for both the filters, impinger solutions, and the rinse solutions. A field log book shall be maintained listing data generated, determinations of volumes measured, and net gain weights of filters to provide a backup to summary data records. Filters are transmitted within plastic enclosed petri dishes.

7.0 QUALITY ASSURANCE METHODOLOGY (continued)

Handling of filters is only done using tweezers.

7.4 Calculations

All calculations are done using a Texas Instruments 59 calculator and programs written for that purpose. The calculations are rechecked by another member of the staff to minimize data numerical transposition.

WHITE MESA PROCEDURES MANUAL

MONITORING GUIDELINES
ENVIRONMENTAL

ANALYTICAL BALANCES

1.0 EQUIPMENT

Equipment used is:

1. Sartorius Model 2432
   Electronic Balance
   200 gram capacity
   0.1 milligram sensitivity
   Serial Number 2903004

2. OHAUS Triple Beam Balance
   2610 gram capacity
   0.1 gram sensitivity

3. Standard Calibration Weights

The electronic analytical balance is mounted on a heavy shock-proof table and balance level is checked prior to each use and adjusted when necessary. The interior of the balance housing is maintained in a clean condition.

2.0 PROCEDURES FOR USE

The procedures for using the analytical electronic balance are:

1. Check for levelness, re-level if necessary.

2. Clean pan using camel-hair brush and shut doors.

3. Check zero:
   Set all controls to zero. Turn release lever down. If scale does not stop on zero, adjust with zero control knob (right side behind micrometer control knob).

4. Weighing an unknown:
   Place object on pan with release lever in OFF position. Ensure object does not touch sidewalls. Shut doors. Turn release lever to "1/2" position. Dial in weights, starting with largest series. When too much weight is added, the scale moves below zero; go back one step (if 30 is too much, go to 20). Then use next lower series (1-9 grams).

5. Reading the result:
   When all weights are used, turn release lever to "1" or down

2.0 PROCEDURES FOR USE (continued)

position. When scale stops, use micrometer knob to superimpose scale pointer on next lower division. Read total weight from left to right observing decimal point.

6. Make all adjustments smoothly:
   Stop momentarily in "arrested" position before "full release."
   If pan swings too much, arrest once more.
7. Never dial weights in full-release position.
8. Clean chamber after use, using camel-hair brush and shut doors.
9. When not in use, the release lever should be in OFF position.

The procedures for using the triple beam balance are:

1. Place balance on level surface.
2. Clean scale surface.
3. Check zero balance, adjust using zero adjust knob under scale-head.
4. Place unknown on scale-head.
5. Adjust weights on triple arm until needle balances at zero.
6. Summate weight readings on all three scales to achieve total weight value.
7. Return weights to zero position.
8. Store balance where it won't be damaged.

3.0 QUALITY CONTROL METHODOLOGY

The analytical balance is verified to be level and zeroed prior to each use. The balance is located on a heavy shockproof table anchored into a concrete foundation. Balance temperature is equilibrated to room temperature. Special precautions are taken to avoid spillage of corrosive chemicals on the pan or inside the balance case. The balance is checked monthly against standard weights. A periodic calibration is performed with Sartorious.

The triple beam balance is leveled and zeroed before each usage and is stored in a safe location.

2.0 WATER MONITORING

WHITE MESA PROCEDURES MANUAL

MONITORING GUIDELINES

ENVIRONMENTAL

SURFACE WATER HYDROLOGY

1.0 EQUIPMENT

Equipment used for monitoring surface hydrology is:

1. Beckman Model 20 pH meter and probe;
2. YSI Model 33 temperature and conductivity meter and probe;
3. Linear polyethylene containers;
4. Sample Bucket;
5. 142mm diameter filter holder;
6. 0.45 micron membrane filters;
7. 0.8 micron glassfiber filters; and
8. Masterflex Peristalic Portable Pump.

2.0 MONITORING METHODOLOGY

2.1 Flow Measurement

No flow measurements are taken of the two drainage creeks: Westwater Canyon and Cottonwood Creek.

2.2 Water Quality

Westwater Canyon and Cottonwood Creek are monitored at two locations adjacent to the White Mesa Mill facility. Samples are obtained quarterly using grab sampling. A five gallon sample is obtained, following rinsing of the sample bucket several times in the monitored stream. A liter unfiltered aliquot is obtained for total suspended solids analysis. Specific conductivity, temperature and pH data is obtained in the field using procedures detailed in Section 2.4. The bucket contents are then well mixed as an aliquot is field pumped, by the peristaltic pump, through a filtration unit containing a 340 point five micron membrane filter, into new polyethylene containers for a soluble, or dissolved sample.

2.0 MONITORING METHODOLOGY (continued)

The filter(s) are retained in a sealed, labeled peto dish container for suspended radionuclide determinations.

The peristaltic pump tubing and filtration unit are rinsed with distilled water prior to being utilized at each site. The equipment is pre-rinsed with a known volume of the sampled stream prior to collection of the dissolved sample portion.

In the event that the sampled streams contain excessive turbidity for proper filtration with the 0.45 micron filters, the samples are pre-filtered using the 0.8 micron filters prior to re-filtering using the 0.45 micron filters; in either case, the filters are retained/composited for suspended radionuclide analysis.

Soluble aliquots are preserved using nitric acid to adjust the sample solution to a pH less than two and storage at four degrees Celsius.

Samples are submitted to the analytical laboratory on a quarterly basis for:

1. Total Suspended Solids;
2. Soluble: U-Natural, Thorium-230, Radium-226, Lead-210, Polonium-210; and,
3. Suspended: U-Natural, Thorium-230, Radium-226, Lead-210, Polonium-210.

Field data is obtained for:

1. Temperature, °C
2. Specific Conductivity, µmhos at 25°C;
3. pH, at 25°C;
4. Sample date;
5. Sampler ID; and,
6. Filtrate Volume.

2.0 MONITORING METHODOLOGY (continued)

Sample containers are labeled with:

1. Site ID:
2. Date;
3. Field Data;
4. Filtration Specs; and
5. Preservation Method.

3.0 RECORD KEEPING

3.1 Water Quality

Radiological and chemical quality data is maintained in the Hydrology-Stream notebook within the Environmental Office. Data is maintained in summary form listing:

1. Sample location,
2. Sample date,
3. Field pH,
4. Field temperature,
5. Field conductivity,
6. Total Suspended Solids concentration,
7. Soluble Radionuclide Concentrations*, and,
8. Suspended Radionuclide Concentrations*.

*U-Nat., Thorium-230, Radium-226, Lead-210, Polonium-210.

4.0 MONITORING LOCATION AND FREQUENCY

The monitoring locations are shown on the monitoring topographic map. They are listed as:

1. Lower Cottonwood Creek, and
2. Lower Westwater Creek.

Samples are obtained once per quarter; except when the creeks are dry.

5.0 ANALYSIS PROCEDURE

5.1 Water Quality

The laboratory analytical procedures utlized are presented in the procedures manuals of the appropriate laboratory.

Field procedures are discussed in subsection 2.0 of Section 2.1 and in Section 2.4.

Data is reviewed and graphed on a quarterly basis to observe abnormalities.

6.0 QUALITY ASSURANCE METHODOLOGY

6.1 Water Quality

Monitoring of pH, temperature, and conductivity procedures is done each quarter. Review of procedures involved with sample preservation is checked each quarter. Sample composition bottles are not re-used and sample contents are maintained in a locked cooler. All filtration equipment is rinsed with distilled water prior to each site use and rinsed with a known stream volume prior to obtaining a dissolved content aliquot for analysis.

On a semi-annual basis, one site sample is split in the field and submitted as a blind duplicate for analytical and sample collection quality assurance. Deviations greater than ten percent result in review of procedures.

WHITE MESA PROCEDURES MANUAL

MONITORING GUIDELINES

ENVIRONMENTAL

GROUNDWATER HYDROLOGY

1.0 EQUIPMENT

Monitor wells to be utilized for the groundwater monitoring system consist of narrow diameter PVC cased wells with locking surface caps.

Sampling equipment consists of:

1. 1.8 inch O.D. Air Driven Bennett Model 180-500 Sampling Pump;
2. 260 psi air compressor and ancillary equipment;
3. Beckman Model 20 pH meter and probe;
4. YSI Model 33 temperature and conductivity meter and probe;
5. Hand air vacuum pumps;
6. Peristaltic pumps; and
7. Field filtration and preservation equipment.

Measurement of phreatic elevations is by use of water level indicators:

1. Built into Bennett Model 180-500 pumping system;
2. Slope Indicator Co.
   Model 5143-200'
   Water Level Indicator; and,
3. SOILTEST, INC.
   Model DR-760A
   800 ft. capability

2.0 MONITORING METHODOLOGY

2.1 Phreatic Elevation

Phreatic elevations are monitored on a quarterly basis with a water level indicator meter. Field log books are maintained listing depth to water from top of casings. Readings are taken by lowering the sensor into the casing until the electrical sensor contact shows that a conductive liquid has been reached. Distance from the point where the electrical wire touches the top of the casing, at point of contact, to a known five-foot interval marker on the wire is determined using a tape measure. This distance plus the five-foot interval marking is total depth to water from top of casing. Data is recorded as phreatic elevation above sea level in the respective section in the Hydrology Data Notebook maintained in the Environmental Office or maintained on computer files.

There are no action levels associated with monitor wells. The water supply well is not equipped for phreatic measurements.

2.2 Groundwater quality is monitored on a quarterly basis by pumping a minimum of two casing volumes or until three successive samples show stabilized readings on pH, temperature, and conductivity.

When the well water has reached equilibrium, the flow is passed through a filtration unit utilizing a point-four-five micron membrane filter. The water is filtered through the unit into polyethylene jugs as the sample.

Field Sample Treatment Procedure is:

1. Rinse all field probes with distilled water.
2. Rinse new filter with distilled water.
3. Rinse sample containers with distilled water.
4. Rinse graduated cylinders.
5. Place new membrane and/or prefilter on glass or plastic frit at base of filtration unit.
6. Pump sample through filtration unit and into sample container after letting the first 100 mL be wasted as a filter rinse.

2.0 MONITORING METHODOLOGY (continued)

7. Rinse lid of sample container with filtered stream and tighten onto sample container.

8. Identify and label sample container; include:

   A. Site - well ID;

   B. Date;

   C. Temperature of original sample;

   D. pH of original sample;

   E. Conductivity of original sample;

   F. Preservation Method

9. Preserve the sample in the field using the appropriate chemical or physical preservation method.

10. If the sample is from the drinking water supply well, preserve the filter by placing it in a plastic petri container and seal the container. Label the container with the site I.D., Date, and volume filtered.

Upon return to the office, the samples are stored in a locked cooler at 4° C until transferred to the Analytical Laboratory.

Field data is transcribed onto computer data worksheets and are held in this form until the analytical laboratory data becomes available. When such data is available, the data is then entered onto the work sheets and typed into the computer record. The worksheets and analytical sheets are filed as reference material.

The following is the analytical data to be requested on the well samples.

CHEMICAL PARAMETER MONITORING SCHEDULE
OPERATIONAL PHASE GROUNDWATER PROGRAM

Quarterly Sampling

Indicator Parameters:

pH (F,L)
Specific Conductance (F,L)
Temperature (F)
Sulfate (L)
Chloride (L)
U-Natural (L)
Thorium-230 (L)
Radium-226 (L)
Pb-210 (L)
Po-210 (L)

Accuracy Assessment Parameters*

Calcium (L)
Magnesium (L)
Potassium (L)
Bicarbonate/Carbonate (F,L)
Sodium (L)
Total Dissolved Solids (L)

Annual Sampling**

pH (F,L)
Specific Conductance (F,L)
Temperature (F)
Total Dissolved Solids (L)
Alkalinity (F,L)
Bicarbonate/Carbonate (L)
Sulfate (L)
Chloride (L)
Ammonia (L)
Phosphate (L)
Aluminum (L)
Arsenic (L)
Cadmium (L)
Calcium (L)
Chromium (L)
Copper (L)
Lead (L)
Magnesium (L)
Manganese (L)
Mercury (L)
Molybdenum (L)
Potassium (L)
Selenium (L)
Sodium (L)
Vanadium (L)
Zinc (L)
Gross Alpha (L)
Gross Beta (L)
Uranium-Nat. (L)
Radium-226 (L)
Thorium-230 (L)
Lead-210 (L)
Polonium-210 (L)

Note: F = Parameter measured in field
L = Parameter measured in laboratory
All radionuclides and metals are analyzed for dissolved concentrations only.

* First and Third Quarters
** First Quarter

Heavy metal and radionuclide samples are preserved by filtration, adjustment of pH to less than two with nitric acid, and storage at 4°C. Ammonia samples are preserved by filtration, adjustment of pH to less than two with sulfuric acid, and storage at 4°C. Other samples are preserved at 4°C.

2.0 MONITORING METHODOLOGY (continued)

Samples are cataloged at White Mesa and sent to the Analytical Laboratory with the appropriate Certificate of Analysis sheets attached. Samples are transmitted by locked and sealed containers by postal contract services. Copies of Certificate of Analysis sheets are returned to White Mesa showing date of receipt by the Laboratory.

Bottles utilized are of linear polyethylene and consist per sample site of:

1. Two gallons - Radionuclides;
2. Two liters - metals;
3. One liter -- ammonia; and,
4. One liter -- T.D.S., CL, SO_4, Ca, CO_3, HCO_3, K, Alkalinity, and Na.

3.0 CALIBRATION

3.1 Phreatic Measurements

Equipment used in phreatic measurements are checked for calibration quarterly.

3.2 Water Quality

Probe and meter equipment including temperature, pH, and conductivity are calibrated at each use. Calibration logs are not maintained.

4.0 RECORD KEEPING

Summarized data is logged onto computer file work sheets. Once all data for a well or quarter is completed, the data from the worksheet is typed into a computer file. Certificates of Analysis and computer work sheets are then maintained as a backup file with the main working file being on the computer system.

Data includes well, I.D., sample date, well phreatic elevations, field analytical data, and all assay data.

5.0 MONITORING LOCATION AND FREQUENCY

Monitor wells are monitored quarterly for phreatic elevation, radionuclide and chemical quality. The active monitor wells are identified as: 1, 2, 3, 4, 5, 11, 12, 13, and the culinary water well. Refer to the environmental monitoring topographic map for the well locations.

6.0 ANALYSIS PROCEDURE

The laboratory analytical procedures utilized are as presented in the Analytical Procedure Manual.

Field data analytical procedures are as per Section 2.4 of this manual.

7.0 QUALITY ASSURANCE METHODOLOGY

7.1 Well Contamination

Well contamination, from external surface factors, is controlled by installation of a cap over the surface casing and cementing off the surface section of the drill hole. Wells have surface casing of mild steel with a lockable cap cover. Environmental Staff have the sole access to the key series locking the wells.

Subsurface well stagnation, for pumped wells, is reduced by pumping the wells until temperature, pH, and conductivity levels of water samples become consistent, verifying that aquifer zone water is being drawn into the well and sampled.

7.2 Phreatic Elevations

Monitoring of phreatic elevations is controlled by usage of a field log book so that the technician can refer to previous field data in case of an abnormal reading while at the site. Also, the field log book serves as a check for numerical transposition of calculated elevations.

7.3 Water Quality

Quality assurance of water quality in field sample analysis, handling, and preservation is maintained by following procedures as described in Sections 2.2 and 2.4 which comply with EPA and Umetco Minerals standards. Observation of technician performance is monitored on a periodic basis to ensure compliance with standards. Blind duplicates of at least one well per quarter are submitted to the analytical laboratory to monitor field and laboratory quality assurance performance. Deviations exceeding ten percent result in analytical and field procedure review.

WHITE MESA PROCEDURES MANUAL

MONITORING GUIDELINES

ENVIRONMENTAL MONITORING

CULINARY SUPPLY

1.0 EQUIPMENT

Equipment for monitoring of domestic water quality and usage consists of flow totalizer meter, an Beckman Model 20 pH meter, chlorine comparator, and fecal coliform bottles supplied by the State of Utah.

Zero point four five micron Gelman membrane filters are used for filtration.

2.0 MONITORING METHODOLOGY

2.1 Quantity

Quantity of domestic water usage is maintained by the Environmental Staff. Currently, there is an in-line flow rate meter located at the domestic water supply wellhead. The flowrate meter consists of a digital readout with two rotating needles reading the last two numerical digits. The larger dial reads tens of gallons, the smaller dial reads units of gallons. The last digit on the digital readout is hundreds of gallons.

Readings are taken the last day of each week and recorded in the Domestic Water record notebook. Values are used by the State Engineer's Office as part of an annual survey.

2.2 Quality

Domestic raw water is grab sampled daily by the environmental crew who transmit the sample to the Environmental Department.

Bacteriological quality is checked by submittal of proper sampled fecal coliform sample to the Utah Department of Health Branch Laboratory on a monthly basis or as required.

Chlorine content is checked during weekdays using chlorine comparator. The pH is monitored weekly.

Compliance with Utah's Drinking Water Regulations is monitored as per E.P.A. guidelines and for elements determined for ground water sources.

3.0 CALIBRATION

Calibration of equipment consists solely of calibrating the Beckman Model 20 probe. This is accomplished by checking probe readout versus buffered solutions of pH one, seven, and ten. The equipment is adjusted to reflect the calibration solution pH as described in the Beckman pH equipment manual and in section 2.4 of this manual.

4.0 RECORD KEEPING

Records are maintained in a Domestic Water Data Notebook covering data summary and is maintained in the Environmental Office at White Mesa.

Recorded data, in summary form, includes:

1. Sample Period;
2. Sample Type;
3. Weekly Consumption;
4. Fecal Coliform, Count; and
5. pH Range.

5.0 MONITORING LOCATION AND FREQUENCY

Samples for water chemical quality are taken daily. Fecal coliform sampling occurs once per month, as per Utah Public Drinking Water Regulations. Sample taps for the mill are rotated monthly. The daily grab sample is checked each Thursday for pH level.

Water quantity readings are obtained weekly at the wellhead by the Environmental staff.

Analysis of pH values is by direct readout from a Beckman Model 20 pH meter.

The analytical procedures utilized are as presented in Section 2.4. Results are reported in final concentration format to White Mesa.

7.0 QUALITY ASSURANCE METHODOLOGY

7.1 Bacteriological Monitoring

Bacteriological monitoring consists solely of fecal coliform checks through usage of Utah Department of Health Laboratories and sample containers. Sampling is done strictly by standard methods.

7.0 QUALITY ASSURANCE METHODOLOGY (continued)

Avoidance of sample jar overtopping, proper flushing of a non-aerated sample tap, labeling of bottles, identification documentation, and transportation processing are actions taken as quality control.

7.2 Quantity Monitoring

Observation of flow rate meter operation is routinely done weekly to ensure proper meter operation. Maintenance or replacement of meters is accomplished with Environmental Staff appraisal.

7.3 Chemical Quality Monitoring

analysis immediately follows sample collection. Residual chlorine concentrations are determined using chlorine comparator.

WHITE MESA PROCEDURES MANUAL
MONITORING GUIDELINES
ENVIRONMENTAL

TEMPERATURE AND CONDUCTIVITY MEASUREMENTS

1.0 EQUIPMENT

Equipment used is:

1. Yellow Springs Model 33
   S-C-T Meter with ten foot probe system

   This model is a portable unit which is operated both by line power and by battery mode. The probe is protected by a plastic enclosure for field usage.

2.0 PROCEDURES FOR USE

The procedures for using the temperature and conductivity meter are:

1. Ensure probe is connected to meter.
2. Switch mode control to off. Use meter screw to adjust meter needle to 0, if necessary.
3. Switch mode to red line. Use red line control to adjust meter needle to red line.
4. TEMPERATURE: Switch mode to temperature; read meter when needle is steady.
5. CONDUCTIVITY: Read temperature; set °C control to indicated temperature. Switch mode to appropriate conductivity scale for on-scale meter readings. Read meter and multiply reading by scale.
6. Replace batteries when Step 3 cannot be accomplished. Use Eveready E95 (or equivalent) alkaline battery.
7. Clean probe tip in distilled water prior to use.
8. Allow needle to stabilize prior to reading values.
9. When reading conductivity when temperature adjusted as per above, conductivity reading is mhos/cm at 25°C.

3.0 QUALITY CONTROL METHODOLOGY

The temperature sensor is periodically checked versus a NBS traceable thermometer. The conductivity meter cell constant is checked monthly versus meter reading values using standard potassium chloride reference solutions.

The solutions are:

Solution	Normality	Method of Preparation	Temp.(^{o}C)	Conductivity (μmhos)
A	0.1	7.4365 g KCL/1 at 20 ^{o}C	0 18 25	7,138 11,167 12,856
B	0.01	0.7440 g KCL/1 at 20 ^{o}C	0 18 25	773 1,220 1,408
C	0.001	Dilute 100 mL of B to 1 liter at 20^{o}C	25	147

Calculate the cell constant as follows:

$$L = \frac{K_1 + K_2}{1,000,000 + K_X}$$

Where:

L = Cell constant
K_1 = Conductiviity, in mhos/cm, of KCL solution at the temperature of measurement
K_2 = Conductivity, in mhos, at the same temperature, of the distilled water used to prepare the reference solution.
K_X = Measured conductance, in mhos.

Temperature readings should not vary from thermometer values. Conductivity readings should be within two percent. Standard solutions can be cross-checked with the White Mesa Analytical Laboratory. Meter conductivity minimum value determinable is ten mhos/cm.

Distilled water conductivity should be less than 10 micromhos/cm.

WHITE MESA PROCEDURES MANUAL

MONITORING GUIDELINES
ENVIRONMENTAL

pH METERS

1.0 EQUIPMENT

Equipment used in the White Mesa Environmental Lab and in the field to monitor pH is:

1. Beckman Model 20 pH meter;
2. Beckman Gel Filled Combination Electrode; Futura II;
3. Beckman pH 1.0 buffer solution, chloride buffer;
4. Beckman pH 7.0 buffer solution, phosphate buffer;
5. B.A.R. pH 10.0 buffer solution, borate buffer:
6. pH combination electrode filling solution: 2M KCL, Sat. AgCL;
7. Magnetic stir rods and stirrer - lab usage;
8. 250 mL glass beakers - lab usage;
9. 250 mL plastic beakers - field usage; and,
10. Deionized distilled water.

2.0 pH ANALYSIS

Procedures for pH analysis are as follows:

1. Field - Perform battery check on Model 20 meter;
2. Inspect pH probe for cracks.
3. Put probe in buffer solution of 7 pH. Buffer solution is placed from supply bottle into beaker. Allow meter to standardize to pH of 7.

2.0 pH ANALYSIS (continued)

Field -- Buffer solution can be swirled using hand motion. Response may take time. Cold temperature of air will affect meter electronics. Keep meter warm. If slope correction falls below 95 percent, relocate meter to a warmer environment.

4. Rinse probe with distilled water and dry with a clean tissue.
5. Put probe in another solution containing either buffer solution of one or ten--depending upon estimated sample pH value. Response time varies.
6. Allow meter to standardize to buffer solution.
7. Rinse probe with distilled water and dry.
8. Put probe in sample stream, let meter stabilize, and read pH level, nearest 0.1 pH. Meter compensates for temperature of solution.
9. Remove probe, rinse with distilled water and dry between samples.
10. Do not put used buffer solution back into supply bottle; discard.

3.0 QUALITY ASSURANCE

The following of the above analysis procedure and usage of standard equipment will give accurate and repeatable results in pH analysis. Technician performance is periodically reviewed by supervisor.

3.0 TAILING MONITORING

WHITE MESA PROCEDURES MANUAL

MONITORING GUIDELINES

ENVIRONMENTAL

TAILING STABILITY INSPECTION PROGRAM

1.0 SUMMARY

The on-site inspection program is a systematic procedure for inspecting and recording observations of the performance of all aspects of the tailings disposal system, including geotechnical, structural, hydraulic, and electrical-mechanical features of the operations. Items of inspection and procedures are divided into groups according to inspection frequency as follows:

1. Daily
2. Weekly
3. Monthly
4. Quarterly
5. Yearly

Changes in inspection frequency will be made as needed, based on observations made during these scheduled inspections. Inspections will also be made after the occurrence of any unusual or severe natural or man-caused events. Each of the inspection item groups is described below.

2.0 DAILY INSPECTION

On a daily basis, all areas connected with active tailings disposal will be patrolled and observations of the current condition and behavior of the disposal system will be made and recorded. This involves visual inspection of the following:

1. Tailings slurry transport system (from the mill to Cell 2 or Cell 3)
2. Tailings Cell 2
3. Tailings Cell 3
4. Evaporation Cell 1-I
5. Dike structures including Dike 1-I, 1A-I, 2, and 3
6. Cell 2 sump and drain structure and associated drain system
7. Cell 3 sump and drain structure and associated drain system.

2.0 DAILY INSPECTION (continued)

The inspection checklist presented in Table 1A will be used as a guide to what conditions are of potential concern, and also to provide a place to record observations of potential concern. For all inspection items, the inspector is required to record whether that item appears to be performing properly (O.K. column of checklist is checked) or that conditions of potential concern have been observed with that item (observations are recorded and the action required column of checklist is checked). If conditions of potential concern are observed, then some further action is required by the inspector. The specific action required is indicated in Table 1B. Table 1B indicates that one of three levels of action may be required based on three types of observations:

Key No.	Observation Type	Required Action
1	Emergency Indicator	Notify plant superintendent, environmental coordinator, and engineer
2	Warning Indicator	Record all data, notify environmental coordinator immediately
3	Clues to Distress	Record all data, notify environmental coordinator at earliest convenience.

In addition to the completion of the daily inspection checklist and concurrently with the daily inspection, certain items of operating data will be recorded by inspection personnel. These items will be recorded on a daily or weekly basis as noted below, unless large changes are noted during the daily visual inspection. These items are listed in the data form presented in Table 2, and include:

1. Pond elevations in Cell 1-I, Cell 2, and Cell 3 (record weekly)
2. Location of slurry discharge within the active cell (record daily)
3. Flow and line pressure for the active tailings slurry line (record daily)
4. Flow in the active cell sump and drain line (record weekly)
5. Liquid level in underdrain risers (record weekly).

These data provide a record of performance against which future observations can be evaluated. Record photographs should be made periodically of all operating facilities for reference when questions about observations and previous conditions arise.

3.0 MONTHLY INSPECTION

Inspection of additional items in the tailings disposal system will be made on a monthly basis. These include:

1. Quantitative evaluation of the active cell tailings slurry pipeline condition.
2. Inspection of diversion and retention structures for surface water runoff.

Results of the inspection will be recorded in Table 3.

Slurry Pipeline

The slurry pipeline will be inspected at certain key locations to determine pipe wear. An ultrasonic device will be used to measure pipe thicknesses and determine the need for replacement pipe and pipe fittings. The critical locations which must be checked include bends, slope changes, valves, and junctions which are critical with respect to the stability of the dike. The exact locations to be checked will be established by the environmental coordinator, depending on the location and configuration of the slurry pipeline at the time of inspection.

Surface Water Control Structures

Diversion Ditches 1 and 2 will be inspected along their entire lengths for alteration of the channel cross-section and characteristics, and for removal of debris. The Mill and Facilities Area Sedimentation Pond will be inspected for volume of storage and general condition. The condition of these structures will be recorded in Table 3.

4.0 QUARTERLY INSPECTION

Items requiring quarterly inspection and/or measurement include:

1. Inspection of Cells 1-I, 2, and 3 emergency spillways.
2. Review of maintenance and operational procedures.
3. Inspection for Post-Construction Changes outside of immediate disposal area.

All items inspected on a monthly basis will be included in the quarterly inspection. The results of the quarterly inspection will be recorded in Table 4.

The emergency spillways for Cell 1-I, 2, and 3 will be examined to detect any changes in the structures. The Safety Dike will be inspected in the

4.0 QUARTERLY INSPECTION (continued)

same manner during the quarterly inspection as the active cell dikes are inspected on a daily basis, using the checklist of Table 1A.

Maintenance and operational procedures will be reviewed for any needed changes, based on observations over the last quarter. General inspection of areas outside the immediate disposal area will be made to determine any changes in site conditions not due to tailings disposal operations.

A summary of observations and recommended actions (if any) for each of the above quarterly inspection items will be prepared for the records by inspection personnel.

5.0 YEARLY INSPECTION

Yearly inspection involves survey measurement of Dikes 1-I, 1A-I, 2, and 3 configuration.

The dike crest and slope alignment and elevations will be surveyed to detect movements which are not observable by visual inspection. Survey measurements will be made at selected sections of Dikes 1-I, 1A-I, 2, and 3. At each section, measurements will be made at the crest, slope midpoint, and toe of the downstream side of the dike (where possible). The location and number of sections to be surveyed will be designated by the environmental coordinator.

Results of the survey work will be submitted to the environmental coordinator for review and placed in the inspection record files.

6.0 TECHNICAL EVALUATION AND INSPECTION REPORT

A technical evaluation of inspection observations will be performed on an annual basis, or at times when it is needed due to significant observations of concern. The detailed scope of the evaluation is difficult to define and will depend on the nature of the observations since the last evaluation. The evaluation will address any changes in the system or items of concern observed during system performance. A report will be prepared which lists these changes and/or significant observations and states the effect on these items on continued operations. The report will also contain a summary of the inspection observations for the time period since the last report. The summary will simply consist of a collection of the data sheets used in the weekly, monthly, and quarterly inspections.

7.0 Freeboard Limits

The freeboard limits are 3.5 feet below crest on Pond One and 5.0 feet below crests on Ponds 2 and 3.

TABLE 1.A

UMETCO MINERALS CORPORATION
WHITE MESA

DAILY INSPECTION DATA

Date:____________________

Inspector:____________________

	Inspection Items	Condition of Potential Concern	O.K.	Observations of Potential Concern	Action Required
DIKE 2	- Slopes	Sloughs or Sliding, Cracks, Bulges, Subsidence, Severe Erosion, Moist Areas, Areas of Seepage Outbreak			
	- Crest	Cracks, Subsidence, Severe Erosion			
DIKE 3	- Slopes	Sloughs or Sliding, Cracks, Bulges, Subsidence, Severe Erosion, Moist Areas, Areas of Seepage Outbreak			
	- Crest	Cracks, Subsidence, Severe Erosion			
CELL 2 DRAIN SYSTEM	- Drain Access and Sump Barge	Sump Blockage, Damage			
	- Pipeline to Cell 1-I	Leaks, Blocks, Damage			

Note: If conditions of potential concern are observed, action is required. See Table entitled "Daily Inspection Checklist."

TABLE 1.A

UMETCO MINERALS CORPORATION
WHITE MESA

DAILY INSPECTION DATA (continued)

Date: ____________________
Inspector: ____________________

	Inspection Items	Condition of Potential Concern	O.K.	Observations of Potential Concern	Action Required
EVAPORATION CELL 1-I	- Water Level	Greater than Operating Level, Large Change Since Previous Inspection			
	- Liner and Cover	Erosion of Cover, Exposure of Liner			
DIKE 1-I	- Slopes	Sloughs or Sliding, Cracks, Bulges, Subsidence, Severe Erosion, Moist Areas, Areas of Seepage Outbreak			
	- Crest	Cracks, Subsidence, Severe Erosion			
DIKE 1A-I	- Slopes	Sloughs or Sliding, Cracks, Bulges, Subsidence, Severe Erosion, Moist Areas, Areas of Seepage Outbreak			
	- Crest	Cracks, Subsidence, Severe Erosion			
Active Cell Slurry Pipeline		Flow Rate _______ gpm			
Active Cell Slurry Pipeline		Pressures: Point 1 ________ psig Point 2 ________ psig Point 3 ________ psig			
Active Cell Slurry Pipeline		Discharge Point			

TABLE 1.B
UMETCO MINERALS CORPORATION
WHITE MESA
DAILY INSPECTION CHECKLIST

	OBSERVATION OF POTENTIAL CONCERN INSPECTION ITEM	Pipeline Break	Leaks	Damage	Sharp Bends	Blockage	Loose Connections	Loss of Support	Improver Location	Too High, Large Changes	Large Cracks	Severe Erosion	Subsidence	Erosion of Cover	Exposed Liner	Bulging	Large Sloughs or Sliding	Moist Area	Seepage Breakout
Cell 2 Tailings Slurry Transport System	Pipeline at Dike	1	2	3	2	1	3	2	3										
	Pipeline at Other Locations	2	2	3	3	1	3	2	3										
	Pipeline Joints	-	2	3	3	1	3	-	-										
	Pipeline Supports	-	-	3	-	-	-	-	3										
	Valves	-	2	3	-	1	-	-	-										
	Point of Discharge	-	-	3	-	1	-	-	2										
Tailings Cell 2	Pond Level									2	-	-	-	-	-				
	Beach										3	3	3	-	-				
	Cell Edge										2	3	2	-	-				
	Liner and Cover										2	3	2	3	3				
Dikes 1-I, 1A-I, 2, 3	Upstream Slope										1	3	2			2	1	-	-
	Downstream Slope										1	3	2			2	1	2	2
	Crest										1	3	2			2	1	-	-
Cell 2 Drain System	Sump		2		2														
	Pipeline	2	2		2														

Key No.	Type of Observation	
1	Emergency Indicator	Notify Plant Superintendent and environmental coordinator immediately.
2	Warning Indicator	Record all data, notify environmental coordinator immediately
3	Distress Indicator	Record all data, notify environmental coordinator at earliest convenience.

TABLE 2.0

UMETCO MINERALS CORPORATION
WHITE MESA
TAILING SYSTEM

WEEKLY INSPECTION REPORT

		Week of	Week of	Week of	Week of	Week of
Pond Elevations	Cell 1					
(0.1 feet elev.)	Cell 2					
	Cell 3					
Mean Slurry Flow (gpm)						
Mean Return Sump Flo gpm)						
Mean Drain Flow (gpm)						
Underdrain phreatic Levels (Ft.)	Cell 1					
	Cell 2					
	Cell 3					

REMARKS ____

____

____

____

____

____

TABLE 3.0

UMETCO MINERALS CORPORATION
WHITE MESA

MONTHLY INSPECTION DATA

Date: __________
Inspector: __________

1. SLURRY PIPELINE

Equipment Used: __________
Equipment Operator: __________
Location Inspected: __________
General Pipeline Condition: __________
Results (thickness, etc.): __________
Action Required: __________
Location Inspected: __________
General Pipeline Condition: __________
Results (thickness, etc.): __________
Action Required: __________

(If more than two pipeline areas are inspected, use additional forms).

2. SURFACE WATER CONTROL STRUCTURES:

Diversion Ditch 1 Condition: __________

Diversion Ditch 2 Condition: __________

Diversion Ditch 3 Condition: __________

Mill and Facilities Area Sedimentation Pond Conditions: __________

Remarks: __________

TABLE 4.0

UMETCO MINERALS CORPORATION

WHITE MESA

QUARTERLY INSPECTION DATA

Date: ____________________
Inspector: ____________________

1. Emergency Spillways:

Cell 1-I Condition: ______________________________

Cell 2 Condition: ______________________________

Cell 3 Condition: ______________________________

Action Required: ______________________________

2. General Area Inspection:

Condition/Observations: ______________________________

Action Required: ______________________________

REMARKS: ______________________________

WHITE MESA PROCEDURES MANUAL

Monitoring Guideline
Environmental

LINER INTEGRITY

The onsite tailing monitoring program for liner integrity consists of monitoring underdrain pipeline collection seepage and visual observation of liner protection from solar deterioration.

During operating periods, the sump and drain structure is monitored daily by assigned inspection personnel. On a weekly basis, underdrains are observed to determine presence of liquids. If liquids are present, the underdrain is pumped or bailed, to determine volume, and an analysis of the liquid contents is completed. The samples are analyzed for:

1. U-Natural, total;
2. Vanadium. total;
3. Molybdenum, total;
4. Sulfates, total;
5. Chlorides, total; and,
6. pH.

Surface access points to the underdrains will remain capped between checks to minimize precipitation entrapment.

Analytical data is reviewed to determine if seepage is due to direct migration of tailing liquor.

The inspection personnel consists of the Environmental Coordinator, Radiation Safety Officer, or their designees. Tailing stability monitoring and analysis is the responsibility of the Environmental Coordinator. Adherence to the referenced document: "Tailing Management System; Monitoring Plan", February 1982, D'Appolonia, Energy Fuels Nuclear, Inc. will be maintained.

4.0 MISCELLANEOUS

WHITE MESA PROCEDURES MANUAL

MONITORING GUIDELINES

ENVIRONMENTAL

EXTERNAL GAMMA, SURFACE SOIL, AND VEGETATION

1.0 EQUIPMENT

Monitoring equipment for surface soils and vegetation consists of a shovel, tape measure, trowels, and collection bags.

Monitoring of external gamma levels is by use of Eberline spherical container TLD chips, one set of five chips per container.

2.0 MONITORING METHODOLOGY

2.1 Surface Soils

Surface soils are sampled at the five air monitoring sites. Surface soil samples consist of samples removed by trowel to a depth of one centimeter, from an undisturbed area, within a surface area of 1.25 square feet. Total sample volume is 0.041 cubic feet. The technique utilized is to remove surface organic material covering the site, remove soil to a depth of one centimeter using a trowel for the entire described surface area, and directly placing the excavated soil into the plastic bag. An identification tag, containing sample location, sample type identification, date, and technician initials is then fastened around the bag closure sealing the contents.

Soil samples are then transmitted to the analytical laboratory along with the appropriate Certificate of Analysis sheet for the following analysis on a dry basis:

1. Natural Uranium; and,
2. Radium-226;

Results are reported as micro Curies per gram.

2.0 MONITORING METHODOLOGY (continued)

2.2 Vegetation

Forage vegetables are sampled per growing season at three locations, northeast, northwest, and southwest of the mill, where such vegetation may exist.

Vegetation is clipped at edible or ground plane elevations and itemized as to species and location sampled. Samples are enclosed in plastic bags for storage and transportation for analysis. Edible portions are defined as that portion normally consumed by the consumer. Weights of vegetation are obtained prior to shipment to the laboratory. The wet basis weight, location, species, date sampled, and technician initials are included on the bag closure tag.

Vegetation is analyzed for:

1. Radium-226; and,
2. Lead-210.

Results are reported as micro Curies per kilogram on wet basis.

2.3 External Gamma

External gamma is monitored at the ambient air sampling sites using the spherical container TLD badges of Eberline. The containers, each containing five TLD chips, are mounted one meter above ground plane at each site with two containers per site. The containers loaded with TLD's are received the first of each quarter from Eberline and exchanged with those in the field. A background TLD is stored in a lead-lined container as a transportation control. The TLD's are returned to Eberline for processing with a report returned within three weeks of transmittal.

Values reported are in millirem per week average for the monitor period along with a counting error term.

3.0 CALIBRATION

There are no calibration requirements associated with soil, vegetation or external gamma measurements as required to be done at the site.

4.0 RECORD KEEPING

4.1 Surface Soils

Surface soil record keeping consists of:

1. Soil type, (clay, sand, silt, gravel);

4.0 RECORD KEEPING (continued)

2. Date sampled;
3. Location sampled;
4. Technician initials; and,
5. Radionuclide analysis.

Records are maintained in the 40 CFR 190 data notebook maintained at the Environmental Office.

4.2 Vegetation

Data maintained in record form for vegetation analysis includes:

1. Sample date;
2. Species identification;
3. Sample location;
4. Sample weight, wet basis;
5. Technician initials; and,
6. Radionuclide analysis.

Data is maintained in 40 CFR 190 notebook in the Environmental Office.

4.3 External Gamma

Data maintained as a record summary for external gamma is:

1. Sample period;
2. Sample location; and,
3. External gamma levels for total radiation.

Data is maintained on computer files.

5.0 MONITORING LOCATION AND FREQUENCY

5.1 Surface Soils

Surface soil sampling occurs during March of each year at the ambient air sampling sites (BHV-1 to BHV-5).

5.0 MONITORING LOCATION AND FREQUENCY (continued)

5.2 Vegetation

Vegetation samples are obtained during each growing season and are divided between forage and direct human food consumption species. Forage species are collected at or near the following sites:

1. BHV-1 - near meteorological station;
2. BHV-1 - west (1/2 mile) of meteorological station; and,
3. BHV-4 - ambient particulate monitoring station (southwest of mill);

5.3 External Gamma

External gamma measurements are taken over a quarterly interval for the twelve months of the year at all ambient air particulate monitoring sites.

6.0 ANALYSIS PROCEDURE

The laboratory analytical procedures utilized are as presented in the appropriate Laboratory Analytical Procedure Manual except for external gamma which is done by Eberline, Inc.

7.0 QUALITY ASSURANCE METHODOLOGY

7.1 Surface Soils

Surface soil samples are obtained from a known surface area by using a standard template. Maintaining one centimeter depth is by tape measure or other suitable graduated measure. Identification tags are installed in the field labeling each sample with tags secured to the sample container. A field log notebook is also maintained. Samples are stored in locked rooms or containers with access only by environmental personnel. Samples are submitted to the analytical laboratory in sealed, locked containers accompanied by Certificate of Analysis sheets.

7.2 Vegetation

Vegetation samples are stored in plastic bags and weighed upon return to the laboratory. Only edible portions are sampled. Samples are identified and tagged in the field and stored in locked containers prior to transhipment to locked, sealed containers to the analytical laboratory. Certificate of Analysis sheets include wet basis weights and accompany samples to the laboratory. Sample sizes are at least two kilograms each.

7.0 QUALITY ASSURANCE METHODOLOGY (continued)

7.3 External Gamma

Quality assurance for external gamma measuremerts consist of:

1. Monitoring the containers once per week to document that TLD's are still there;
2. Checking the containers to insure Eberline enclosed chips;
3. Changing containers one by one to insure same numbered container is at same site;
4. Insuring all containers are present when receiving or shipping to Eberline; and,
5. Review of Eberline data for consistency and data transposition.

5.0 ADMINISTRATIVE CONTROL - ALARA

WHITE MESA PROCEDURES MANUAL

MONITORING GUIDELINES
ENVIRONMENTAL

RADIOLOGICAL
AUDIT PROGRAM

1.0 FREQUENCY

An ALARA audit of the environmental monitoring program is conducted on a semi-annual basis. The audit is conducted with the presence of the Plant Superintendent, Environmental Coordinator, corporate environmental personnel and/or corporate health and safety personnel.

2.0 PURPOSE

The purpose of the audit is to administer the environmental monitoring program to maintain conditions as low as reasonably achievable through administrative familiarization and reaction to observed environmental conditions. A detailed audit report is submitted to the Operations Manager-Uranium with a copy forwarded to the Nuclear Regulatory Commission.

3.0 REPORT

The audit committee reviews and documents in a semi-annual report the following items:

1. Documented inspection reports,
2. Environmental radiological effluent and monitoring data, including Quality Assurance data, and
3. Reviews of operating and monitoring procedures completed during this time period.

The audit report specifically addresses:

1. Trends in environmental radiological effluent and monitoring data.
2. Performance of effluent control equipment
3. Administrative controls/policies on ALARA management of retention systems and releases.