EPA/450/2-82/003

Protection Agency

Air Pollution Training Institute
MD 20
Environmental Research Center
Research Triangle Park, NC 27711

EPA 450/2-82-003
July, 1982

Air

APTI Correspondence Course 414 Quality Assurance for Source Emission Measurement Methods

Guidebook

Course Developed By:
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Under Contract No.
68-02-3573
EPA Project Officer
R. E. Townsend

United States Environmental Protection Agency
Office of Air, Noise, and Radiation
Office of Air Quality Planning and Standards
Research Triangle Park, NC 27711

EPA/450/2-82/003

Notice

This is not an official policy and standards document. The opinions and selections are those of the authors and not necessarily those of the Environmental Protection Agency. Every attempt has been made to represent the present state of the art as well as subject areas still under evaluation. Any mention of products or organizations does not constitute endorsement by the United States Environmental Protection Agency.

Availability

This document is issued by the Manpower and Technical Information Branch, Control Programs Development Division, Office of Air Quality Planning and Standards, USEPA. It was developed for use in training courses presented by the EPA Air Pollution Training Institute and others receiving contractual or grant support from the Institute. Other organizations are welcome to use the document.

This publication is available, free of charge, to schools or governmental air pollution control agencies intending to conduct a training course on the subject covered. Submit a written request to the Air Pollution Training Institute, USEPA, MD 20, Research Triangle Park, NC 27711.

Others may obtain copies, for a fee, from the National Technical Information Service (NTIS), 5825 Port Royal Road, Springfield, VA 22161.

Table of Contents

Course Introduction

Overview of Course

Course Description

This training course is a 35-hour correspondence course dealing with quality assurance procedures for EPA manual source measurement Methods 1 through 8. The course reviews, in detail, essentials of equipment calibration, proper testing methods, proper use of standardized testing forms, and EPA data tolerances. Course topics include:

- quality assurance principles
- procurement of apparatus and supplies
- calibration of apparatus
- presampling operations
- on-site measurement methods
- postsampling operations
- equipment maintenance methods
- auditing procedures

During 1980 and 1981 a series of workshops on quality assurance for source measurements was presented by the EPA Manpower and Technical Information Branch in collaboration with the EPA Quality Assurance Division and the EPA Division of Stationary Source Enforcement. Over 250 individuals, including agency personnel and source testing contractors, attended the workshops held at Research Triangle Park, Dallas, Denver, and San Francisco. This correspondence course is an outgrowth of these workshops. It has been designed to provide you with training similar to that experienced by the workshop attendees.

The course is intended for those who have had training or experience in source sampling methods. It is not designed to give the theory of source sampling methods or teach how they are done. APTI Courses 450 and 468 are intended for that purpose. This course is intended to help refine the technique of a testing organization so that quality data can be assured.

The following documents will be used as texts:

- EPA 600/4-77-0276 *Quality Assurance Handbook for Air Pollution Measurement Systems. Volume III—Source Measurements*
- EPA 600/9-76-005 *Quality Assurance Handbook for Air Pollution Measurement Systems. Volume I—Principles* (selected sections)

Course Goal

This course is designed to familiarize you with quality assurance guidelines prescribed in Volume III of the EPA *Quality Assurance Handbook for Air Pollution Measurement Systems*. It is intended to assist you in applying quality control methods in the performance of Federal source testing methods.

Course Objectives

Upon completion of this course, you should be able to:

1. explain why quality assurance procedures are a vital part of the Environmental Protection Agency's air monitoring programs.
2. initiate the development of a source test team quality assurance program.
3. list at least eight elements of an effective quality assurance program.
4. define: quality assurance, quality control, representative samples, chain of custody, collaborative test, audit.
5. use the procurement sections of Volume III and associated activity matrices in obtaining the proper equipment for a source sampling testing team.
6. employ the guidelines of Volume III in calibrating source testing equipment.
7. use the checklists provided in Volume III in preparing sampling equipment before a source test.
8. list at least five procedures which should be routinely followed in order to maintain source testing equipment.
9. use the data and calculation forms provided in Volume III.
10. describe how audit samples can be used in a quality assurance program designed for a source test team.
11. list at least three functions of a test auditor and point out at least two procedures that the auditor should specifically observe during the performance of each of the EPA reference methods for particulate matter, SO_2, NO_x, and O_2/CO_2.

Sequence, Lesson Titles, and Trainee Involvement Time

Requirements for Successful Completion of this Course

In order to receive 3.5 Continuing Education Units (CEUs) and a certificate of course completion you must:

- take two supervised quizzes and a supervised final examination.
- achieve a final course grade of at least 70 (out of 100) determined as follows:
  - Quiz 1 is 20% of the final grade.
  - Quiz 2 is 20% of the final grade.
  - The final examination is 60% of the final grade.

Use of Course Materials

The Materials You'll Need

- APTI Correspondence Course 414 *Quality Assurance for Source Emissions—Guidebook*
- *Quality Assurance Handbook for Air Pollution Measurement Systems. Volume III—Source Measurements* EPA 600/4-77-0276 (with updates to January 15, 1980)

How to Use this Guidebook

Relationship Between Guidebook and Assigned Reading Materials

This guidebook directs your progress through Volume III of the *Quality Assurance Handbook for Air Pollution Measurement Systems.* Excerpts from Volume I are included in the guidebook to provide you with a basic introduction to quality assurance principles. If you wish to obtain the complete text of Volume I, it may be ordered from the Quality Assurance Division of EPA in Research Triangle Park, NC.

If you use the guidebook instructions with the provided reading material, we think you will find the subject material both interesting and enjoyable. Review exercises and problems focus on specific and important aspects of the quality assurance manuals. Although not all aspects of the QA guidelines are covered by the review exercises and quizzes, after you complete the course you will be able to effectively use QA Volume III.

Description of Guidebook Sections

This guidebook contains ten lessons separated into four units. Each lesson contains the following:

- lesson learning goal and objectives
- reading assignment(s)
- reading guidance
- review exercises
- answers to review exercises

Complete the review exercises immediately after reading the assigned materials. You may find it helpful to look over the review questions before reading. By having an idea of what to look for in the reading materials, your attention will be better focused and your study will be more efficiently directed.

NOTE: If more than one person will be using these materials, we recommend that you use a separate sheet of paper to record your answers to the review exercises.

Instructions for Completing the Quizzes and the Final Examination

- You should have received, along with this guidebook, a separate sealed envelope containing two quizzes and a final examination.
- You must arrange to have someone serve as your test supervisor.
- You must give the ***sealed*** envelope containing the quizzes and final examination to your test supervisor.
- At designated times during the course, under the supervision of your test supervisor, complete the quizzes and the final exam.
- After you have completed a quiz or the exam, your test supervisor must sign a statement on the quiz/exam answer sheet certifying that the quiz or exam was administered in accordance with the specified test instructions.
- After signing the quiz/exam answer sheet, your test supervisor must mail the ***quiz or exam and its answer sheet*** to the following address:

  Air Pollution Training Institute
  Environmental Research Center
  MD-17
  Research Triangle Park, NC 27711

- After completing a quiz, continue with the course. Do ***not*** wait for quiz results.
- Quiz/exam and course grade results will be mailed to you.

If you have questions, contact:

Air Pollution Training Institute
Environmental Research Center
MD-17
Research Triangle Park, NC 27711

Telephone numbers:
Commercial: (919) 541-
FTS: 629-

20

30

2401

2402

Quality Assurance Principles

Lesson A

EPA Quality Assurance Policy and Volume I Review

Lesson Goal

The goal of this lesson is to familiarize you with EPA's quality assurance policies and its approach to implementing the use of quality assurance plans for manual source sampling programs.

Lesson Objectives

After completing this lesson, you should be able to:

1. explain why quality assurance procedures are a vital part of the Environmental Protection Agency's air monitoring programs.
2. initiate the development of a quality assurance program for source test teams.
3. list at least eight elements of an effective quality assurance program.
4. define: quality assurance, quality control, accuracy, precision, quality assurance plan, quality assurance manual, performance audit, system audit.
5. describe the importance of quality assurance programs to an organization.
6. recognize the contribution of training programs to the reporting of high quality data.
7. list at least four items that should be included in a quality assurance report to management.
8. distinguish between a quality assurance manual and a quality assurance plan.

Materials

Assignment 1

- Memorandum—May 30, 1979, Douglas Costle
- Strategy for the Implementation of EPA's Mandatory Quality Assurance Program
- Memorandum—June 14, 1979, Douglas Costle
- Memorandum—November 2, 1981, Anne Gorsuch

Assignment 2

General information (contained in this guidebook) on the topic of quality assurance. This material has been extracted from Volume I of the QA Handbook series.

Reading Guidance—Assignment 1

This lesson reviews the Environmental Protection Agency's quality assurance policies for air pollution data. Reading Assignment 1 includes memos giving the rationale for the development of quality assurance programs, and Reading Assignment 2 provides guidance for their development.

The Quality Assurance Handbook aids agencies, contracting firms, and industries that want to initiate quality assurance programs and plans within their organizations. This correspondence course is intended to help make using the handbook easier.

Begin by reading pages 13 through 18 of this guidebook.

Attachment B

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D C 20460

THE ADMINISTRATOR

May 30, 1979

MEMORANDUM

TO: Deputy Administrator
Director, Science Advisory Board
Director, Office of Regional and Intergovernmental Operations
Regional Administrators
Assistant Administrators
General Counsel

SUBJECT: Environmental Protection Agency (EPA) Quality Assurance Policy Statement

The EPA must have a comprehensive quality assurance effort to provide for the generation, storage, and use of environmental data which are of known quality. Reliable data must be available to answer questions concerning environmental quality and pollution abatement and control measures. This can be done only through rigorous adherence to established quality assurance techniques and practices. Therefore, I am making participation in the quality assurance effort mandatory for all EPA supported or required monitoring activities.

An Agency quality assurance policy statement is attached which gives general descriptions of program responsibilities and basic management requirements. For the purpose of this policy statement, monitoring is defined as all environmentally related measurements which are funded by the EPA or which generate data mandated by the EPA.

A detailed implementation plan for a total Agency quality assurance program is being developed for issuance at a later date. A Select Committee for Monitoring, chaired by Dr. Richard Dowd, is coordinating this effort, and he will be contacting you directly for your participation and support. I know that each of you shares my concern about the need to improve our monitoring programs and data; therefore, I know that you will take the necessary actions that will ensure the success of this effort.

Douglas M. Costle

Attachment

Strategy for the Implementation
of the
Environmental Protection Agency's
Mandatory Quality Assurance (QA) Program

I. Introduction

The EPA must have a comprehensive QA program to provide for the generation, storage, and use of environmental data. Valid data of verifiable quality must be available to provide a sound basis for effective decisions concerning environmental quality, pollution abatement, and control measures. The QA program can succeed only through rigorous adherence to established QA techniques and practices.

In the past, there has been a high degree of fragmentation, lack of coordination, poorly identified needs and resources, and duplication of efforts in the QA program. For these reasons, it is now Agency policy, as enunciated by the Administrator in memoranda of May 30, 1979 and June 14, 1979, that all Regional Offices, Program Offices, EPA Laboratories, and those monitoring and measurement efforts supported or mandated through contracts, regulations, or other formalized agreements participate in a centrally managed QA program. Regional Offices should work cooperatively with States to assist them in developing and implementing QA programs.

The mandatory QA program covers all environmentally-related measurements. Environmentally-related measurements are defined as "essentially all field and laboratory investigations that generate data involving the measurement of chemical, physical, or biological parameters in the environment; determining the presence or absence of pollutants in waste streams; health and ecological effect studies; clinical and epidemiological investigations; engineering and process evaluations;

studies involving laboratory simulation of environmental events; and studies or measurements on pollutant transport, including diffusion models.

This document presents the strategy for the development of an Agency QA program in accordance with the Agency policy. This strategy describes, in general, the total program effort with respect to what must be done. This strategy does not attempt to describe how, in detail, the program is to be implemented within the individual Program and Regional Offices, or the EPA Laboratories. Subsequent guidance documents will enable the Program and Regional Offices and the EPA Laboratories to develop detailed QA plans.

II. Quality Assurance Goals and Objectives

The primary goal of the QA program is to insure that all environmentally-related measurements supported or required by the EPA result in data of known quality. To meet this goal, the QA program must provide for the establishment and use of reliable monitoring and measurement systems to obtain data of necessary quality to meet planned Agency needs.

Initial objectives are the development and implementation of QA program plans by each of the Program and Regional Offices and EPA Laboratories which will ensure that the QA goal can be achieved nationally.

Long-term objectives include (1) providing quantitative estimates of the quality of all data supported or required by the Agency, (2) improving data quality where indicated, and (3) documenting progress in achieving data quality.

A continuing objective is to promote and develop optimally uniform approaches,

procedures, techniques, reporting methods, etc., across media and across Regional Offices, Program Offices, and EPA Laboratories. It is important (and most efficient and effective) for all organizations within EPA to employ the same QA language, consistent policies, procedures, and techniques when interacting with the States, industry, the public, contractors, grantees, QA-involved professional societies, other Governmental agencies, and national and international organizations.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON D C 20460

Attachment C

THE ADMINISTRATOR

June 14, 1979

MEMORANDUM

SUBJECT: Quality Assurance Requirements for all EPA Extramural Projects Involving Environmental Measurements

FOR: The Deputy Administrator
Assistant Administrators
Regional Administrators
General Counsel

Over the past several years, the EPA has become more and more dependent on extramural projects to provide the environmental measurements we use as a foundation for our standards, regulations and decisions. While in most instances these projects are providing data of proven quality that is acceptable for the Agency's purposes, there have been, regrettably, some instances of Agency funds paid for poor quality, unusable data.

In order to assure that all environmental measurements done by extramural funding result in usable data of known quality, I am making the inclusion of the attached "Quality Assurance Requirements" mandatory for all EPA grants, contracts, cooperative agreements, and interagency agreements that involve environmental measurements. In addition to these general requirements, I expect every Project Officer to include whatever additional specific quality assurance requirements are necessary in each extramural project under his control. Criteria and guidelines in this area will be forthcoming from the Agency's Quality Assurance Implementation Work Group. Further, I direct the Assistant Administrator for Planning and Management to provide the appropriate contract and grant regulations such that the attached form "Quality Assurance Review for Extramural Projects Involving Environmental Measurements" will be satisfactorily completed where appropriate prior to the approval of any contracts or grants in FY-80.

I recognize that this may increase the cost per environmental measurement, but the benefits of a credible Agency data base that provides a level of quality that meets the needs of users far outweigh any such increases.

Douglas M. Costle

Attachment

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON D C 20460

THE ADMINISTRATOR

November 2, 1981

MEMORANDUM

TO: Associate Administrators
Assistant Administrators
Regional Administrators

SUBJECT: Mandatory Quality Assurance Program

One of the major concerns of this administration and myself is that we support all of our actions and decisions with statistically representative and scientifically valid measurement of environmental quality. To meet this objective, it is essential that each of you continue to support and implement the Agency's mandatory Quality Assurance program which is being implemented by the Office of Research and Development. It is especially essential that you assure that the appropriate data quality requirements are included in all of your extramural and intramural environmental monitoring activities. I also am particularly concerned that you do not sacrifice quality for quantity when adjusting your program to meet our new resource targets.

The attached Second Annual Quality Assurance Report demonstrates the importance of this program in achieving our goals and objectives. Recognizing its importance, I have asked Dr. Hernandez to closely monitor this program's implementation and advise me of any problems that affect the scientific data bases of the Agency.

Anne M. Gorsuch

Attachment

cc: Deputy Administrator
Office Directors

You have completed your reading for Assignment 1. Do the review exercises which follow and check your answers after you complete them. The correct answers are given on the page following the review exercises.

Reading Assignment 1 Review Exercises

1. Valid and verifiable source-test data is needed by regulatory agencies:
   a. so that source testing costs can be increased.
   b. to provide a sound basis for regulatory decisions.
   c. to derive theoretical models for combustion processes.
   d. to ensure that data follow a log-normal distribution.

2. Which one of the follow is **not** a long-term objective of EPA's mandatory QA program?
   a. to provide quantitative estimates of data quality
   b. to document progress in improving the quality of environmental measurements reported to the agency
   c. to improve data quality
   d. to increase the amount of data reported to the agency

3. EPA's mandatory QA program affects:
   a. EPA laboratories.
   b. organizations receiving EPA grants or contracts.
   c. organizations with cooperative agreements with EPA.
   d. all of the above

4. Volume III of the *Quality Assurance Handbook for Air Pollution Measurement Systems* is important to EPA's mandatory QA program since it:
   a. promotes the use of uniform procedures for source testing.
   b. requires all testing contractors to use the same data reporting forms.
   c. is the only available handbook on source testing methods.
   d. promotes the use of uniform procedures for continuous source emissions monitoring.

5. True or False? EPA's mandatory QA program is applicable only to air pollution measurements.

6. The SST source test team reported a Method 5 compliance test result of 0.063582 gr/dscf ± 0.0316 gr/dscf for a subpart D fossil-fuel-fired steam generator. Would this report be consistent with EPA's mandatory QA program?
   Yes _____
   No _____
   If no, why not?

Answers to
Reading Assignment 1 Review Exercises

1. a (b) c d
2. a b c (d)
3. a b c (d)
4. (a) b c d
5. T (F)
6. No, since the number of significant figures reported is inconsistent with the accuracy of the reference method itself. The figures are also not reported in the units specified in the method.

Reading Guidance—Assignment 2

This assignment reviews many of the concepts involved in the subject of quality assurance and gives directions for setting up a quality assurance program within an organization. Portions of Volume I of the EPA Quality Assurance Handbook are included in this section. You may obtain the complete handbook by writing to:

Quality Assurance Division
Environmental Monitoring and Support Laboratory
US Environmental Protection Agency
Research Triangle Park, NC 27711

and requesting: Environmental Protection Agency (EPA). 1976. *Quality Assurance Handbook for Air Pollution Measurement Systems. Volume I—Principles* EPA 600/9-76-005.

In-depth training in the principles discussed in Volume I can be obtained by attending EPA APTI Course 470 *Quality Assurance for Air Pollution Measurement Systems.* This four-day lecture course is designed for quality assurance coordinators or managers involved with quality assurance activities.

Volume I of the Quality Assurance Handbook focuses on 23 elements of the *quality assurance wheel.* Eight of these elements are considered essential when setting up a new program. The discussion given in Volume I for each of these eight elements is reproduced here for your review.

> Begin by reading the excerpts from Volume I. They are on pages 25 through 82 in this guidebook.
>
> The following comments introduce you to the reading assignment. Read them along with the excerpts; they will help familiarize you with some of the main ideas of quality assurance, and will make the reading assignment easier to understand.

1. Definition of quality assurance
   - Be sure to note the distinction between *quality control* and *quality assurance.*
   - What is the *product* with which EPA quality assurance programs are conceived?

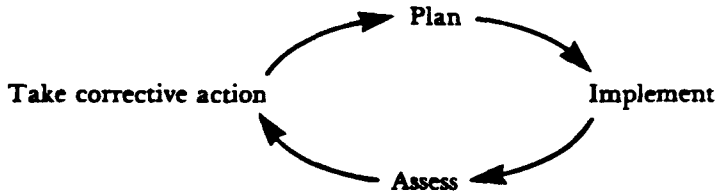
2. Elements of quality assurance
   - The elements have been placed on the wheel in such a way that the quality assurance coordinator can select those applicable to his or her particular program.
   - The eight elements reviewed here should be considered from the very beginning in the development of a program. They are as follows:
     (1) document control and revisions
     (2) policy and objectives
     (3) organization
     (4) training
     (5) audit procedures
     (6) quality reports to management
     (7) quality assurance manual
     (8) quality assurance plans for projects and programs
   - The 23 quality assurance elements can be grouped into four general categories:
     (1) management activities
     (2) measurement activities
     (3) routine systems for program operation and support
     (4) statistical techniques

3. Document control and revisions
   - Note the indexing format of the Volume I excerpts included here. Also note the indexing format in Volume III. The format provides a means of tracking and updating entries in QA manuals and plans.
   - The purpose of document control is to provide the latest procedures to all concerned.
   - The last page of this lesson is an outline of Volume III. We will discuss this in depth later in this course. The manner in which Volume III is documented is very important for its use.

4. Quality assurance policy and objectives
   - The QA policy of an organization must have the support of upper-level management if the QA program is to be effective. Note the memos reproduced in Reading Assignment 1.
   - When reading the discussion of this element, note that data objectives should be specified for:
     completeness
     precision
     accuracy
     representativeness
     comparability

     However, cost should be kept in mind.

5. Organization
   - Quality assurance is normally a separate function. The separation helps prevent bias and provides easier access to upper-level management.
   - A QA coordinator should be appointed and his function spelled out in a position description.
6. Training
   - You cannot produce high quality data with people who do not know how to do their jobs. It is the responsibility of management to see that the job is done right.
7. Audit procedures
   - Audit procedures should be implemented as quickly as possible when setting up a QA program.
   - Audits are one of the best ways of checking the quality of data.
8. Quality reports to management
   - Feedback is very important for managers. Anything that affects management decisions should be included in the report.
   - Reports should be understood at a glance. When possible, use charts and graphs to present data, rather than tables.
9. The quality assurance manual
   - Note that a quality assurance manual is general. It covers all of the QA programs and methods used by an organization.
10. The quality assurance plan
    - Note that the quality assurance plan is method specific. It gives the specific QA requirements for a sampling procedure.
    - In this sense, the EPA QA Volume I provides guidance for the development of an organization's QA *manual*. Volume III provides guidance for the development of source sampling QA *plans* for EPA Reference Methods 1 through 8.
11. Final note: Although not included in the excerpts here, it is important to mention the *QA cycle*. The cycle is shown below.



The effect of applying quality assurance techniques should result in the development of an on-going corrective action system. Once a QA plan is written and implemented, assessment procedures point out necessary corrective action which, in effect, revises the plan. The cycle continues in this manner, providing quality source sampling data.

EPA-600/9-76-005
January 1976

QUALITY ASSURANCE HANDBOOK FOR AIR POLLUTION MEASUREMENT SYSTEMS

Volume I — Principles

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Environmental Monitoring and Support Laboratory
Research Triangle Park, North Carolina 27711

1.3 DEFINITION OF QUALITY ASSURANCE[1-4]

Quality assurance and quality control have been defined and interpreted in many ways. The more authoritative usages differentiate between the two terms by stating that quality control is "the system of activities to provide a quality product," whereas quality assurance is "the system of activities to provide assurance that the quality control system is performing adequately." In other words, quality assurance is quality control for quality control.

Quality control may also be understood as "internal quality control;" namely, routine checks included in normal internal procedures; e.g., periodic calibrations, duplicate checks, split samples, and spiked samples. Quality assurance may also be viewed as "external quality control," those activities that are performed on a more occasional basis, usually by a person outside of the normal routine operations; e.g., on-site system surveys, independent performance audits, interlaboratory comparisons, and periodic evaluation of internal quality control data.

In this Handbook, the term quality assurance is used to include the above meanings of both quality assurance and quality control.

While the objective of EPA's air programs is to improve the quality of the air, the objective of quality assurance for air programs is to improve or assure the quality of measured data on pollutant concentrations. Thus the "product"

with which quality assurance is concerned is data.

Since air pollution measurements are made by numerous agencies and private organizations at a large number of field stations and laboratories, quality assurance is also concerned with establishing and assessing comparability of data quality among organizations contributing to data bases.

1.3.1 REFERENCES

1. Juran, J.M. Quality Control Handbook, 3rd Ed. McGraw-Hill, 1974. Section 2.

2. ASTM. Designation E-36, "Standard Recommended Practice for Generic Criteria for Use in the Evaluation of Testing and/or Inspection Agencies."

3. ASQC. Standard A3-1971 (ANSI Standard Z1.7-1971), "Glossary of General Terms Used in Quality Control."

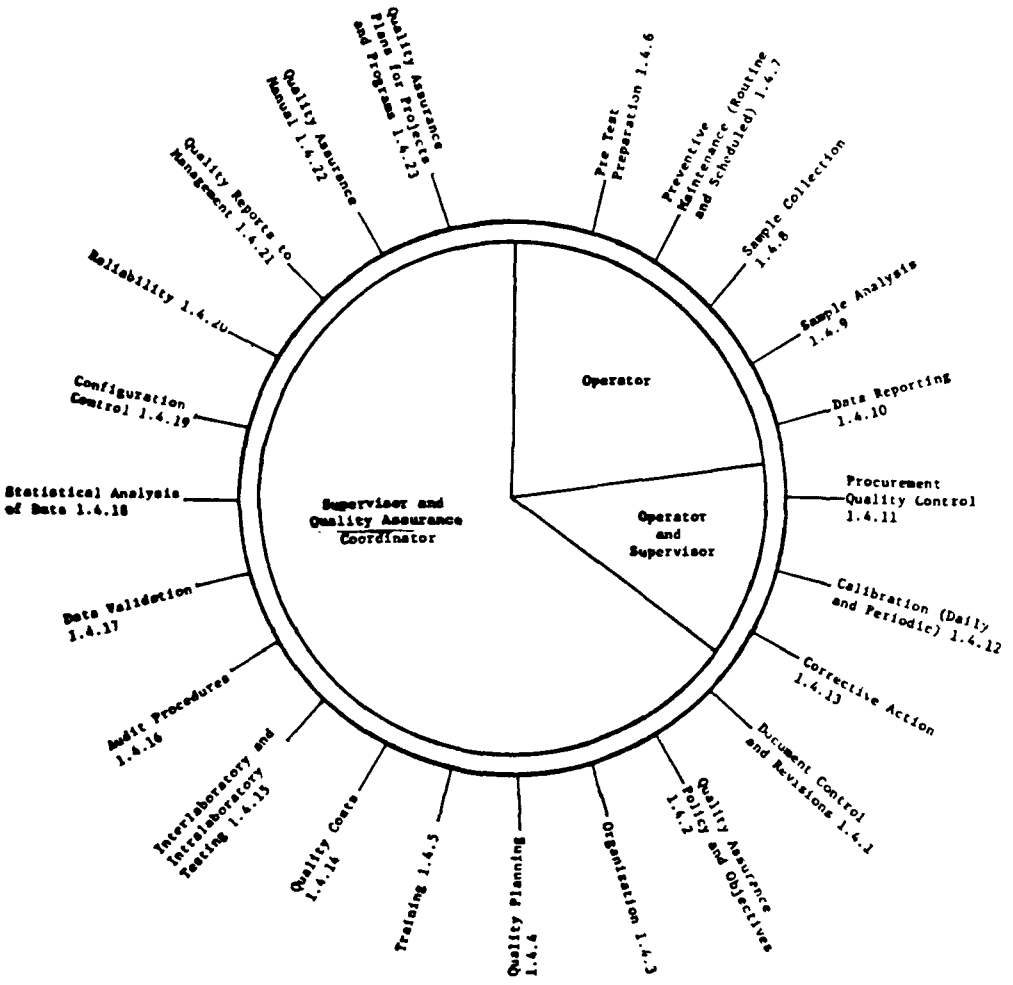
4. Feigenbaum, A.V. Total Quality Control, Engineering and Management. McGraw-Hill, 1961.

1.4 ELEMENTS OF QUALITY ASSURANCE

A quality assurance program for air pollution measurement systems should cover a number of areas or elements. These elements are shown in Figure 1.4.1 in a "Quality Assurance Wheel." The wheel arrangement illustrates the need for a quality assurance system that will address all elements and at the same time will allow program managers the flexibility to emphasize those elements that are most applicable to their particular program. Quality assurance elements are grouped on the wheel according to the organization level to which responsibility is normally assigned. These organizational levels are the quality assurance coordinator (normally a staff function), supervisor (a line function), and the operator. Together the supervisor and quality assurance coordinator must see that all these elements form a complete and integrated system and are working to achieve the desired program objectives.

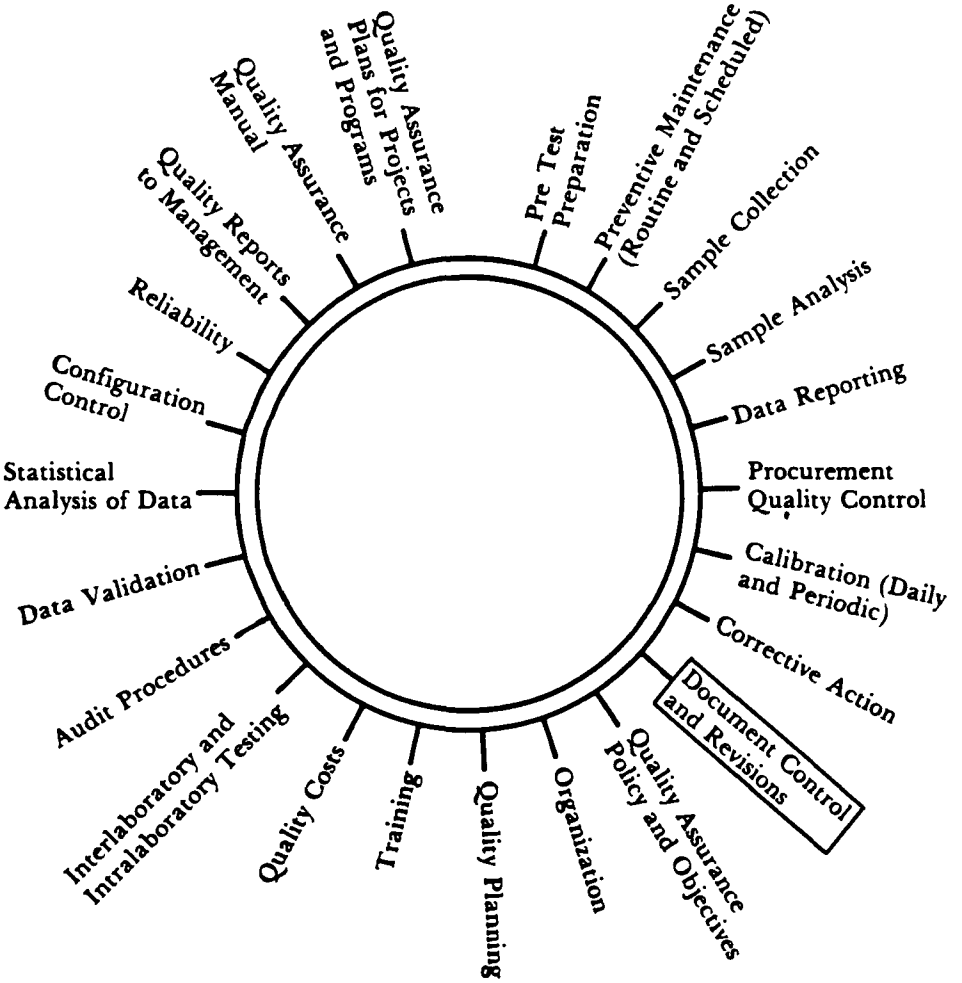
The three-digit numbers shown on the wheel show the location in Section 1.4 where a description of the element is provided. Each element is described in three subsections as follows:

1. ABSTRACT - A brief summary that allows the program manager to review the section at a glance.
2. DISCUSSION - Detailed text that expands on items summarized in the ABSTRACT.


Operator
Operator and Supervisor
Supervisor and Quality Assurance Coordinator
Pre Test Preparation 1.4.6
Preventive Maintenance (Routine and Scheduled) 1.4.7
Sample Collection 1.4.8
Sample Analysis 1.4.9
Data Reporting 1.4.10
Procurement Quality Control 1.4.11
Calibration (Daily and Periodic) 1.4.12
Corrective Action 1.4.13
Document Control and Revisions 1.4.1
Quality Assurance Policy and Objectives 1.4.2
Organization 1.4.3
Quality Planning 1.4.4
Training 1.4.5
Quality Costs 1.4.14
Interlaboratory and Intralaboratory Testing 1.4.15
Audit Procedures 1.4.16
Data Validation 1.4.17
Statistical Analysis of Data 1.4.18
Configuration Control 1.4.19
Reliability 1.4.20
Quality Reports to Management 1.4.21
Quality Assurance Manual 1.4.22
Quality Assurance Plans for Projects and Programs 1.4.23

3. REFERENCES - List of resource documents used in preparation of the discussion. In addition, where applicable, a list of resource documents for recommended reading is shown under BIBLIOGRAPHY.

The DISCUSSION subsection is designed to be relatively brief. In those cases where a topic would require considerable detailed discussion, the reader is referred to the APPENDIX.


Quality Assurance Plans for Projects and Programs
Quality Assurance Manual
Quality Reports to Management
Reliability
Configuration Control
Statistical Analysis of Data
Data Validation
Audit Procedures
Interlaboratory and Intralaboratory Testing
Quality Costs
Training
Quality Planning
Organization
Quality Assurance Policy and Objectives
Document Control and Revisions
Corrective Action
Calibration (Daily and Periodic)
Procurement Quality Control
Data Reporting
Sample Analysis
Sample Collection
Preventive Maintenance (Routine and Scheduled)
Pre Test Preparation

1.4.1 DOCUMENT CONTROL AND REVISIONS

1.4.1.1 ABSTRACT

A quality assurance program should include a system for documenting operating procedures and revisions in the procedures. The system used for this Handbook is described and is recommended.

1.4.1.2 DISCUSSION

A quality assurance program should include a system for updating formal documentation of operating procedures. The suggested system is the one used in this Handbook and described herein. This system uses a standardized indexing format and provides for convenient replacement of pages that may be changed within the technical procedure descriptions.

The indexing format includes, at the top of each page, the following information:

Section No.
Revision No.
Date (of revision)
Page

A digital numbering system identifies sections within the text. The "Section No." at the top of each page identifies major three-digit or two-digit sections, where applicable. For example, Section 1.4.4 represents "Quality Planning" and

Section 1.4.5 represents "Training." "Revision No." represents the most current version of the section in question, where the first version is represented as "0." "Date" represents the date of the latest revision. "Page No." includes not only the number of the specific page, but also the total number of pages in this section. An example of the page label for the first page of "Quality Planning" in Section 1.4.4 follows:

Section No. 1.4.4
Revision No. 0
Date May 1, 1975
Page 1 of 6

For each three-digit level, the text begins on a new page. This format groups the pages together to allow convenient revision of the section. Each time a new page is added or expanded within a section, the number of the preceding or original page is included on the new page, and a letter is added to it. For example, if Page 4 of 8 were revised and expanded to include an extra paragraph, the overflow would appear on a page designated 4a. The original Page 4 would then be removed from the Handbook and replaced by revised Page 4 and Page 4a. This allows expansion within a section without retyping the section or renumbering all of the pages.

The Table of Contents follows the same structure as the text. It contains a space for "Revision No." and "Pages"

within each section heading. When a revision to the text is made, the Table of Contents page would be updated by re-typing, or by striking out the old revision number and printing the current revision number. For example, the Table of Contents page detailing Section 1.4 might appear as follows:

		Pages	Revision	Date
1.4.1	Document Control and Revisions	5	0	5/1/75
1.4.2	Quality Assurance Policy and Objectives	5	0	5/1/75
1.4.3	Organization	7	0	5/1/75

A revision to "Organization" would change the Table of Contents to appear as follows:

		Pages	Revision	Date
1.4.1	Document Control and Revisions	5	0	5/1/75
1.4.2	Quality Assurance Policy and Objectives	5	0	5/1/75
1.4.3	Organization	7	1	7/1/75

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The document control system described herein applies to this Handbook and it can be used, with minor revisions, to maintain control of quality assurance procedures developed by users of this Handbook and quality assurance coordinators. The most important elements of the quality assurance program to which document control should be applied include:

1. Sampling procedures.
2. Calibration procedures.
3. Analytical procedures.

4. Data collection and reporting procedures.

5. Auditing procedures.

6. Sample shipping and storage procedures.

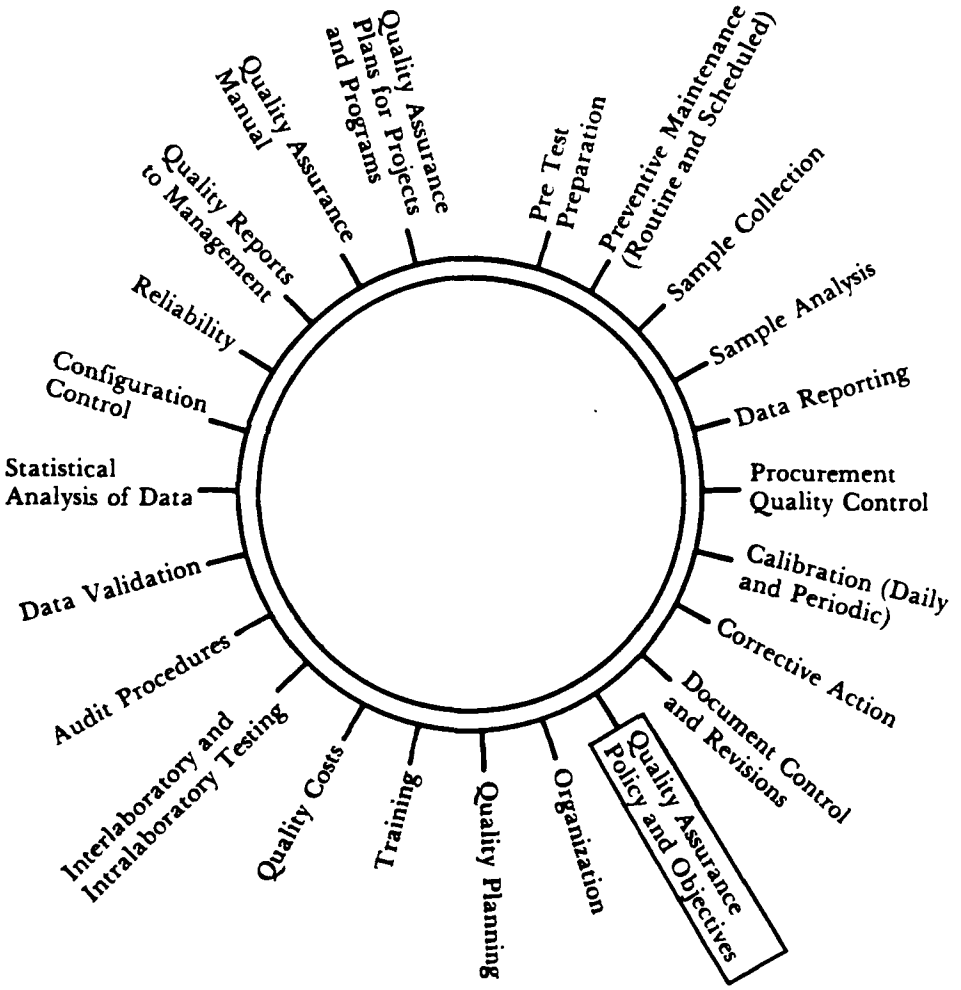
7. Computational and data validation procedures.

8. Quality assurance manuals.

9. Quality assurance plans.

1.4.1.3 REFERENCE

1. Industrial Hygiene Service Laboratory Quality Control Manual. DHEW, PHS, National Institute of Occupational Safety and Health, Cincinnati, Ohio. Technical Report No. 78, 1974. Section VII.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1.4.2 QUALITY ASSURANCE POLICY AND OBJECTIVES

1.4.2.1 ABSTRACT

1. Each organization should have a written quality assurance policy that should be made known to all organization personnel.

2. The objectives of quality assurance are to produce data that meet users' requirements measured in terms of completeness, precision, accuracy, representativeness and comparability and at the same time reduce quality costs.

1.4.2.2 DISCUSSION

Quality assurance policy - Each organization should have a written quality assurance policy. This policy should be distributed so that all organization personnel know the policy and scope of coverage.

Quality assurance objectives[1,2] - To administer a quality assurance program, the objectives of the program must be defined, documented, and issued to all activities that affect the quality of the data. Such written objectives are needed because they:

1. Unify the thinking of those concerned with quality assurance.

2. Stimulate effective action.

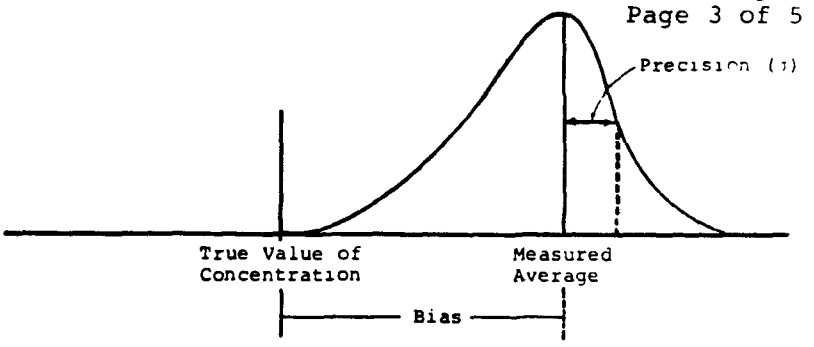
3. Are a necessary prerequisite to an integrated, planned course of action.

4. Permit comparison of completed performances against stated objectives.

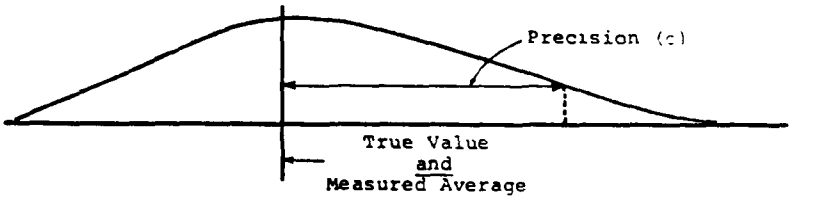
The objectives of a quality assurance system as described in this Handbook are to produce data that are complete, precise, accurate, representative, and comparable.

Data can be considered to be complete if a prescribed percentage of the total possible measurements is present. Precision and accuracy represent measures of the data quality. Data must be representative of the condition being measured. Ambient air sampling at midnight is not representative of carbon monoxide levels during rush hour traffic. Stationary source emission measurements are not representative if measured at reduced load production conditions when normal operation is at full load. Data available from numerous agencies and private organizations should be in consistent units and should be corrected to the same standard conditions of temperature and pressure to allow comparability of data among groups.

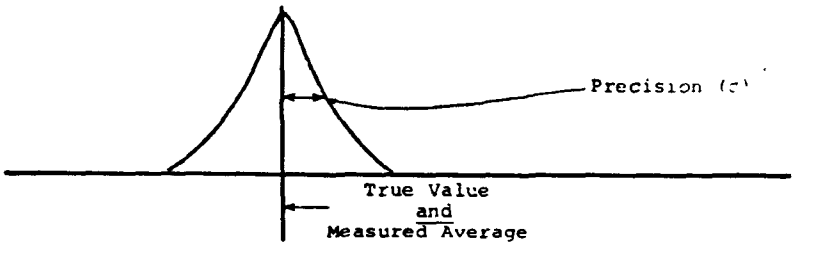
Figure 1.4.2.1 shows three examples of data quality with varying degrees of precision and accuracy. These examples hypothesize a true value that would result if an accurate measurement procedure were available and an infinitely large number of measurements could be made under specified conditions. If the average value coincides with the true value (reference standard), then the measurements are accurate. If the measurement values also are closely clustered about the true value, the measurements are both



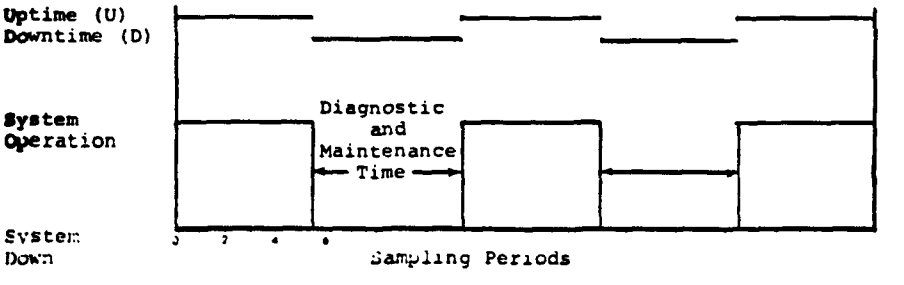
Example of Positive Biassed (Inaccurate) but Precise Measurements



Example of Accurate (No Bias) but Imprecise Measurements



Example of Precise and Accurate Measurements



Example Indicating a Measure of Completeness of Data U/(D + U)

Figure 1.4.2.1 Examples of data with varying degrees of precision, accuracy, and completeness

precise and accurate. Figure 1.4.2.1 also shows an example of completeness of data.

Each laboratory should have quantitative objectives set forth for each monitoring system in terms of completeness, precision, and accuracy of data. An example is included below for continuous measurement of carbon monoxide (non-dispersive infrared spectrometry) to illustrate the point.

1. Completeness - For continuous measurements, 75 percent or more of the total possible number of observations must be present. A summary of a minimum number of observations by time intervals (i.e., yearly, monthly, and hourly) is shown in Figure 1.4.17.1 (Data Validation).[3]

2. Precision - Determined with calibration gases, precision is $\pm$0.5 percent full scale in the 0 through 58 mg/m^3 range.[4]

3. Accuracy - Depends on instrument linearity and the absolute concentrations of the calibration gases. An accuracy of $\pm$1 percent of full scale in the 0 through 58 mg/m^3 range can be obtained.[4]

For further discussion of completeness, precision, accuracy and comparability, see the following:

1. Completeness and comparability, Section 1.4.17.

2. Precision and accuracy, Appendix G.

Employment of the elements of quality assurance discussed in Section 1.4. should lead to the production of data that are complete, accurate, precise, and comparable.

1.4.2.3 REFERENCES

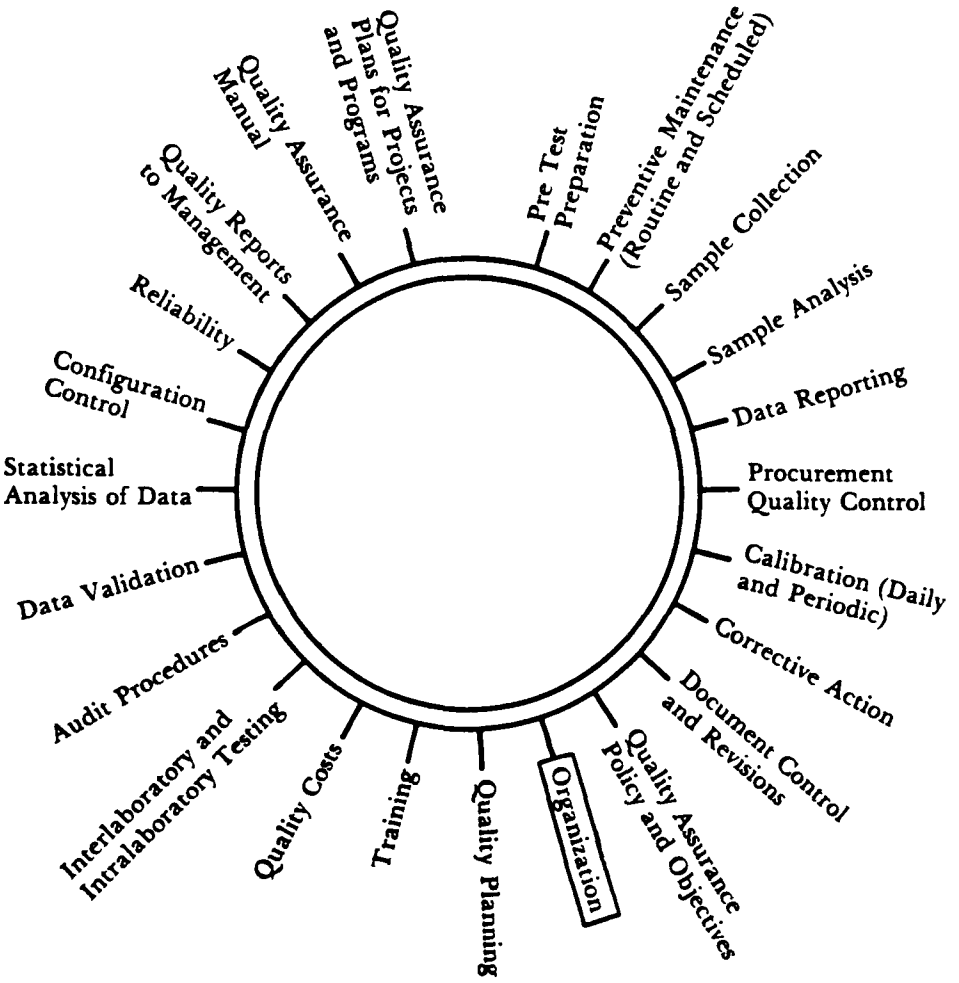
1. Juran, J.M., (ed.). Quality Control Handbook. 2nd Ed. McGraw-Hill, New York, 1962. Sec. 2, pp. 4-8.

2. Feigenbaum, A.V. Total Quality Control. McGraw-Hill, New York, 1961. pp. 20-21.

3. Juran, J.M., and Gryna, F.M. Quality Planning and Analysis. McGraw-Hill, New York, 1970. pp. 375-377.

4. Nehls, G.J., and Akland, G.G. Procedures for Handling Aerometric Data. Journal of the Air Pollution Control Association, 23 (3):180-184, March 1973.

5. National Primary and Secondary Ambient Air Quality Standards. Federal Register, 36(84): 8194-8195, April 30, 1971. (Note: Copy available in Volume II of this Quality Assurance Handbook.)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1.4.3 ORGANIZATION

1.4.3.1 ABSTRACT

1. Organizing a quality assurance function includes establishing objectives, determining the amount of emphasis to place on each quality assurance element, preparing a quality assurance plan, identifying quality assurance problems to be resolved, and implementing the quality assurance plan.

2. Quality assurance is normally a separate function in the organization.

3. Quality assurance has input into many functions of an air pollution control agency. (See Figure 1.4.3.2 for details.)

4. The basic tools for quality assurance implementation are:

a. Organization chart

b. Job description. (See Figure 1.4.3.3 for job description for the Quality Assurance Coordinator.)

c. Quality assurance plan

1.4.3.2 DISCUSSION

Organizing the quality assurance function[1] - Because of the differences in size, workloads, expertise, and experience in quality assurance activities among agencies adopting the use of a quality assurance system, it is useful here to outline the steps for planning an efficient quality assurance system.

1. Establish quality assurance objectives (precision, accuracy, and completeness) for each measurement system (Section 1.4.2).

2. Determine the quality assurance elements appropriate for the agency (Figure 1.4.).

3. Prepare a quality assurance plan (normally on a project basis) for all measurement systems (Section 1.4.23).

4. On the basis of the quality assurance plan, identify quality assurance problems which must be resolved.

5. Implement the quality assurance plan.

Location of the quality assurance function in the organization[2] - If practical, one individual within an organization should be designated the Quality Assurance (QA) Coordinator. The QA Coordinator should undertake activities such as quality planning, auditing, and reliability. The QA Coordinator should also have the responsibility for coordinating all quality assurance activity so that complete integration of the quality assurance system is achieved. The QA Coordinator should, therefore, gain the cooperation of other responsible heads of the organization with regard to quality assurance matters.

As a general rule, it is not good practice for the quality assurance function to be directly located in the organization responsible for conducting measurement programs. This arrangement could be workable, however, if the person in charge maintains an objective viewpoint.

Relationship of the quality assurance function to other functions - The functions performed by a comprehensive air pollution control program at the state or local level are shown in Figure 1.4.3.1.[3] The relationship of the quality assurance function to the other agency functions is shown in Figure 1.4.3.2. The role of quality assurance can be grouped into two categories:

1. Recommend quality assurance policy and assist its formulation with regard to agency policy, administrative support (contracts and procurements), and staff training.

2. Provide quality assurance guidance and assistance for monitoring networks, laboratory operations, data reduction, instrument maintenance and calibration, litigation, source testing, and promulgation of control regulations.

Basic tools for quality assurance implementation are:

1. The organization chart[4] - The quality assurance organization chart should display line and staff relationships, and lines of authority and responsibility. The lines of authority and responsibility, flowing from the top to bottom, are usually solid, while staff advisory relationships are depicted by dashed lines.

2. The job description[5] - The job description lists the responsibilities, duties, and authorities of the job and relationships to other positions, individuals, or groups. A sample job description for a Quality Assurance Coordinator is shown in Figure 1.4.3.3.

Management Services

- Agency policy
- Administrative and clerical support
- Public information and community relations
- Intergovernmental relations
- Legal counsel
- Systems analysis, development of strategies, long-range planning
- Staff training and development

Technical Services

- Laboratory operations
- Operation of monitoring network
- Data reduction
- Special field studies
- Instrument maintenance and calibration

Field Enforcement Services

- Scheduled inspections
- Complaint handling
- Operation of field patrol
- Preparation for legal actions
- Enforcement of emergency episode procedures
- Source identification and registration

Engineering Services

- Calculation of emission estimates
- Operation of permit system
- Source emission testing
- Technical development of control regulations
- Preparation of technical reports, guides, and criteria on control
- Design and review of industrial emergency episode procedures

Figure 1.4.3.1 List of functions performed by comprehensive air pollution control programs

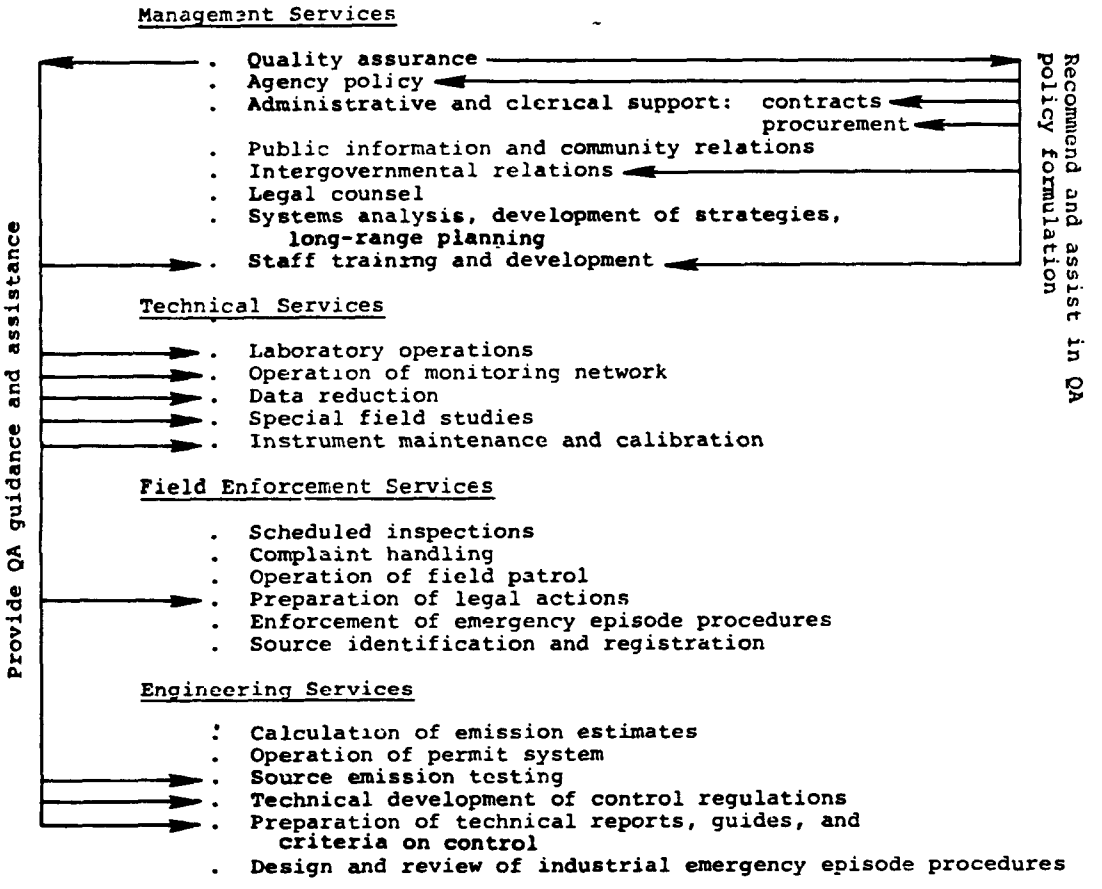


Figure 1.4.3.2 Relationship of the quality assurance function to other air pollution control program functions

TITLE: Quality Assurance Coordinator

Basic Function

The Quality Assurance Coordinator is responsible for the conduct of the quality assurance program and for taking or recommending measures.

Responsibilities and Authority

1. Develops and carries out quality control programs, including statistical procedures and techniques, which will help agencies meet authorized quality standards at minimum cost.

2. Monitors quality assurance activities of the agency to determine conformance with policy and procedures and with sound practice; and makes appropriate recommendations for correction and improvement as may be necessary.

3. Seeks out and evaluates new ideas and current developments in the field of quality assurance and recommends means for their application wherever advisable.

4. Advises management in reviewing technology, methods, and equipment, with respect to quality assurance aspects.

5. Coordinates schedules for measurement system functional check calibrations, and other checking procedures.

6. Evaluates data quality and maintains records on related quality control charts, calibration records, and other pertinent information.

7. Coordinates and/or conducts quality-problem investigations.

Figure 1.4.3.3 Job description for the Quality Assurance Coordinator

3. The quality assurance plan - To implement quality assurance in a logical manner and identify problem areas, quality assurance plans are needed. These are normally prepared on a project basis. For details on preparation of quality assurance plans, see Section 1.4.23.

1.4.3.3 REFERENCES

1. Feigenbaum, A.V. Total Quality Control. McGraw-Hill, New York. 1961. Chapter 4, pp. 43-82.

2. Covino, C.P., and Meghri, A.W. Quality Assurance Manual. Industrial Press, Inc., New York. 1967. Step 1, pp. 1-2.

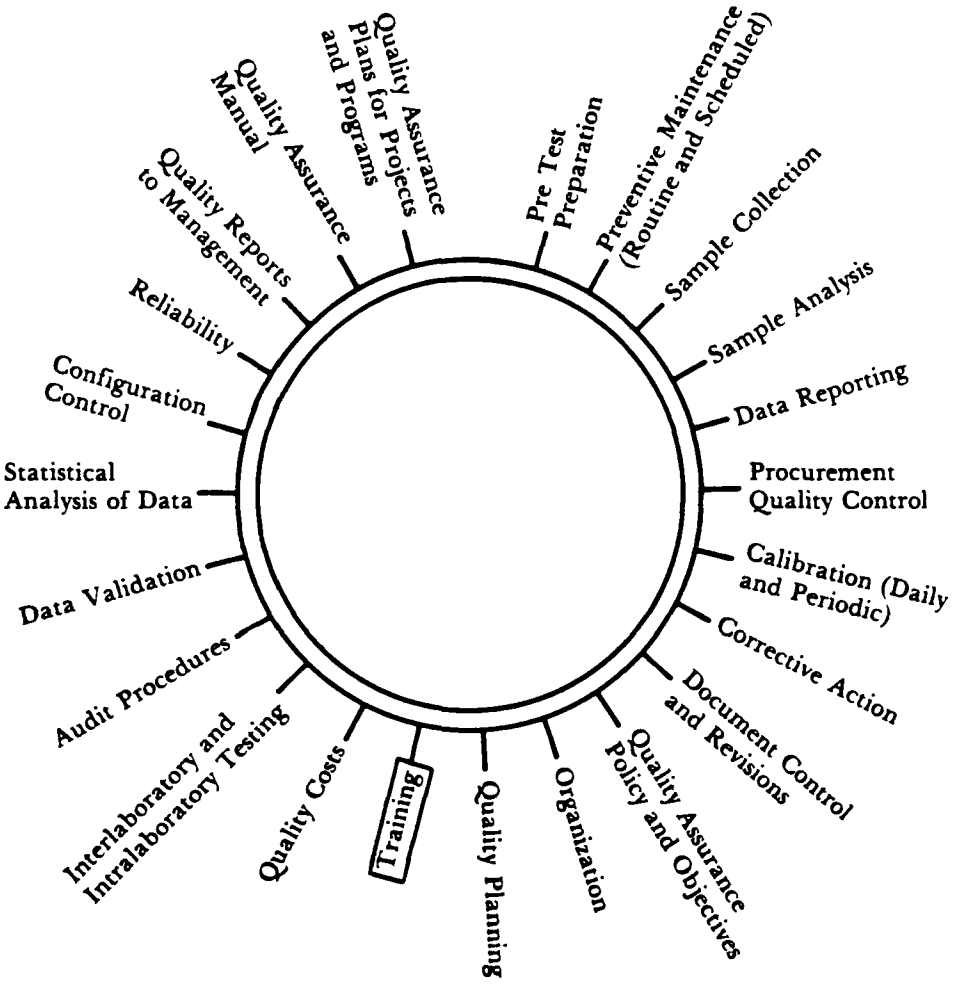
3. Walsh, G.W., and von Lehmden, D.J. Estimating Manpower Needs of Air Pollution Control Agencies. Presented at the Annual Meeting of the Air Pollution Control Association, Paper 70-92, June 1970.

4. Juran, J.M., (ed.). Quality Control Handbook, 2nd Edition. McGraw-Hill, New York. 1962. Section 6, pp. 242.

5. Industrial Hygiene Service Laboratory Quality Control Manual. Technical Report No. 78, National Institute for Occupational Safety and Health, Cincinnati, Ohio. 1974.

BIBLIOGRAPHY

1. Brown, F.R. Management: Concepts and Practice. Industrial College of the Armed Forces, Washington, D.C. 1967. Chapter II, pp. 13-34.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1.4.5 TRAINING

1.4.5.1 ABSTRACT

All personnel involved in any function affecting data quality (sample collection, analysis, data reduction, and quality assurance) should have sufficient training in their appointed jobs to contribute to the reporting of complete and high quality data.

The training methods commonly used in the air pollution control field are the following:

1. On-the-job training (OJT).
2. Short-term course training (normally 2 weeks or less).
3. Long-term course training (quarter or semester in length).

A list of recommended short-term course training is shown in Figure 1.4.5.1.

Training should be evaluated in terms of the trainee and the training per se. The following are techniques commonly used in the air pollution control field to evaluate training.

1. Testing (pre-training and post-training tests).
2. Proficiency checks.
3. Interviews (written or oral with the trainee's supervisor and/or trainee).

1.4.5.2 DISCUSSION

All personnel involved in any function affecting data quality (sample collection, analysis, data reduction, and quality assurance) should have sufficient training in their appointed jobs to contribute to the reporting of complete and high quality data. The Quality Assurance Coordinator should be concerned that the required training is available for these personnel and, when it is not, should recommend to management that appropriate training be made available.

Training objective[1,2] - The training objective should be to develop personnel to the necessary level of knowledge and skill required for air pollution measurement systems (ambient air and source emissions).

Training methods and availability - Several methods of training are available to promote achievement of the desired level of knowledge and skill required. The following are the training methods most commonly used in the air pollution control field; a listing of available training courses for 1975-1976 is given in Figure 1.4.5.1.

1. On-the-job training (OJT) - An effective OJT program could consist of the following:

a. Observe experienced operator perform the different tasks in the measurement process.

b. Study the operational portion of the method as described in the pollutant-specific portion of this Handbook, and use it as a guide for performing the operations.

c. Perform operation under the direct supervision of an experienced operator.

d. Perform operations independently but with a high level of quality assurance checks, utilizing the evaluation technique described later in this section to encourage high quality work.

2. Short-term course training - A number of short-term courses (normally 2 weeks or less) are available that provide knowledge and skills to implement more effectively an air pollution measurement system. Some of the courses are on the measurement methods per se and others provide training useful in the design and operation of the total or selected portions of the measurement system.

3. Long-term course training - Numerous universities, colleges, and technical schools provide long-term (quarter and semester length) academic courses in statistics, analytical chemistry, and other disciplines. The agency's training or personnel officer should be contacted for information on the availability of long-term course training.

Training evaluation - Training should be evaluated in terms of (1) level of knowledge and skill achieved by the operator from the training; and (2) the overall effectiveness of the training, including determination of training areas that need improvement. If a quantitative performance rating can be made on the operator during the training

period (in terms of knowledge and skill achieved), this rating may also provide an assessment of the overall effectiveness of the training as well.

Several techniques are available for evaluating the operator and the training per se. One or more of these techniques should be used during the evaluation. The most common types of evaluation techniques applicable to training in air pollution measurement systems are the following:

1. Testing - A written test before (pretest) and one after (post-test) training are commonly used in short-term course training. This allows the trainee to see areas of personal improvement and provides the instructor with information on training areas that need improvement.

2. Proficiency checks - A good means of measuring skill improvement in both OJT and short-term course training is to assign the trainee a work task. Accuracy and/or completeness are commonly the indicators used to score the trainee's proficiency. The work tasks could be of the following form:

a. Sample collection - Trainee would be asked to list all steps involved in sample collection for a hypothetical case. In addition, the trainee could be asked to perform selected calculations. Proficiency could be judged in terms of completeness and accuracy.

b. Analysis - Trainee could be provided unknown samples for analysis. As defined here, an unknown is a sample whose concentration is known to the work supervisor (OJT) or training instructor (short-term course training) but unknown to the trainee. Proficiency could be judged in terms of accuracy.

c. Data reduction - Trainees responsible for data reduction could be provided data sets to validate. Proficiency could be judged in terms of completeness and accuracy.

If proficiency checks are planned on a recurring basis, a quality control or other type chart may be used to show progress during the training period as well as after the training has been completed. Recurring proficiency checks are a useful technique for determining if additional training may be required.

3. Interviews - In some cases, a written or oral interview with the trainee's supervisor and/or trainee is used to determine if the training was effective. This interview is normally not conducted until the trainee has returned to the job and has had an opportunity to use the training. This technique is most often used to appraise the effectiveness of a training program (OJT or short-term course) rather than the performance of the trainee.

1.4.5.3 REFERENCES

1. Feigenbaum, A.V. Total Quality Control. McGraw-Hill, New York. 1961. pp. 605-615.

2. Feigenbaum, A.V. Company Education in the Quality Problem. Industrial Quality Control, X(6):24-29, May 1974.

BIBLIOGRAPHY

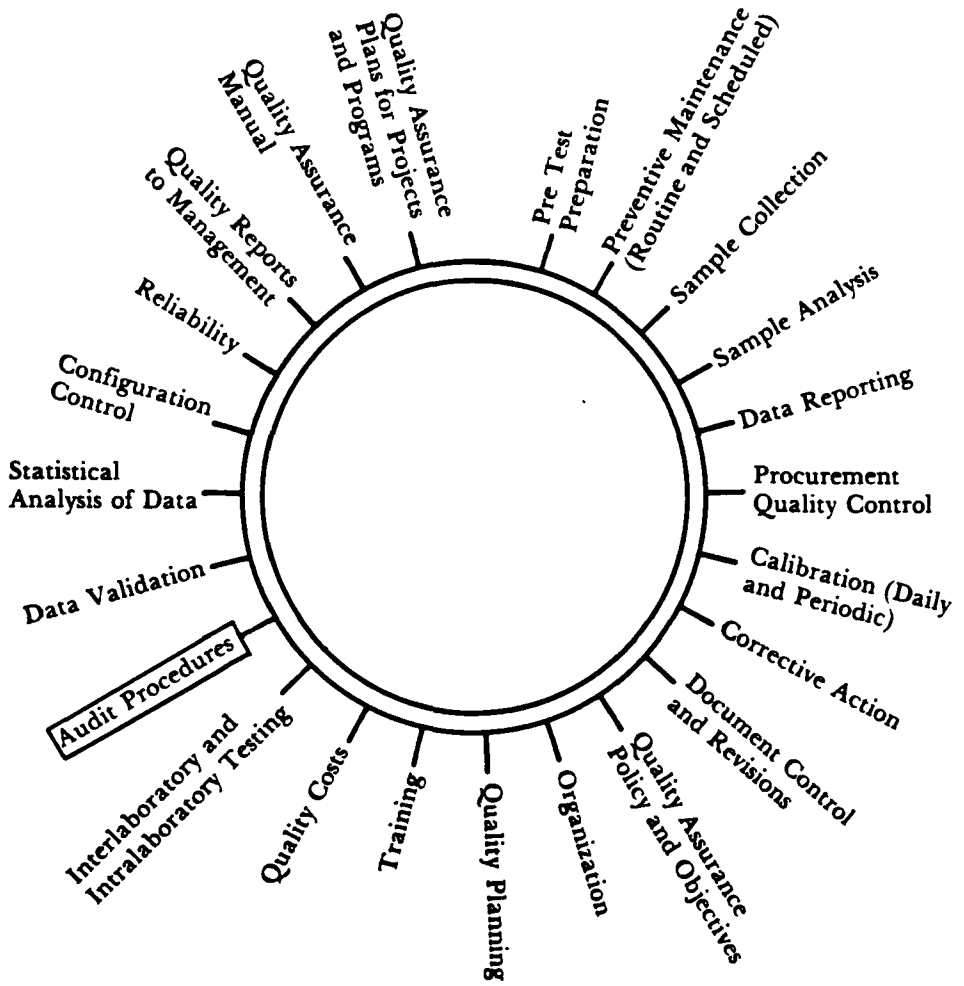
1. Juran, J.M., (ed.). Quality Control Handbook. 2nd edition. , McGraw-Hill, New York, 1962. Section 7, pp. 13-20.

2. Reynolds, E.A. Industrial Training of Quality Engineers and Supervisors. Industrial Quality Control, X(6):29-32, May 1954.

3. Industrial Quality Control, 23(12), June 1967. (All articles deal with education and training.)

4. Seder, L.A. QC Training for Non-Quality Personnel. Quality Progress, VII(7):9.

5. Reynolds, E.A. Training QC Engineers and Managers. Quality Progress, III(4):20-21, April 1970.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1.4.16 AUDIT PROCEDURES

1.4.16.1 ABSTRACT

1. Performance audits are independent checks made by the supervisor or auditor to evaluate the quality of data produced by the total measurement system (sample collection, sample analysis and data processing). Performance audits are normally a quantitative appraisal of quality.

2. A system audit is an on-site inspection and review of the quality assurance system used for the total measurement system (sample collection, sample analysis, data processing, etc.). Quality assurance plans discussed in Section 1.4.23 should be used as the basis for conducting a system audit. System audits are normally a qualitative appraisal of quality.

1.4.16.2 DISCUSSION

Performance audit - Performance audits refer to independent checks made by the supervisor or auditor to evaluate the quality of data produced by the total sampling and analysis system. Performance audits generally are categorized as follows:

1. Sampling audits.
2. Analysis audits.
3. Data processing audits.

These audits are performed independent of and in addition to normal quality control checks by the operator/analyst. Independence can be achieved by having the audit made by a

different operator/analyst from the one conducting the routine measurements or, in the case of sampling or analysis, by the introduction of audit control standards into the sampling or analysis system and the subsequent plotting of results on control charts by the supervisor. The use of audit control standards should be applied without the knowledge of the operator/analyst, if possible, to insure that recorded results reflect normal operating conditions.

Performance audits made by a different operator/analyst from the one conducting the routine measurement may be conducted in several ways. The following are examples of the most common type of audits.

1. Sampling audit - The auditor uses a separate set of calibrated flowmeters and reference standards to check the sample collection system:

   a. Flow rate devices.

   b. Instrument calibration.

   c. Instrument calibration gases, when applicable.

2. Analysis audits - The auditor is commonly provided a portion or aliquot of several routine samples for analysis.

3. Data processing audits - Data reporting commonly involves a spot-check on calculations and data validation may be checked by inserting in the data processing system a dummy set of raw data followed by review of these validated data.

A major problem in audit design is determining the auditing frequency and the lot size (number of samples required to estimate population concentration with a specified percentage confidence).

After the auditing schedule has been implemented, the audit results should be plotted on control charts to give the supervisor a visual picture of changes in the performance so that corrective actions may be taken when necessary. A discussion of recommended control chart types, instructions on construction, and criteria for interpretation are included in Appendix H, "Control Charts."

System audit - A system audit is an on-site inspection and review of the quality assurance system used for the total measurement system (sample collection, sample analysis, data processing, etc.) for each monitoring sensor. Whereas performance audits are a quantitative appraisal, system audits are normally a qualitative appraisal.

The quality assurance plans for projects and programs discussed in Section 1.4.23 should be used as the basis for conducting a system audit. For convenience, some of the items recommended for a QA plan are repeated here:

1. Organization and responsibility - Is the quality assurance organization operational?

2. Sample collection - Are written sample-collection procedures available and are these followed as written?

3. Sample analysis - Are written analysis procedures available and are these followed as written?

4. Data validation - Is a list of criteria for data validation available and is it used?

5. Calibration - Are written calibration procedures available and are these followed as written? In addition, a review should be made of procedures used to establish traceability of calibration standards to standards of higher accuracy and of the calibration schedule and data by measurement sensor.

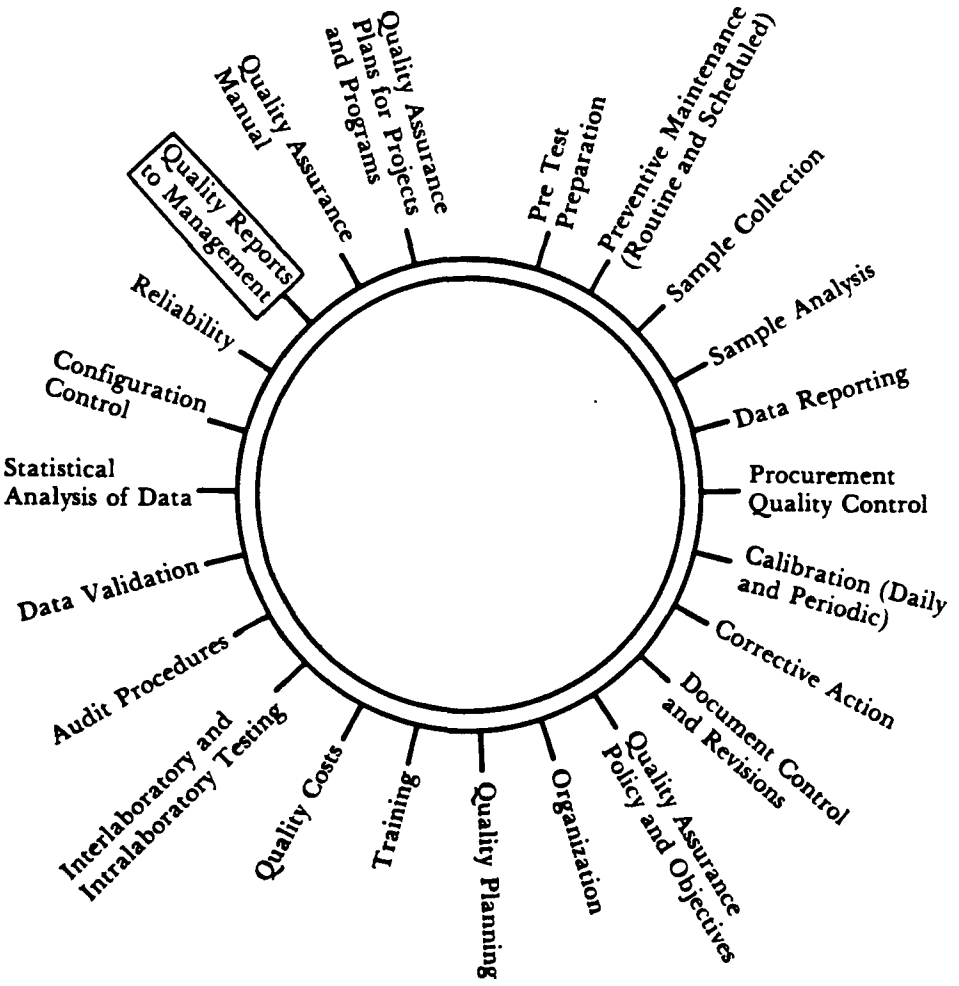
6. Audits - Are control charts for performance audits reviewed?

7. Interlaboratory tests - Are results from interlaboratory testing reviewed?

8. Preventive maintenance - Is the preventive maintenance schedule being followed as recommended in the QA plan?

Other items for consideration in a system audit are shown in Section 2.0 of Volume II, Ambient-Air Specific Methods and Section 3.0 of Volume III, Stationary-Source Specific Methods.

A system audit may be made at any time during the life of a project but is normally conducted before or just after monitoring has been initiated.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1.4.21 QUALITY REPORTS TO MANAGEMENT

1.4.21.1 ABSTRACT

Quality facts to be reported should be defined and methods for summarizing and reporting data should be determined.

Quality facts usually reported are:

1. Percentage duplication or replication of determinations.
2. Instrument or equipment downtime.
3. Percentage voided samples versus total samples.
4. Quality cost in terms of prevention, appraisal, and correction costs.
5. System audit (on-site inspection) results.
6. Performance audit results.
7. Interlaboratory test results and, where appropriate, intralaboratory test results (precision and accuracy).
8. Status of solutions to major quality assurance problems.

The Quality Assurance Coordinator should consult with line staff to determine the necessary reporting requirements. Reports should be obtained from source documents. Reports should have a baseline for comparison and should be easy to interpret. Similar reports should be combined if possible.

1.4.21.2 DISCUSSION[1]

Quality assurance facts to be reported may vary widely from one air pollution control agency to another, depending on size and organization of the agencies. The quality reports listed in the abstract represent the kinds of information commonly needed by management.

The details or sources of such facts are obtained from other sections of the Handbook as follows:

	Facts	Source
1.	Percentage of duplication or replication of determinations.	Methods (Vols. 2 and 3)
2.	Instrument or equipment downtime.	1.4.20 (Vol. 1)
3.	Percentage of voided samples versus total samples.	Methods (Vols. 2 and 3) 1.4.17 (Vol. 1)
4.	Quality costs (prevention, appraisal, and correction costs).	1.4.14 (Vol. 1)
5.	System audit (on-site inspection) results.	1.4.16 (Vol. 1)
6.	Performance audit results.	1.4.16 (Vol. 1)
7.	Interlaboratory test results and, where appropriate, intralaboratory tests results (precision and accuracy).	1.4.15 (Vol. 1)
8.	Status of solutions to major quality problems.	1.4.13 (Vol. 1)

Principles of reporting to management - The following are general principles that should be considered:

1. In order to determine what periodic reports are needed or desired by management, the Quality Assurance

Coordinator should discuss with superiors and staff members what reports need to be published on a regular basis to provide information required for rational decision-making.

2. In order to minimize errors in transmission, translation, and interpretation, facts used in reports to management should be obtained from source documents when possible.

3. Facts should be presented in summary form. When not presented graphically, the report should provide abstracts of essential points.

4. Reports should compare current data with some base. The basic standard of comparison can be:

a. Based on previous performance (historic).

b. Based on engineering judgment.

5. The report should be understood at a glance. If the report is not presented graphically, essential points should be reduced to a single page, preferably double-spaced. An example of a graphic report is shown in Figure 1.4.21.1. Figure 1.4.14.1 for quality costs (in terms of prevention, appraisal, and corrective costs) is another example of graphic presentation.

Where applicable, quality control charts should be used to report results from performance audits, intralaboratory tests and, when performed on a recurring basis, interlaboratory test results.

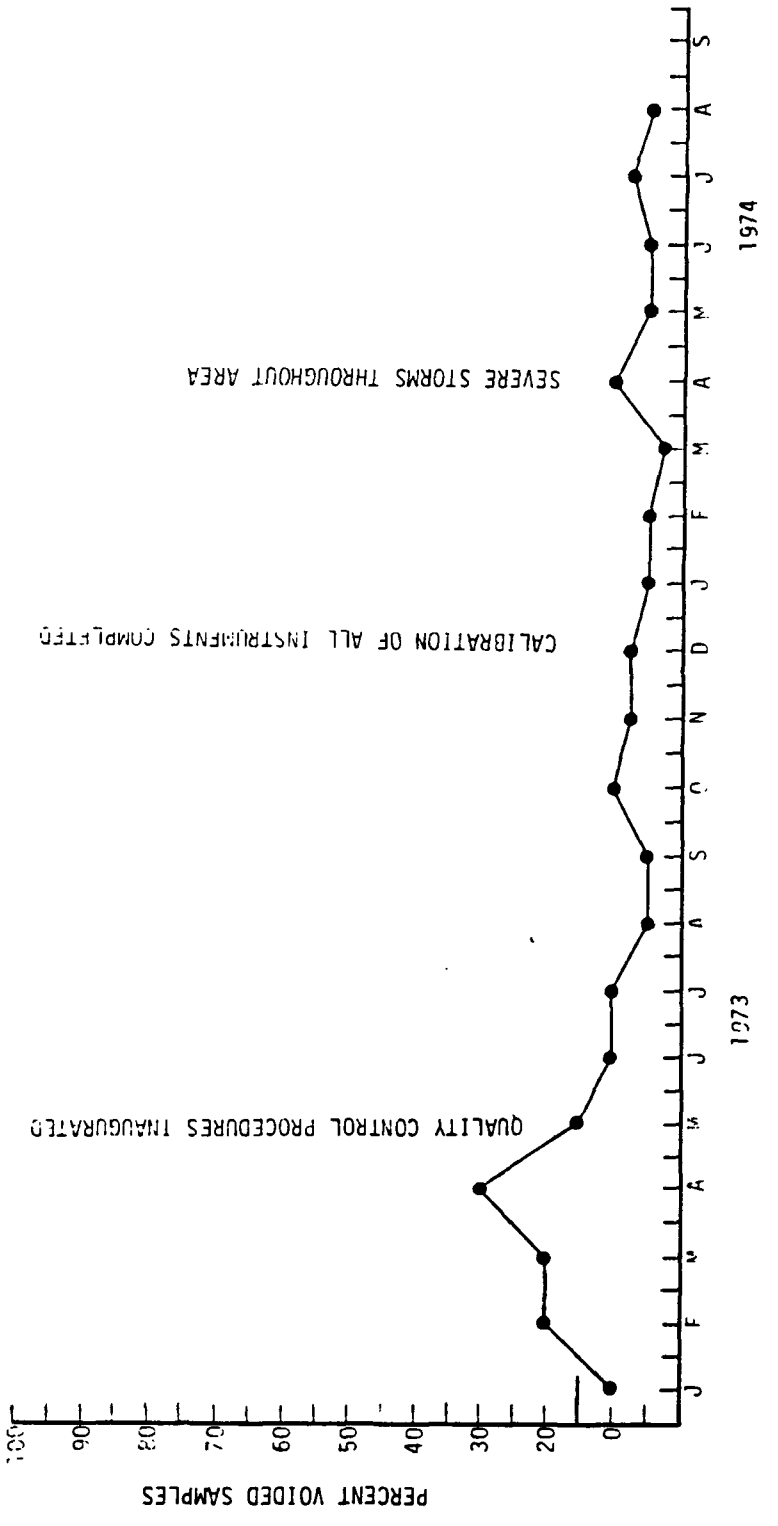


Figure 1.4.21.1 Graphic report to management.

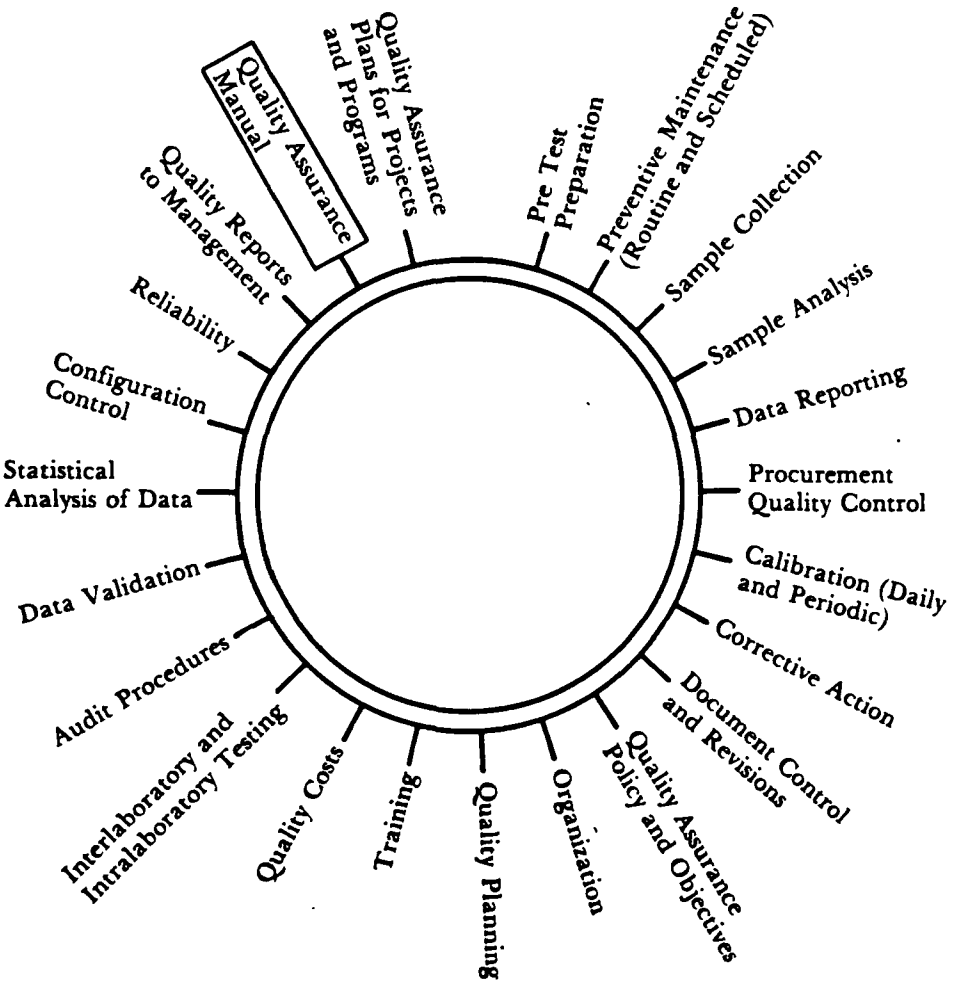
1.4.21.3 REFERENCES

1. Juran, J.M., (ed.). Quality Control Handbook. 2nd ed. McGraw-Hill, New York. 1962. Sec. 12, pp. 26-32.

BIBLIOGRAPHY

1. Juran, J.M., and Gryna, F.M. Quality Planning and Analysis. McGraw-Hill, New York. 1970.

2. Spear, M.E. Charting Statistics. McGraw-Hill, New York. 1952.


Quality Assurance Manual
Quality Assurance Plans for Projects and Programs
Pre Test Preparation
Preventive Maintenance (Routine and Scheduled)
Sample Collection
Sample Analysis
Data Reporting
Procurement Quality Control
Calibration (Daily and Periodic)
Corrective Action
Document Control and Revisions
Quality Assurance Policy and Objectives
Organization
Quality Planning
Training
Quality Costs
Interlaboratory and Intralaboratory Testing
Audit Procedures
Data Validation
Statistical Analysis of Data
Configuration Control
Reliability
Quality Reports to Management

1.4.22 QUALITY ASSURANCE MANUAL

1.4.22.1 ABSTRACT

1. A quality assurance manual is a manual of general requirements needed to assure quality. Specific requirements for each project are contained in a quality assurance plan.

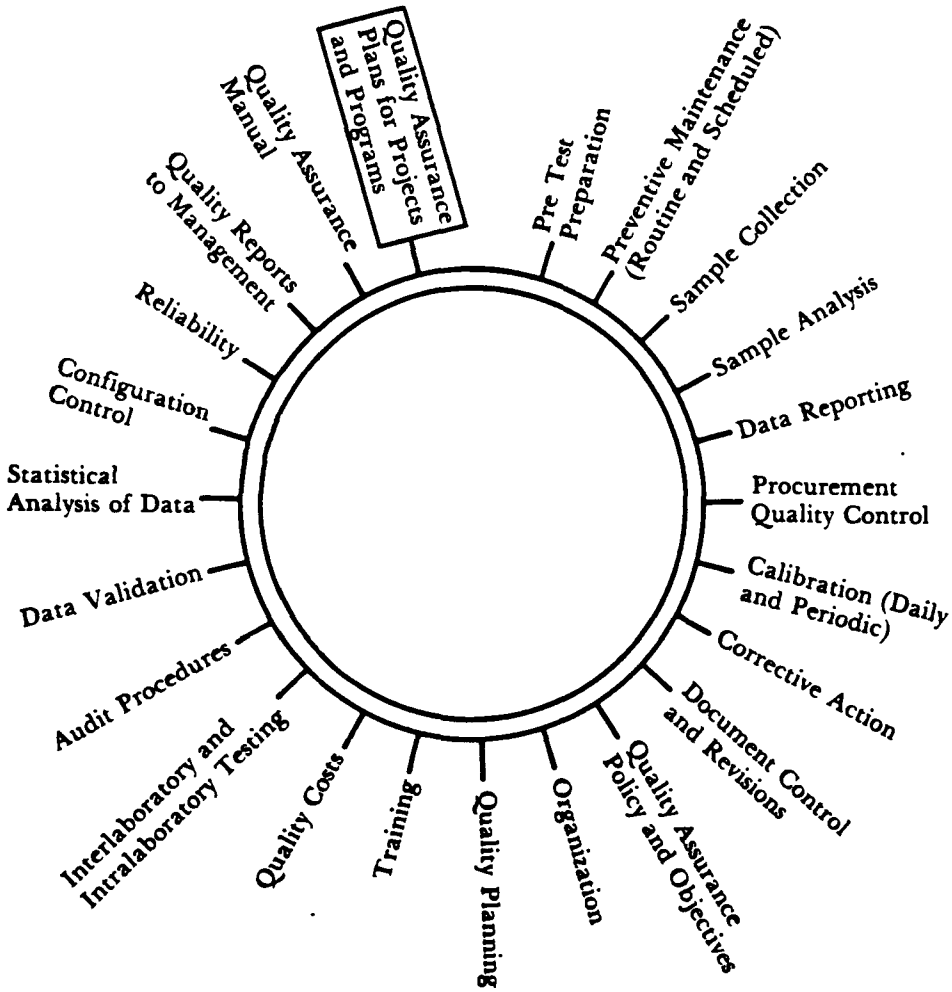
2. Each state and local air pollution control agency and others involved in air program research and monitoring activities need a quality assurance manual. These organizations should use the "Elements of Quality Assurance" described in Section 1.4 of this Handbook as a guide in developing their own manuals.

1.4.22.2 DISCUSSION

Quality assurance manual content - A common practice for quality assurance in industry is for each company concerned with the design, development, and manufacturing of a product to prepare a quality assurance manual. This manual states the company's general philosophy and requirements with respect to quality assurance by functional activities that are associated with the quality of the product. The specific requirements for each product (or group of products) are contained in a quality assurance plan.

State and local air pollution control agencies and others need a quality assurance manual - Each state and local air pollution control agency and others involved in

air program research and monitoring activities need a quality assurance manual. The manual should define management's position on quality assurance and provide guidelines that should be followed by subordinates in preparing quality assurance plans. These organizations should use the "Elements of Quality Assurance" described in Section 1.4 of this Handbook as a guide in developing their own manual.


Quality Assurance Plans for Projects and Programs
Quality Assurance Manual
Quality Reports to Management
Reliability
Configuration Control
Statistical Analysis of Data
Data Validation
Audit Procedures
Interlaboratory and Intralaboratory Testing
Quality Costs
Training
Quality Planning
Organization
Quality Assurance Policy and Objectives
Document Control and Revisions
Corrective Action
Calibration (Daily and Periodic)
Procurement Quality Control
Data Reporting
Sample Analysis
Sample Collection
Preventive Maintenance (Routine and Scheduled)
Pre Test Preparation

1.4.23 QUALITY ASSURANCE PLANS FOR PROJECTS AND PROGRAMS

1.4.23.1 ABSTRACT

1. A quality assurance plan (QA plan) is specific quality assurance requirements prepared for a project or program for the elements discussed in Section 1.4 of this Handbook.

2. QA plans should be prepared for both inhouse and contract projects.

3. The depth of a QA plan is influenced primarily by:

   a. Intended use of the data.

   b. Whether the project is new or on-going.

4. An example format for a QA plan is given.

1.4.23.2 DISCUSSION

Definition of quality assurance plan (QA plan) - The specific requirements prepared by or for a project officer (single project) or a program manager* (group of similar projects) to ensure quality data from each air measurement system are called a QA plan.

Quality assurance plans for inhouse and contract projects - A QA plan should be prepared and implemented to include each air pollution measurement system in an inhouse or contract project. The plan should be prepared by the project officer for inhouse projects and for the project officer's review and approval for contracts. The Quality

* For simplicity the terms project officer and project will be used throughout the discussion.

Assurance Coordinator should be involved in the review of the QA plan prior to implementation. The plan should be based on project objectives and should be consistent with management's general requirements on quality assurance that are specified in the quality assurance manual (see Section 1.4.22).

Quality assurance elements in a QA plan - All quality assurance elements discussed in Section 1.4 should be considered in the preparation of the QA plan. However, different projects will require emphasis on different elements. Factors that will have the most influence on the QA plan design are:

1. Intended use of the data - Monitoring for enforcement or the establishment of air pollution criteria levels (normally for health effects) will result in closer scrutiny of the project from a quality assurance standpoint than projects in which legal actions may not be involved; e.g., research on the development of a new monitoring instrument.

2. New project versus improvement in an on-going project - Improving quality assurance on a current project normally means that the operators are on the job and the air measurement equipment is in operation. For on-going projects, the quality assurance elements that are particularly beneficial to consider in the QA plan are:

a. Organization (Section 1.4.3); namely, the project quality organization and responsibility.

b. Sample collection (Section 1.4.8), includes sampling handling and storage requirements. Include a copy of all procedures, or if standard procedures are used, a reference to these will suffice.

c. Sample analysis (Section 1.4.9). Include a copy of all procedures, or if standard procedures are used, a reference to these will suffice.

d. Data reporting (Section 1.4.10).

e. Data validation (Section 1.4.17), including specific criteria that are to be applied for validation of data.

f. Audit procedures (Section 1.4.16) including specific performance audits that are to be performed during routine project operations.

g. Calibration (Section 1.4.12), including description of procedure for establishing traceability, description of calibration standards, and a schedule for calibration. Include a copy of all calibration procedures, or if standard procedures are used, a reference to these will suffice.

h. Preventive maintenance (Section 1.4.7), including a schedule for maintenance.

i. Interlaboratory tests (Section 1.4.15), including a list of any planned or anticipated participation in interlaboratory tests.

j. Quality reports to management (Section 1.4.21), including the information content of the reports and the frequency of reporting.

Project officers or managers for new projects will need to consider all elements mentioned previously for on-going projects and, in addition, should probably pay special attention also to the following areas:

a. Pretest preparation (Section 1.4.6). For new monitoring projects, a preliminary visit to select sampling sites for ambient air monitoring and source emission monitoring will normally be required. Criteria or factors used to select the sample collection sites should be listed.

b. Training (Section 1.4.5). Commonly, new projects may result in the hiring of new personnel or the assignment of current personnel to different work tasks for which training may be required. Anticipated training should be listed.

c. Procurement quality control (Section 1.4.11). A new project may mean procurement of new equipment and reagents. Equipment and reagents that should be placed under procurement quality control should be listed and, if available, acceptance limits should also be included.

d. Reliability (Section 1.4.20). When equipment reliability is important, the equipment should be listed and reliability limits provided.

e. Configuration control (Section 1.4.19). Any plans for configuration control on large projects should be summarized.

Quality assurance plan document control and distribution - The preparation of a QA plan requires that the project officer consider all quality assurance elements listed previously and plan for those elements that will have the most quality impact in the project. The result of this planning is the QA plan per se. The QA plan represents the project officer's best judgment on the quality assurance level required for the project. Therefore, the QA plan should be readily available to all project personnel with distribution copies made available to those personnel most affected by the plan. The plan should be in a document control format in order to maintain a historical record of major revisions in the project quality assurance program.

Quality assurance plan format - The format used for the QA plan will vary from project officer to project officer. The following format is suggested, primarily to highlight points for consideration by the project officer in preparing the plan.

1. Table of Contents. Show in document control and revision format similar to the Handbook table of contents.

2. Project Description. Give a brief project description including references to be consulted for more details.

3. Organization and Responsibility. Give a table or

chart showing key individuals or groups involved in the project quality assurance activities.

4. Project Quality Assurance Program. The following is a minimum that should be contained in QA plans in regard to the elements of quality assurance:

a. Pretest preparation. Include criteria or factors used to select sample collection sites. For ambient air monitoring, a map of the site with the location of air pollution and meteorological sensors should be included; and for source emission monitoring, a flow diagram of the process to be tested and a diagram (with dimensions) of the stack or duct to be tested, plus pretest engineering data on stack velocity, stack temperature, etc.

b. Sample collection. Include a copy of each sensor sample collection procedure. For manual sample collection, include a procedure for sample handling and storage requirements. If standard procedures are used, reference to these procedures will suffice (e.g., EPA Method 5 - Stationary Source Emission Monitoring for Particulates).

c. Sample analysis. Include a copy of all analytical procedures. If standard procedures are used, reference to these procedures will suffice.

d. Data reporting. Include a copy of all forms for reporting data and an example calculation for each pollutant measurement.

e. Data validation. Include a list of criteria that should be used to validate data.

f. Calibration. Include a copy of all calibration procedures. If standard procedures are used, reference to these procedures will suffice. By sensor, provide: (1) a schedule for calibration; (2) description of type of standard; and (3) procedure planned to establish traceability of calibration standards to available standards of higher quality.

g. Audits. Include a plan for performance and system (on-site inspection) auditing. For performance audits, recommend a frequency for an initial audit by sensor.

h. Interlaboratory tests. Any planned or anticipated participation in interlaboratory tests should be listed.

i. Preventive maintenance. Include a copy of preventive maintenance procedures for sensor and auxiliary equipment. Where vendor preventive maintenance procedures are available, reference to these will suffice. A preventive maintenance schedule should be recommended.

j. Other. Any other quality assurance elements, e.g., procurement quality control, configuration control, training, etc., that are applicable to the project should also be included.

5. Forms. Any forms that are not included in part 4a through 4g should be included.

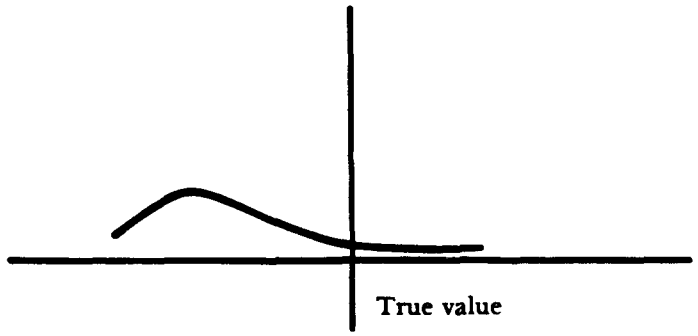
6. Distribution list. A complete list of all organizations and persons to whom the QA plan has been distributed should be included.

You have completed your reading for Assignment 2. Do the review exercises which follow and check your answers after you complete them. The correct answers are given on the page following the review exercises.

Reading Assignment 2 Review Exercises

1. Quality control is:
   a. a system of management equivalent to a quality assurance system.
   b. a system of data management used to ensure correct reporting of test results.
   c. the system of statistical procedures used to ensure data quality.
   d. the system of activities used to provide a quality product or result.
2. Quality assurance is:
   a. the system of activities used to provide assurance that the quality control system is performing adequately.
   b. the system of activities used to statistically determine confidence levels for air quality data.
   c. the system of activities used to provide a quality product.
   d. the system of activities used to provide assurance that air quality is improving.
3. The quality assurance wheel illustrates:
   a. the elements that should be considered when planning a quality assurance program.
   b. the costs associated with quality assurance programs.
   c. mandatory requirements for any quality assurance program.
   d. the management structure of the EPA Quality Assurance Division.
4. The objectives of a quality assurance program should be to produce data that are:
   a. _____
   b. _____
   c. _____
   d. _____
   e. _____

5. The data illustrated below are:



   a. both precise and accurate.
   b. precise but not accurate.
   c. both imprecise and inaccurate.
   d. accurate but not precise.

6. The quality assurance coordinator of a source testing organization should:
   a. be the newest employee on the sampling team.
   b. be a test team leader.
   c. be independent from other organizational programs.
   d. report to the laboratory supervisor.

7. List the three most common training methods used in the field of air pollution control.

   a. _____

   b. _____

   c. _____

8. List at least three techniques that can be used to check the effectiveness of a training program.

   a. _____

   b. _____

   c. _____

9. An auditing procedure is one of the elements of a QA program that should be implemented as soon as possible. What are two types of audits that could be set up in a source sampling QA program?

   a. _____

   b. _____

10. What is the difference between a performance audit and a system audit?
    a. A performance audit is qualitative, whereas a system audit is quantitative.
    b. A performance audit is quantitative, whereas a system audit is qualitative.

11. Which one of the following statements would **not** be true of a performance audit?
    a. An aliquot of an unknown standard is submitted for lab analysis.
    b. The source tester is required to solve a sample problem on his nomograph before testing.
    c. The sample-collection procedures are checked off as the test is done.
    d. The dry-gas meter of the Method 5 train is checked using an EPA audit device.

12. Which one of the following statements would **not** be included in a QA report to management?
    a. Aliquots of the recovered Method 6 sample sent to ACME labs agreed with 2% of those determined by the test contractor.
    b. Laboratory analysis indicated a 30% disagreement with the EPA NO_x audit sample stated value.
    c. Inaccuracies in determining ΔH values from a nomograph were avoided in Tests 2 and 3 by using a calculator.
    d. The nozzle was attached to the probe and then connected to the sample case in the normal manner.

13. What is the difference between a quality assurance plan and a quality assurance manual?

Answers to
Reading Assignment 2 Review Exercises

1. a b c (d)
2. (a) b c d
3. (a) b c d
4. a. complete
   b. precise
   c. accurate
   d. representative
   e. comparable
5. a b (c) d
6. a b (c) d
7. a. on-the-job
   b. short-term
   c. long-term
8. a. written tests
   b. proficiency checks
   c. interviews
9. a. performance audit
   b. system audit
10. a (b)
11. a b (c) d
12. a b c (d)
13. A quality assurance manual is general and covers the QA programs and methods used by an organization, whereas a quality assurance plan is method specific.

Lesson B

Volume III Overview

Lesson Goal

The goal of this lesson is (a) to show you how to use Volume III and (b) to review for you some aspects of source sampling before proceeding with the specific QA procedures developed for each method.

Lesson Objectives

After completing this lesson, you should be able to:

1. describe how Volume III of the QA Manual is organized with respect to reference methods and topics.
2. explain how you can develop other manuals from Volume III.
3. define the responsibilities of the source tester.
4. list at least four activities involved in planning a source test.
5. understand the procedures outlined in EPA Reference Method 1 for locating sampling points in rectangular and circular ducts.
6. outline how one can check for the presence of cyclonic flow.
7. locate in Volume III a listing of suggested equipment for a source-test team.
8. use proper labeling procedures for source-test reagents and samples.
9. discuss why chain-of-custody procedures are important in compliance test cases.
10. summarize EPA Reference Methods 2 through 8.

Materials

Assignment 3

Table of Contents of Volume III and pages 1 through 4 of Volume III, *Purpose and Overview of the Quality Assurance Handbook*

Assignment 4

First three parts of Section 3.0 of Volume III, including:

3.0 Summary
3.0.1 Planning the Test Program
3.0.2 General Factors Involved in Stationary Source Testing
3.0.3 Chain-of-Custody Procedure for Source Sampling

Assignment 5

- Section 3.1 Summary and Method Highlights for Method 2
- Section 3.1.12 Data Forms for Method 2

Reading Guidance—Assignment 3

Volume III of the US EPA *Quality Assurance Handbook* contains a great deal of accumulated material. At first glance, it may seem more than one can deal with, but on closer examination, you will find that much of Volume III contains reference material, check sheets, or data forms.

The handbook contains a great deal of information that you would normally have to look up in periodicals, books, or EPA reports. The handbook brings together much scattered information and gives detailed procedures which in many cases amplify those given in the Code of Federal Regulations. Volume III expands on the methods required, what is suggested, and what is not required.

> Begin your reading of Volume III by reviewing the Table of Contents, pages 1 of 4 through 4 of 4, and *Purpose and Overview of the Quality Assurance Handbook,* which follows, pages 1 of 5 through 5 of 5.

Many points in the reference methods can be subject to interpretation. Different agencies have established their own interpretations of many of these points. In fact, if we consider several agencies, there may be five or six ways of doing the same thing. Volume III presents detailed and consistent procedures so that ambiguity can be held to a minimum. The procedure that was elected may or may not be better than some of the others that are possible. Ideally, the methods will be made more uniform throughout the agencies through the use of Volume III.

Volume III is organized for compliance testing. If source testers closely follow the procedures, they should get results with good reproducibility. Hopefully, the results will be accurate; however, it is difficult to determine accuracy in source sampling because the *true value* of the emissions is seldom known. The reference method procedures may in some cases give results higher than true because of the way in which calibrations or corrections are made. However, this is not a great problem for agencies in terms of compliance testing since if a source is within the emission standard as determined by the reference method, any positive testing bias would imply that the source is even cleaner than determined.

When testing is performed for tuning process operations, determining efficiency guarantees, and so on, even more care must be taken. For example, pitot-tube—probe assemblies would need to be calibrated as used in the test, impinger catches accounted for, etc.

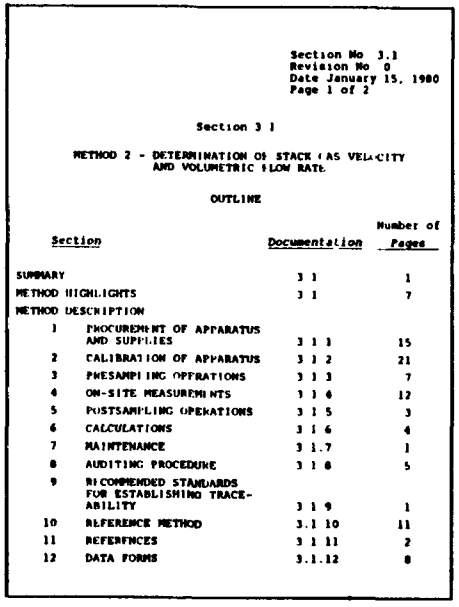
In Volume III, each reference method is divided into the following sections.

Summary **Method Highlights** **Method Description**	**Documented as**
1. Procurement of Apparatus and Supplies	__.__.1
2. Calibration of Apparatus	__.__.2
3. Presampling Operations	__.__.3
4. On-site Measurements	__.__.4
5. Postsampling Operations	__.__.5
6. Calculations	__.__.6
7. Maintenance	__.__.7
8. Auditing Procedures	__.__.8
9. Recommended Standards for Establishing Traceability	__.__.9
10. Reference Method	__.__.10
11. References	__.__.11
12. Data Forms	__.__.12

This makes it easy to know where to go for information about a reference method. This organization also allows you to prepare separate manuals for specific purposes. For example, a calibration manual can be developed by removing Subsections 3.1.2, 3.2.2, 3.3.2, etc., and combining them. A test team checking equipment before a test could then easily find and follow the required procedures.

Similarly, an on-site testing manual can be prepared by combining Subsections 3.1.4, 3.2.4, 3.3.4, etc. A manual for the analytical laboratory can be prepared by using Subsections 3.1.5, 3.2.5, 3.3.5, and so on.

The figure on the following page shows how the seven major sections of Volume III are organized:



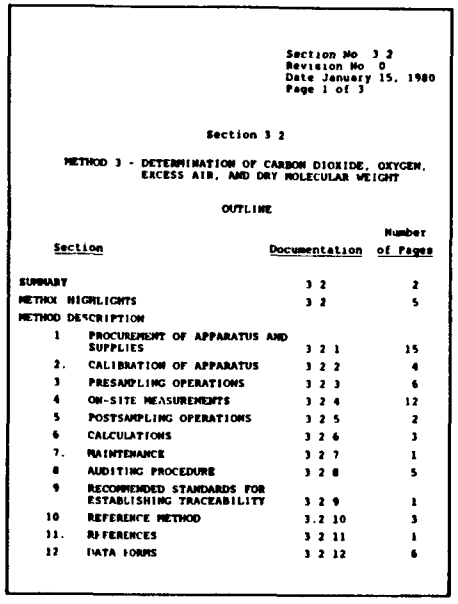
Section No 3.1
Revision No 0
Date January 15, 1980
Page 1 of 2

Section 3 1

METHOD 2 - DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE

OUTLINE

Section		Documentation	Number of Pages
SUMMARY		3 1	1
METHOD HIGHLIGHTS		3 1	7
METHOD DESCRIPTION			
1	PROCUREMENT OF APPARATUS AND SUPPLIES	3 1 1	15
2	CALIBRATION OF APPARATUS	3 1 2	21
3	PRESAMPLING OPERATIONS	3 1 3	7
4	ON-SITE MEASUREMENTS	3 1 4	12
5	POSTSAMPLING OPERATIONS	3 1 5	3
6	CALCULATIONS	3 1 6	4
7	MAINTENANCE	3 1.7	1
8	AUDITING PROCEDURE	3 1 8	5
9	RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY	3 1 9	1
10	REFERENCE METHOD	3.1 10	11
11	REFERENCES	3 1 11	2
12	DATA FORMS	3.1.12	8



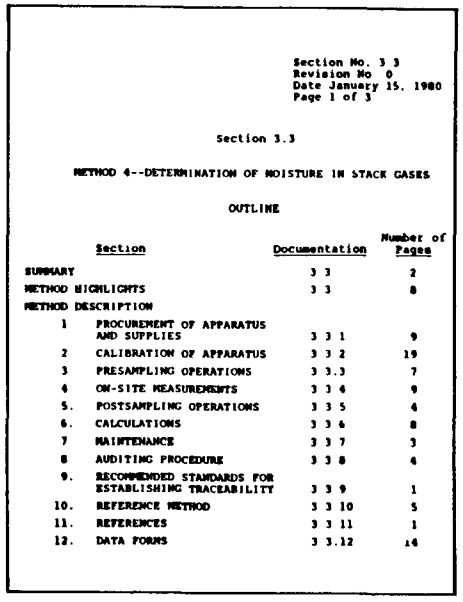
Section No 3.2
Revision No 0
Date January 15, 1980
Page 1 of 3

Section 3.2

METHOD 3 - DETERMINATION OF CARBON DIOXIDE, OXYGEN, EXCESS AIR, AND DRY MOLECULAR WEIGHT

OUTLINE

Section		Documentation	Number of Pages
SUMMARY		3 2	2
METHOD HIGHLIGHTS		3 2	5
METHOD DESCRIPTION			
1	PROCUREMENT OF APPARATUS AND SUPPLIES	3 2 1	15
2.	CALIBRATION OF APPARATUS	3 2 2	4
3	PRESAMPLING OPERATIONS	3 2 3	6
4	ON-SITE MEASUREMENTS	3 2 4	12
5	POSTSAMPLING OPERATIONS	3 2 5	2
6	CALCULATIONS	3 2 6	3
7.	MAINTENANCE	3 2 7	1
8	AUDITING PROCEDURE	3 2 8	5
9	RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY	3 2 9	1
10	REFERENCE METHOD	3.2 10	3
11.	REFERENCES	3 2 11	1
12	DATA FORMS	3 2 12	6



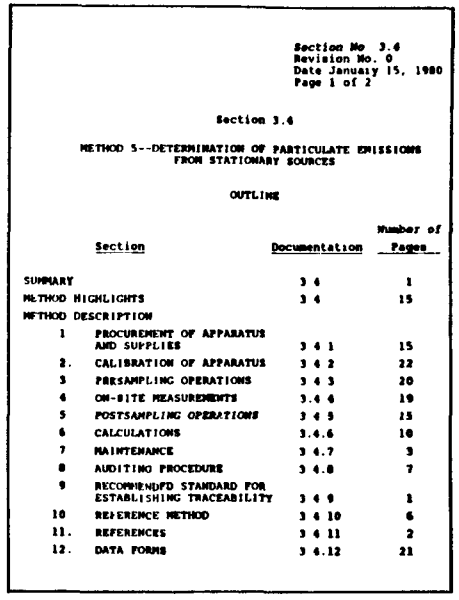
Section No. 3.3
Revision No 0
Date January 15, 1980
Page 1 of 3

Section 3.3

METHOD 4--DETERMINATION OF MOISTURE IN STACK GASES

OUTLINE

Section		Documentation	Number of Pages
SUMMARY		3 3	2
METHOD HIGHLIGHTS		3 3	8
METHOD DESCRIPTION			
1	PROCUREMENT OF APPARATUS AND SUPPLIES	3 3 1	9
2	CALIBRATION OF APPARATUS	3 3 2	19
3	PRESAMPLING OPERATIONS	3 3.3	7
4	ON-SITE MEASUREMENTS	3 3 4	9
5.	POSTSAMPLING OPERATIONS	3 3 5	4
6.	CALCULATIONS	3 3 6	8
7	MAINTENANCE	3 3 7	3
8	AUDITING PROCEDURE	3 3 8	4
9.	RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY	3 3 9	1
10.	REFERENCE METHOD	3 3 10	5
11.	REFERENCES	3 3 11	1
12.	DATA FORMS	3 3.12	16



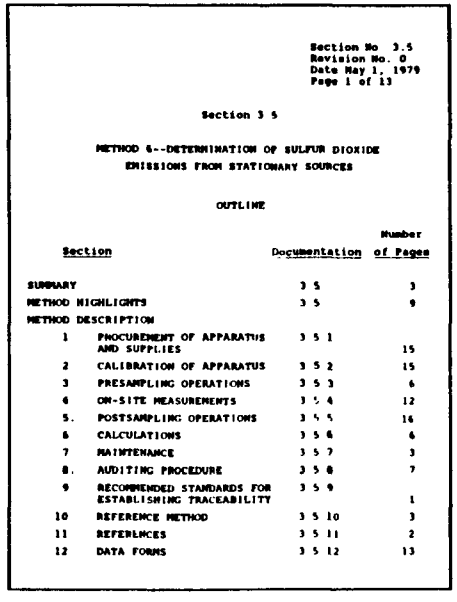
Section No 3.4
Revision No. 0
Date January 15, 1980
Page 1 of 2

Section 3.4

METHOD 5--DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

OUTLINE

Section		Documentation	Number of Pages
SUMMARY		3 4	1
METHOD HIGHLIGHTS		3 4	15
METHOD DESCRIPTION			
1	PROCUREMENT OF APPARATUS AND SUPPLIES	3 4 1	15
2.	CALIBRATION OF APPARATUS	3 4 2	22
3	PRESAMPLING OPERATIONS	3 4 3	20
4	ON-SITE MEASUREMENTS	3.4 4	19
5	POSTSAMPLING OPERATIONS	3 4 5	15
6	CALCULATIONS	3.4.6	10
7	MAINTENANCE	3 4.7	3
8	AUDITING PROCEDURE	3 4.8	7
9	RECOMMENDED STANDARD FOR ESTABLISHING TRACEABILITY	3 4 9	1
10	REFERENCE METHOD	3 4 10	6
11.	REFERENCES	3 4 11	2
12.	DATA FORMS	3 4.12	21



Section No 3.5
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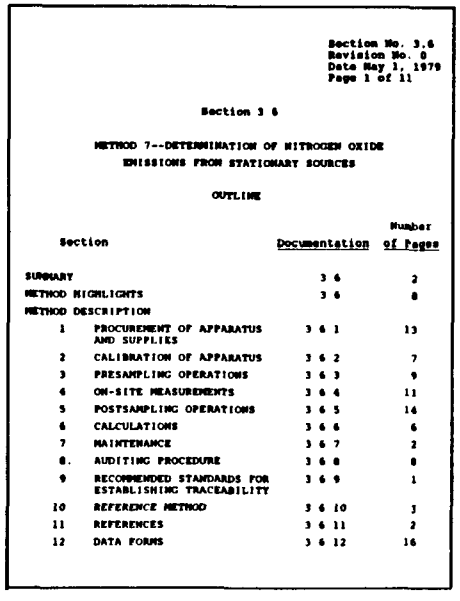
Section 3 5

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

OUTLINE

Section		Documentation	Number of Pages
SUMMARY		3 5	3
METHOD HIGHLIGHTS		3 5	9
METHOD DESCRIPTION			
1	PROCUREMENT OF APPARATUS AND SUPPLIES	3 5 1	15
2	CALIBRATION OF APPARATUS	3 5 2	15
3	PRESAMPLING OPERATIONS	3 5 3	6
4	ON-SITE MEASUREMENTS	3 5 4	12
5.	POSTSAMPLING OPERATIONS	3 5 5	16
6	CALCULATIONS	3 5 6	6
7	MAINTENANCE	3 5 7	3
8.	AUDITING PROCEDURE	3 5 8	7
9	RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY	3 5 9	1
10	REFERENCE METHOD	3 5 10	3
11	REFERENCES	3 5 11	2
12	DATA FORMS	3 5 12	13



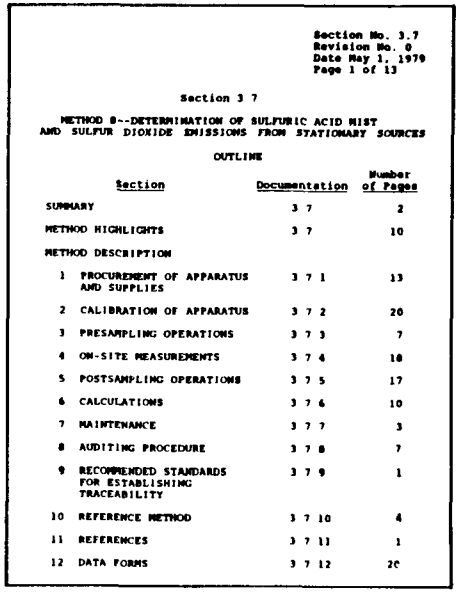
Section No. 3.6
Revision No. 0
Date May 1, 1979
Page 1 of 11

Section 3 6

METHOD 7--DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

OUTLINE

Section		Documentation	Number of Pages
SUMMARY		3 6	2
METHOD HIGHLIGHTS		3 6	8
METHOD DESCRIPTION			
1	PROCUREMENT OF APPARATUS AND SUPPLIES	3 6 1	13
2	CALIBRATION OF APPARATUS	3 6 2	7
3	PRESAMPLING OPERATIONS	3 6 3	9
4	ON-SITE MEASUREMENTS	3 6 4	11
5	POSTSAMPLING OPERATIONS	3 6 5	14
6	CALCULATIONS	3 6 6	6
7	MAINTENANCE	3 6 7	2
8.	AUDITING PROCEDURE	3 6 8	8
9	RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY	3 6 9	1
10	REFERENCE METHOD	3 6 10	3
11	REFERENCES	3 6 11	2
12	DATA FORMS	3 6 12	16



Section No. 3.7
Revision No. 0
Date May 1, 1979
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Section 3 7

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

OUTLINE

Section		Documentation	Number of Pages
SUMMARY		3 7	2
METHOD HIGHLIGHTS		3 7	10
METHOD DESCRIPTION			
1	PROCUREMENT OF APPARATUS AND SUPPLIES	3 7 1	13
2	CALIBRATION OF APPARATUS	3 7 2	20
3	PRESAMPLING OPERATIONS	3 7 3	7
4	ON-SITE MEASUREMENTS	3 7 4	18
5	POSTSAMPLING OPERATIONS	3 7 5	17
6	CALCULATIONS	3 7 6	10
7	MAINTENANCE	3 7 7	3
8	AUDITING PROCEDURE	3 7 8	7
9	RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY	3 7 9	1
10	REFERENCE METHOD	3 7 10	4
11	REFERENCES	3 7 11	1
12	DATA FORMS	3 7 12	20

Volume III Format

Each of these sections (e.g., 3.1.5) contains an important summary sheet called the *Activity Matrix*. The activity matrix for the procedure discussed (calibration, on-site measurement, etc.) provides a readily referenced table of the quality assurance activities, acceptance limits, and action requirements.

In the section for each method, Part 10 contains the promulgated reference method as it appears in the Code of Federal Regulations, Part 11 contains a list of references, and Part 12 contains a compilation of clean data forms which can be reproduced and used by the testing team.

You have completed your reading for Assignment 3. If you are satisfied that you understand this material, go on to Reading Assignment 4. It begins on the following page.

Reading Guidance—Assignment 4

Continue your reading of Volume III. To review some general aspects of source testing, read the first four parts of Section 3.0, including:

Summary
3.0.1 Planning the Test Program
3.0.2 General Factors Involved in Stationary Source Testing
3.0.3 Chain-of-Custody Procedure for Source Sampling

This reading assignment incorporates a review of the purposes and the planning of a source test. This part of Volume III also contains a discussion of a number of general factors involved in source testing.

The discussion and check-off sheets of Section 3.0.1, pages 1 through 8, provide information which when used, may help the tester avoid problems when conducting the actual test.

Note that EPA Reference Method 1 appears in Section 3.0.1, pages 8 through 19, as part of the discussion on planning the test program. Since site selection is fairly general for all of the reference methods, the authors of the handbook felt that it would more appropriately be discussed here than separately.

Note that in Figure 1.4 (page 9 of 19) the *minimum number of traverse points* are different for velocity measurements and particulate matter measurements. Recent studies have determined that for velocity measurements, a greater number of sampling points do not necessarily give greater reproducibility or accuracy. For this reason fewer traverse points are specified for velocity measurements.

Note: In Figure 1.4, the graphs for nonparticulate traverses were mislabeled on the May 1, 1979 Revision 0. The upper dashed line is for stack diameters greater than 0.61 m (24 in.). The lower is for stack diameters from 0.30 to 0.61 m (12-24 in.).

Since an even matrix arrangement of traverse points will give more representative data, the *balanced matrix* approach for rectangular ducts was incorporated into the August 18, 1977 revisions to the reference methods.

Measuring gases or particulate matter where cyclonic flow is present is indeed a problem. Paragraph 1.2.3, on page 14 of Section 3.0.1, tells how to detect cyclonic flow. If cyclonic flow is present, you should try to either sample at a better location or straighten the flow. Various methods have been proposed for measuring under such conditions. They do, however, take considerable time to perform, and it has

not been generally shown that the data produced will be representative. The following references can provide additional information on these procedures:

Baker, D.W. and Sayre, C.L. "Decay of Swirling Turbulent Flow of Incompressible Fluids in Long Pipes." *Flow: Its Measurement and Control in Science and Industry, Volume 1, Part 1, Flow Characteristics,* Instrument Society of America, 1974.

Mason, K.W. *Location of the Sampling Nozzle in Tangential Flow.* M.S. Thesis, University of Florida, Gainesville, Florida, 1974.

Chigier, N.A. "Velocity Measurement in Vortex Flows." *Flow: Its Measurement and Control in Science and Industry, Volume 1, Part 1, Flow Characteristics,* Instrument Society of America, 1974.

Lundgren, D.A., Durham, M.D. and Mason, K.W. "Sampling of Tangential Flow Streams." *Am. Ind. Hgy. Assoc. J.,* 39:640, 1978.

Peeler, J.W. "Isokinetic particulate sampling in non-parallel flow systems—cyclonic flow," paper prepared by Entropy Environmentalists, Inc. for the US Environmental Protection Agency, EPA Contract #68-01-4148, 1977, 27 p. (Contact EPA, DSSE for copies).

Phoenix, F.J. and Grove, D.J. "Cyclonic flow—characterization and recommended sampling approaches," paper prepared by Entropy Environmentalists, Inc. for the US Environmental Protection Agency, EPA Contract #68-01-4148, November 1977, 14 p. (Contact EPA, DSSE for copies).

The discussion on *General Factors Involved in Stationary Source Testing* contains a convenient checklist of the sampling tools and equipment that a source test team may need on a job. It is a good compilation to use if your team has not already devised one of its own.

Paragraphs 2.2 and 2.3 of Section 3.0.2 are important reminders to samplers. In the rush of completing a job, or especially when problems crop up, the proper documentation of readings often is neglected. When data forms are used, they should be properly completed. The identification of samples is extremely important and is discussed in more detail in the next section on *Chain of Custody.*

Section 3.0.3 describes chain-of-custody procedures that should be implemented in the testing organization. Such procedures should be set up before the test is done, and each individual involved should be aware of his responsibilities in the chain.

The labels and forms shown in Figures 3.1 through 3.5 are examples recommended for your program. These or similar forms should be used in your organization to properly document the handling of compliance samples.

Chain-of-custody procedures are used to document who did what to a sample, and when and how they did it. The chain of custody starts with the preparation of anything that becomes an integral part of the sample (such as a filter) and continues through to the disposal of the sample.

Note: We will not cover Section 3.0.4 in this course since it is more directly applicable to quality assurance activities related to continuous emission monitoring.

You have completed your reading for Assignment 4. If you are satisfied that you have mastered this material, go on to Reading Assignment 5. It begins on the following page.

Reading Guidance—Assignment 5

> Read Section 3.1 Summary and Method Highlights for Method 2. If you would like a review of other reference methods before proceeding with this correspondence course, review the Summary of Sections 3.3 through 3.7.

This reading assignment is intended as a review of the reference methods. Do read all of Section 3.2, Summary and Method Highlights (9 pages). Each of the other reference method sections is organized in the same fashion. In fact, the Method Highlight sections for the other methods discussed in Volume III all say about the same thing.

But note the format. Section 3.1 briefly gives an overview of what will be discussed about the method. Just after this overview, blank *check sheets* are provided for you to reproduce and use. We recommend that they be used in the test program to properly document procedures. Note that blank *data forms* are provided in Part 12.0, at the end of each discussion of the method. Considerable controversy surrounds the use of the check sheets and data forms in a source test program. Remember that the forms and quality assurance procedures given in Volume III are *recommended.* Their use is not part of a promulgated Federal regulation. An agency should avoid *requiring* that these forms or procedures be used rather than other forms or procedures currently used by established testing firms. Many testing organizations had already adopted their own procedures for assuring quality data before Volume III was published. In many cases, their procedures may be as good as or better than those given in Volume III. In other cases, they may not. Volume III provides the first compilation of a standard set of QA methods for source testing. The procedures given in Volume III are recommended, but do not necessarily preclude equivalent or more stringent techniques.

The numbering scheme for blank data forms is given in Section 3.1.12.

> Read Page 1 of 8, Section 3.1.12 Data Forms.
>
> Review pages 2 of 8 through 8 of 8, Section 3.1.12.

This scheme is consistent for each reference method throughout the manual. Note that not all of the Volume III data forms are included in Section 12 of each method. Others are located in the Method Highlights sections or elsewhere in the text. The form numbers are not printed on those placed in the Method Highlights sections. These are, however, identified and referenced in each of the Data Forms sections (Section 12).

If you would like a quick review of Reference Methods 3 through 8, read the Summary and Method Highlights of Sections 3.3, 3.4, 3.5, 3.6, 3.7, and 3.8.

You have completed your reading for Assignment 5. Do the review exercises for Assignments 3, 4, and 5. They begin on the following page. When you have completed them, check your answers with those given on the page following the review exercises.

Reading Assignments 3, 4, and 5 Review Exercises

1. Super Stack Testers (SST) has been providing manual stack testing services for industrial clients for two years. The company has been doing well and now has a staff of nine people. It normally can send two test teams at a time into the field, and in a pinch can send out three. SST has recently had the opportunity to bid on several sampling jobs, but the jobs require that the company have an established quality assurance program.

   Carl, the president of the company, has decided that separate manuals should be prepared for each function of his testing company. A calibration manual is especially needed. To make a calibration manual from Volume III, which sections would you abstract?

   a. 3.1.1, 3.2.1, 3.3.1,...,3.7.1
   b. 3.1.1, 3.1.2, 3.1.3,...,3.1.12
   c. 3.1.2, 3.2.2, 3.3.2,...,3.7.2
   d. 3.1.9, 3.2.9, 3.3.9,...,3.7.9

2. Since Carl's turnover rate for lab technicians is high, he also wants to develop a manual that could be used to assure quality data from his analytical lab. Which sections of Volume III could he abstract to do this?

   a. 3.1.8, 3.2.8, 3.3.8,...,3.7.8
   b. 3.1.12, 3.2.12, 3.3.12,...,3.7.12
   c. 3.5.5, 3.6.5, and 3.7.5
   d. 3.1.5, 3.2.5, 3.3.5,...,3.7.5

3. Mike has just recently joined the staff and is to accompany a team on a job in which Method 7 samples are to be taken. Mike has done Method 7 before, but wants to review the procedures to make sure that he can follow all of them correctly. Which section of Volume III should he review?
   a. 3.5
   b. 3.6
   c. 3.6.4
   d. 3.0

4. What does Section 3.0 of Volume III contain?
   a. a review of how to do Methods 2 through 8
   b. statistical procedures for determining test accuracy
   c. a discussion of how to plan and document a source test
   d. a discussion of quality assurance requirements for continuous emission monitoring

5. What are the responsibilities of the source tester?
   a. to follow standard testing procedures and document each step of the work
   b. to get the data as quickly as possible and get home
   c. to get a sample from the stack, no matter how
   d. to fill in all the check lists and data forms of Volume III

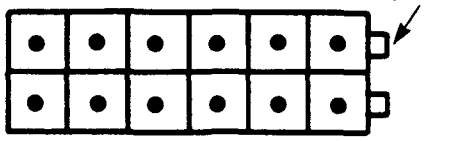
6. When planning a Method 5 source test, which one of the following activities would **not** be performed?
   a. determine applicable emission control regulations
   b. determine sampling site and sampling points
   c. determine process information
   d. determine SRM values

7. Dennis, a team leader of SST, has been having trouble with everyone else borrowing his tools. In order to make sure he has everything he needs on a stack test, he developed a checklist from the tools and equipment listing given in Volume III. Where is this listing?
   a. in Section 3.0.1
   b. in Section 3.0.2
   c. in Section 3.0.3
   d. in Section 3.0.4

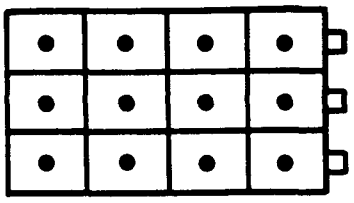
8. Dennis is planning a Method 5 test at Sheer Power Company. Platforms and ports are in a breeching six equivalent duct diameters downstream from a bend and two from the stack. What are the minimum number of points required for a Method 2 traverse and for a Method 5 traverse?
   a. 12 for Method 2; 12 for Method 5
   b. 16 for Method 2; 24 for Method 5
   c. 12 for Method 2; 24 for Method 5
   d. 24 for Method 2; 16 for Method 5

9. Which one of the following depicts a balanced matrix for 12 traverse points in a rectangular duct?

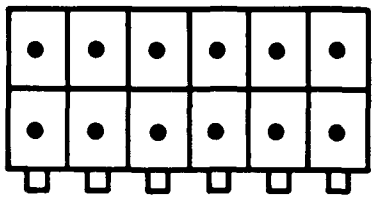
a.



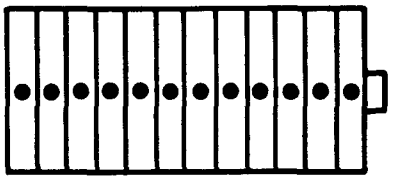
c.



b.



d.



10. When checking for cyclonic flow, no reading will be obtained on a pitot tube manometer if:
    a. the direction of gas flow is perpendicular to the plane of the pitot tube face openings.
    b. the direction of gas flow is parallel to the plane of the pitot tube face openings.

11. True or False? The Federal reference methods require that a degree-indicating level be used when determining the angles of rotation of cyclonic flow.

12. Admissible data in a court case must show that the sample was:
    a. collected properly.
    b. handled properly.
    c. analyzed properly.
    d. all of the above

13. Mike, when labeling the filters for his first Method 5 test with SST, carefully put the run numbers 1 cm from the edge of the filters. After the test, he could identify the filters from runs 1 and 3, but could not find the number on the filter from the second run. What happened?
    a. Acid in the stack erased the number.
    b. He probably inadvertently put in a filter he did not label.
    c. The filter was put in the holder with the number on the face side.
    d. It never could have happened.

14. SST has been using *felt pens* to label their plastic collection bottles, but the bottles are beginning to look pretty messy because the ink does not come off afterwards. Mike has decided to buy some *lick-and-stick* labels to use instead. Will this work?
    a. Yes. It is a great idea because labels that you can lick are inexpensive.
    b. Yes. It is a great idea because you have sample identification and can reuse your bottles, too.
    c. No. Water-soluble labels do not stick well on plastic. He should have bought self-adhesive labels instead.
    d. No. Sample bottles do not have to be identified because you can remember which is which anyway.

15. Which of the following would be included in chain-of-custody procedures?
    a. Method 5 filters
    b. Method 7 absorbing solution
    c. Method 5 probe wash
    d. signature of test team leaders
    e. all of the above
    f. none of the above

16. Consider the format of Section 3.1, which discusses Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate. Where can blank check sheets for the method be found?
    a. in Section 3.1.12
    b. in Section 3.1 after the discussion on Method Highlights
    c. at the end of Sections 3.1.1, 3.1.2, 3.1.3, etc.
    d. at the end of Volume III

17. The activity matrices provide a means of quickly reviewing the operations and quality assurance activities involved in the different phases of a reference method. How many activity matrices are given for Reference Method 2?
    a. 1
    b. 12
    c. 8
    d. 6

18. Give the titles of the forms listed below:
    a. M2-1.2 _____
    b. M2-2.5 _____
    c. M2-8.1 _____
    d. M2-3.2 _____

19. Give the form number for the following data forms:
    a. Method 2 Stack Temperature Sensor Calibration Data Form_____
    b. Method 2 Gas Velocity Data Form_____
    c. Method 2 Pretest Sampling Checks_____
    d. Method 2 Posttest Sampling Checks_____

Answers to
Reading Assignments 3, 4, and 5 Review Exercises

1. a b (c) d
2. a b c (d)
3. a (b) c d
4. a b (c) d
5. (a) b c d
6. a b c (d)
7. a (b) c d
8. a (b) c d
9. a b (c) d
10. a (b)
11. T (F)
12. a b c (d)
13. a b (c) d
14. a b (c) d
15. a b c d (e) f
16. (a) (b) c d
17. a b (c) d
18. a. Procurement Log
    b. Pitot Tube Calibration Data
    c. Method 2—Auditor's Checklist
    d. Pretest Preparations
19. a. M2-2.10
    b. M2-4.1
    c. M2-3.1
    d. M2-5.1

Pretest Operations

Lesson C

Procurement of Equipment

Lesson Goal

The goal of this lesson is for you to be able to purchase source sampling equipment which will perform in the field and will meet the specifications given in Federal regulations.

Lesson Objectives

After completing this lesson, you should be able to:

1. use a procurement log to track the status of purchased equipment.
2. use the activity matrices given at the end of each Subsection 3.__.1 as aides when checking purchased equipment.
3. measure pitot tube misalignment angles for the determination of tube acceptability.
4. leak check a differential pressure gauge.
5. list at least two desirable design qualities of an Orsat analyzer.
6. describe how to check the acceptability of a sample probe.
7. check common laboratory equipment such as impingers, vacuum pumps, thermometers, graduated cylinders, barometers, etc., for acceptability.
8. check the acceptability of acetone and isopropanol used in source sampling.
9. check reagent grades of chemicals used in reference method analytical procedures.

Materials

Assignment 6

- Section 3.1.1, Procurement of Apparatus and Supplies in Section 3.1 Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate

Assignment 7

- Section 3.2.1, Procurement of Apparatus and Supplies in Section 3.2 Method 3—Determination of CO_2, O_2, Excess Air, and Dry Molecular Weight
- Section 3.3.1, Procurement of Apparatus and Supplies in Section 3.3 Method 4—Determination of Moisture in Stack Gases
- Section 3.4.1, Procurement of Apparatus and Supplies in Section 3.4 Method 5—Determination of Particulate Matter from Stationary Sources

Assignment 8

- Section 3.5.1, Procurement of Apparatus and Supplies
  Method 6—Determination of Sulfur Dioxide Emissions from Stationary Sources
- Section 3.6.1, Procurement of Apparatus and Supplies
  Method 7—Determination of Nitrogen Oxide Emissions from Stationary Sources

Reading Guidance—Assignment 6

Begin your reading of Lesson C with Section 3.1.1—Procurement of Apparatus and Supplies, Method 2. Read pages 1 of 15 through 15 of 15.

Figure 1.2 is provided to assist in documenting procurement actions. For those involved in purchasing equipment and air analyzers, an excellent discussion on procurement quality control is given by M.J. Kopecky and B. Rodger (Wisconsin Department of Natural Resources) in *Quality Assurance for Procurement of Air Analyzers,* ASQC Technical Conference Transactions; 1979, Houston, Texas. American Society for Quality Control. 1979:35-40.

Many of the quality assurance activities given in Volume III for the procurement of apparatus and supplies merely deal with inspecting the purchased items after they are received. This section is useful since it points out what you should look for or, in some cases, how you can properly check to see that you received a quality product for your money. Since less experienced testers may not be aware of all of the things to look for, the activity matrices of Table 1.1, etc., can be useful and convenient guides.

Note, on pages 4 and 5 of Section 3.1.1, the many measurements one can make on a pitot tube. Method 2 gives geometric standards for the assignment of a coefficient, $C_p = 0.84$, for a bare Type S pitot tube. The EPA did not, however, specify a measurement technique to determine the various angles and dimensions associated with a Type S tube's construction. Volume III describes a technique discussed by T.R. Clark, W. Mason and P. Reinermann, III (PEDCo. Environmental), Source Evaluation Society Newsletter, Volume V, No. 1, 1980.

Note that the data sheet (Figure 1.7) has the acceptance criteria on it.

In the field, the worst case misalignments would be similar to those shown in Figure 1.4. A 10° misalignment angle should be obvious to the eye, but if there is doubt as to acceptability for smaller angle misalignments, the tube should be measured.

Care should be taken in specifying a $C_p = 0.84$ for a pitot tube attached to a probe or to anything else where aerodynamic interferences may occur. Even a one-inch separation of the tube from the probe may not necessarily eliminate all such interferences. In such a case, the true C_p will normally be lower than 0.84. By assuming a value of 0.84, the velocity and volumetric flow rate will be larger than true, giving a positive bias to the data. For accurate data, the tube-probe assembly should be calibrated in a wind tunnel.

Differential pressure gauges, liq' d-filled manometers, or magnehelics should be checked upon receipt for leaks. Page 12 of Section 3.1.1 offers a procedure for doing this.

Take note of other apparatus used. Liquid-filled bulb thermometers should be checked periodically for liquid separation. Thermocouple systems should be checked frequently for consistency in reading—problems are hard to check visually, but if the LED readout says the ambient air temperature is 150°F, you have a problem.

> You have completed your reading for Assignment 6. Do the review exercises which follow; then check your answers. The correct answers are given on the page following the review exercises.

Reading Assignment 6 Review Exercises

1. Carl had to order a new pitot tube for Method 2 traverses since Mike dropped a meter box on the old one. Carl got a deal from Fast-Weld Products in Hialeah, Florida. Carl filled out Purchase Order SST-238, on June 6, 1981 for pitot tube model #FWB-3951. The catalogue cost was $68.00. Fill in the form on page 3 of 15, Section 3.1.1, with this information.
2. When Carl received the tube on August 29, 1981, Fast-Weld sent along a certification stating that "The Type S pitot model #FWB-3951 No. 68 was calibrated and determined to have a C_p value equal to 0.899 ± 0.001 in." Fast-Weld charged SST $35.00 for the calibration and an additional $19.76 for taxes, shipping, and handling. Log this information into the procurement form.
3. Carl gave the tube to Mike so that he could check the design parameters. Something about the tube looked odd. By using the measurement methods described in Section 3.1.1, he found the following:

   $D_t = 0.373$ in. $\quad \alpha_1 = 6°$ $\quad \alpha_2 = 0°$ $\quad \Theta = 5°$

   $P_A = 0.398$ in. $\quad \gamma = 10°$

   $P_B = 0.402$ in. $\quad \beta_1 = \beta_2 = 0$

   Look at pages 8 of 15 and 9 of 15 of Section 3.1.1 to see how Mike determined the values for α, β, and γ.

   Calculate the value of z.

   a. z = 0.139 in.
   b. z = 0.069 in.
   c. z = 0.070 in.
   d. z = 0.052 in.

4. Calculate the value of w.
   a. $w = 0.698$ in.
   b. $w = 0.139$ in.
   c. $w = 0.070$ in.
   d. $w = 0.052$ in.

5. Based upon the values given and calculated in Exercise 3, what should be done with the probe?
   a. It should be judged acceptable and should be used in the field with the certified value of 0.840.
   b. It should be judged acceptable, but the C_p value should be corrected by subtracting 5% of the certified value.
   c. It should be judged acceptable, but should be recalibrated by SST in a wind tunnel.
   d. It should be returned to the vendor.

6. A 10-inch oil-filled inclined manometer was purchased to determine Δp values across the pitot tube. When it was received, Mike leveled it and zeroed it. He then blew into the positive leg of the manometer and sealed it at a reading of 4.5 in. H_2O. Over a minute, no change in the reading was observed. What conclusions should be made?
   a. The fluid flow is blocked.
   b. The manometer leaks.
   c. The positive side of the manometer is leak free.
   d. The negative side of the manometer is leak free.

Answers to
Reading Assignment 6 Review Exercises

1.

Item description	Quantity	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ordered	Received			
Pitot tube	1	764308	Ace Metal	9-6-79	9-29-79	86.00	In service	
Pitot tube Model FWB-3951	1	SS7-238	Fast-Weld	6-6-81		68.00	Order pending	

2.

Item description	Quantity	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ordered	Received			
Pitot tube	1	764308	Ace Metal	9-6-79	9-29-79	86.00	In service	
Pitot tube Model FWB-3951	1	SS7-238	Fast-Weld	6-6-81	8-29-81	68.00	Rec'd.	No. 68 $C_p = 0.899 \pm 0.001$ in.
Calibration					8-29-81	35.00		
Taxes and handling					8-29-81	19.76		

3. (a) b c d

4. a b (c) d

5. a b c (d)

6. a b (c) d

Reading Guidance—Assignment 7

This reading assignment covers the procurement of apparatus and supplies for Methods 3, 4, and 5. By now, you have either figured out the organization scheme of Volume III or else are saying unkind things about this correspondence course. Instead of carrying the whole of Volume III around with you when doing these lessons, you might want to take Volume III apart and combine all of the procurement sections, all of the on-site measurement sections, etc. This course deals with each of these topics separately. Separating the sections will take about 15 minutes to do, and it will save you time because it will eliminate the need for you to continually leaf through the volume.

Of course, when you are finished with all of the lessons, you can recombine the sections and have the handbook organized as it was originally.

> Continue your reading with Section 3.2.1—Procurement of Apparatus and Supplies, Method 3. Read pages 1 of 15 through 15 of 15.

In Section 3.2.1 on Procurement of Apparatus and Supplies for EPA Method 3, pay primary attention to the discussion of the Orsat analyzer, starting with paragraph 1.3.1 on page 8 of 15. In particular, note the *Desirable Design Qualities* given on page 11 of 15 and the method for leak-checking a new apparatus, given on page 10 of 15.

You might often have wondered what is in the various solutions of the Orsat analyzer. Volume III provides this information for you. The discussion on Orsat reagents is given on page 12 of 15 in Section 3.2.1.

You should note that the Orsat method of determining O_2 and CO_2 is the EPA Reference Method. Alternate methods, such as the Fyrite or continuous O_2 or CO_2 analyzers may be used to determine molecular weight. However, when using O_2 or CO_2 measurements for reporting emissions in units of the standard (i.e., lbs/10^6 Btu heat input for combustion sources), Orsat data is used. Other methods, however, may be used for this purpose if approval is first obtained from the administrator.

> Continue your reading with Section 3.3.1—Procurement of Apparatus and Supplies, Method 4. Read pages 1 of 9 through 9 of 9.

Section 3.3.1 covers the apparatus that is needed for EPA Reference Method 4—Determination of Moisture in Stack Gases. By now, you should have noticed the organization in the procurement sections 3.__.1. First, a schematic of the sampling train is discussed, then procurement logs are discussed. The section proceeds with a discussion of individual items and tells exactly what equipment is needed to do the reference method. Descriptions of pumps, thermometers, manometers, etc., are generally the same throughout the reference method discussions, so if you thoroughly read about this apparatus once, you can just skim over these parts in subsequent reading assignments.

Carefully read the discussion of the probe. Since you must have a heated probe for the moisture determination, special care should be taken to inspect it for electrical continuity and for leaks.

Note the method of checking a Greenburg-Smith impinger for leaks. It is given on page 5 of 9 in Section 3.3.1.

Paragraph 1.1.9 on page 7 of 9 in Section 3.3.1 refers to the measurement of moisture in saturated gas streams. The exhaust gas from wet scrubbers or certain industrial processes may be saturated with water vapor and may even contain entrained water droplets. In such cases, the condensation technique of Method 4 is not applicable. Instead, one merely measures the stack gas temperature and looks up the moisture percentage on a psychometric chart or in saturation vapor pressure tables. It is recommended, however, that the temperature sensor used have an accuracy of ±1°C (±2°F) when applying this method. This is in contrast to an accuracy requirement of 1.5% of the minimum absolute stack temperature for normal stack temperature measurements. At 40°C (100°F), a 1.5% accuracy of the absolute temperature would give a range of ±8.4%, and this would lead to greater ambiguity in reading the saturation vapor pressure tables.

Obtaining a temperature sensor with an accuracy within ±1°C (2°F) should not be too difficult since the temperature range for this technique will generally be below 100°C (212°F).

> Continue your reading with Section 3.4.1—Procurement of Apparatus and Supplies, Method 5. Read pages 1 of 15 through 15 of 15.

Section 3.4.1 covers the procurement of apparatus and supplies for EPA Reference Method 5. You have had to read a lot of material to get to this point, but this may be the most important section for you if you are planning to purchase a Method 5 sampling train. Ten years ago, many people used to construct their own Method 5 trains. Now, over half a dozen commercial firms sell sampling equipment. Although the catalogue prices may seem high, it is usually cost-effective, just in terms of labor hours, to purchase commercial equipment. You can consult trade journals such as *Pollution Equipment News, Journal of the Air Pollution Control Association,* and *Pollution Engineering* for the names of vendors of this equipment.

First, obtain the vendor literature on your instrumentation. Then cross-check the equipment specifications against the QA requirements given in Section 3.4.1. Since most commercial systems meet these requirements, you might consider other factors, such as ease of handling and ease of repair. Ask around and see what other stack testers are using and what they like and do not like about different types of apparatus. The more information you have, the better decision you will be able to make. Refer to the article by Kopecky, discussed on page 107 of this course, for further guidelines on procurement quality control.

The probe discussion is much the same as that given for Method 4, but do read the probe nozzle discussion (page 5 of 15, Section 3.4.1) carefully.

Carefully read Section 1.1.8 on page 7 of 15. Note that the metering system should be checked for leaks upon receipt.

Sections 1.2, 1.3, and 1.4 cover the supplies that will be needed for performing a Method 5 test. The recommendations are based upon experience with sampling under field conditions.

You have completed your reading for Assignment 7. Do the review exercises which follow; then check your answers. The correct answers are given on the page following the review exercises.

Reading Assignment 7 Review Exercises

1. Which one of the following would **not** be a proper criterion for selecting a sampling probe?
   a. The probe material should be inert to the stack gas constituents.
   b. The probe should be resistant to temperature effects at sampling conditions.
   c. The probe should provide for some means of filtering out particulate matter.
   d. The probe should be traceable to an NBS standard probe.
2. What is normally used to reduce the pulsation effect of diaphragm pumps on rate meters and volumetric flow meters?
   a. surge tanks
   b. baffles
   c. glass wool
   d. slider valves

3. At what pressure should a Tedlar® bag used in Method 3 be leak checked?
   a. 5 to 10 cm (2 to 4 in.) vacuum
   b. 5 to 10 cm (2 to 4 in.) H_2O
   c. 15 psi (1 atm)
   d. 1 cm (0.39 in.) H_2O

4. List the reagents used for the three absorbing solutions in an Orsat analyzer.
   a. CO_2 _____
   b. O_2 _____
   c. CO _____

5. Why should the volume reference mark be on the capillary tube of an Orsat analyzer and not on the larger-diameter glass burette?
   a. Precision in reading is increased if it is on the capillary.
   b. Accuracy in reading is increased if it is on the capillary.
   c. both of the above
   d. none of the above

6. Bob, a member of SST's test team, was leak checking a new Orsat analyzer. After displacing the burette liquid to obtain a reading of 25 ml, he closed the manifold valve and waited 4 minutes. What change in the meniscus level would indicate that the apparatus is acceptable?

7. Of what is the confining solution of an Orsat analyzer composed?
   a. water colored with red food dye
   b. water, sodium sulfate, sulfuric acid, and methyl orange
   c. antifreeze

8. Four steps are given in Volume III for checking a probe heating system.
   - connect probe to pump
   - connect heater and turn on
   - turn on pump and adjust flow rate
   - maintain temperature

   Answer the following:
   a. For how long do you warm up the probe? _____
   b. At what flow rate do you pump? _____
   c. What temperature is to be maintained? _____

9. How do you check a standard Greenburg-Smith impinger?
   a. fill inner tube with water and check drain rate
   b. check visually for flaws or cracks
   c. pressurize at two atmospheres and check with Snoop®
   d. both a and b
   e. a, b, and c

10. What value is subtracted from a sea-level barometric reading for each increase of 30 m (100 ft) in altitude?
    a. 2.5 mm Hg
    b. 760 mm Hg
    c. 0.76 mm Hg
    d. 250 mm Hg

11. What precision is required for a thermometer used to measure the temperature of a saturated stack gas?
    a. ± 1.5% of stack temperature in °C
    b. ± 1.5% of stack temperature in °R
    c. ± 2% of stack temperature in °C
    d. ± 2°F (1°C) of stack temperature

12. What should you do to a nozzle after it is received from the manufacturer?
    a. Bang it on a table to see if it will keep its tolerances.
    b. Measure the nozzle diameter with a ruler to see if you agree with the manufacturer's value.
    c. Engrave it for identification and inventory purposes.
    d. Throw it in the tool box, so you will not forget it for tomorrow's test.

13. Mike ordered filters, for use in the Method 5 train, from Roger's High School Chem Lab Supply Company. Would these be acceptable for Method 5 testing?
    Yes _____
    No _____
    Why or why not?

14. What grade of acetone should be used for cleaning the apparatus used for Method 5 testing?

Answers to
Reading Assignment 7 Review Exercises

1. a b c (d)
2. (a) b c d
3. a (b) c d
4. a. CO_2—KOH or NaOH solution
   b. O_2—pyrogallic acid or chromous chloride solution
   c. CO—cuprous chloride or cuprous sulfate solution
5. a b (c) d
6. A change of ≤0.2 ml
7. a (b) c d
8. a. 2 or 3 minutes
   b. 0.02 m^3/min (0.75 ft^3/m)
   c. 100°C (212°F) minimum
9. a b c (d) e
10. (a) b c d
11. a b c (d)
12. a (b)(c) d
13. No, because they may not give the required collection efficiency and may deteriorate at the elevated sample case temperatures.
14. ACS grade

Reading Guidance—Assignment 8

Reading Assignment 8 covers primarily Methods 6 and 7. The discussion of the sampling equipment is much the same as that given in previous discussions of Methods 3, 4, and 5. Pay close attention to details of the equipment and supplies used in the analytical procedures.

In particular, certain grades of reagents are required in the laboratory analysis. A review of the different types of chemical grades is given below.

Technical or commercial grade. Chemicals labeled technical or commercial grade are of indeterminate quality and should be used only where high purity is not of paramount importance. In general, technical or commercial grade chemicals are not used in analytical work.

Chemically Pure, or C.P. grade. The term, *Chemically Pure,* has little definite meaning. Such reagents are usually more refined than are the technical grades, but no specifications define what is meant by the term. Thus, it is prudent to avoid the use of C.P. reagents in analytical work. If this is not possible, testing the reagent for contaminants of importance and running frequent reagent blanks may be necessary.

U.S.P. grade. U.S.P. chemicals have been found to conform to tolerances specified in the *United States Pharmacopoeia.* The specifications are designed to control the presence of contaminants dangerous to health; thus, chemicals passing U.S.P. tests may still be quite heavily contaminated with impurities that are not physiological hazards.

Reagent grade. For the most part, the analytical chemist uses reagent grade chemicals in his work. These have been tested and found to conform to the minimum specifications set down by the Reagent Chemicals Committee of the American Chemical Society. In addition to meeting these requirements, the results of the analysis are, in some instances, printed on the label. Thus reagent grade chemicals fall into two categories: namely, those that simply pass the tests and those for which the actual results of the tests are additionally supplied.

Primary standard grade. These are substances that are obtainable in extraordinarily pure form. Primary standard grade reagents are available commercially; these have been carefully analyzed and the assay value is printed on the label. An excellent source for primary standard chemicals is the National Bureau of Standards.

Begin by reading Section 3.5.1—Procurement of Apparatus and Supplies, Method 6. Read pages 1 of 15 through 15 of 15.

Note paragraph 1.1.9 on page 7 of 15 in Section 3.5.1. This paragraph presents a method of checking a metering system for leaks. You have not read about this yet, since for Reference Methods 4 and 5, leak checking procedures are discussed in the calibration Sections 3.3.2 and 3.4.2 (Subsections 2.1), respectively. Read carefully the procedure given here so that you can recognize the method when similar descriptions are presented in the larger discussions on calibration procedures.

On page 10 of 15 of Section 3.5.1, a discussion of analytical laboratory supplies begins. Although selecting such supplies may appear straightforward, you should, when doing routine testing, take care to assure that the laboratory and its supplies remain clean and uncontaminated.

The problem of contaminated isopropanol has led to many cases of inconsistent and invalid data. The preparation and testing of blanks is an important practice in any analytical laboratory.

> Continue your reading with the discussion of Method 7 procurement on page 4 of 13 of Section 3.6.1.

Note the specifications for the collection flask.

Also, note the requirements for the evaporating dishes given on page 8 of 13 of Section 3.6.1.

Method 8 is very similar to Method 5. It is not necessary to read Section 3.6.1 at this time unless you have an immediate interest in the procurement of the analytical reagents used in the method.

> You have completed your reading for Assignment 8. Do the review exercises which follow; then check your answers. The correct answers are given on the page following the review exercises.

Reading Assignment 8 Review Exercises

1. Which one of the following is **not** a criterion for rejecting a new dry gas meter?
   a. The meter face is broken.
   b. Readings are less than 3% of that determined with a spirometer.
   c. The meter dial frequently sticks.
   d. The meter is painted green instead of gray.

2. What should be the capacity of the silica gel drying tube used in Method 6?
   a. 5 to 10 g silica gel
   b. 30 to 50 g silica gel
   c. 100 to 150 g silica gel
   d. 6 to 16 g silica gel

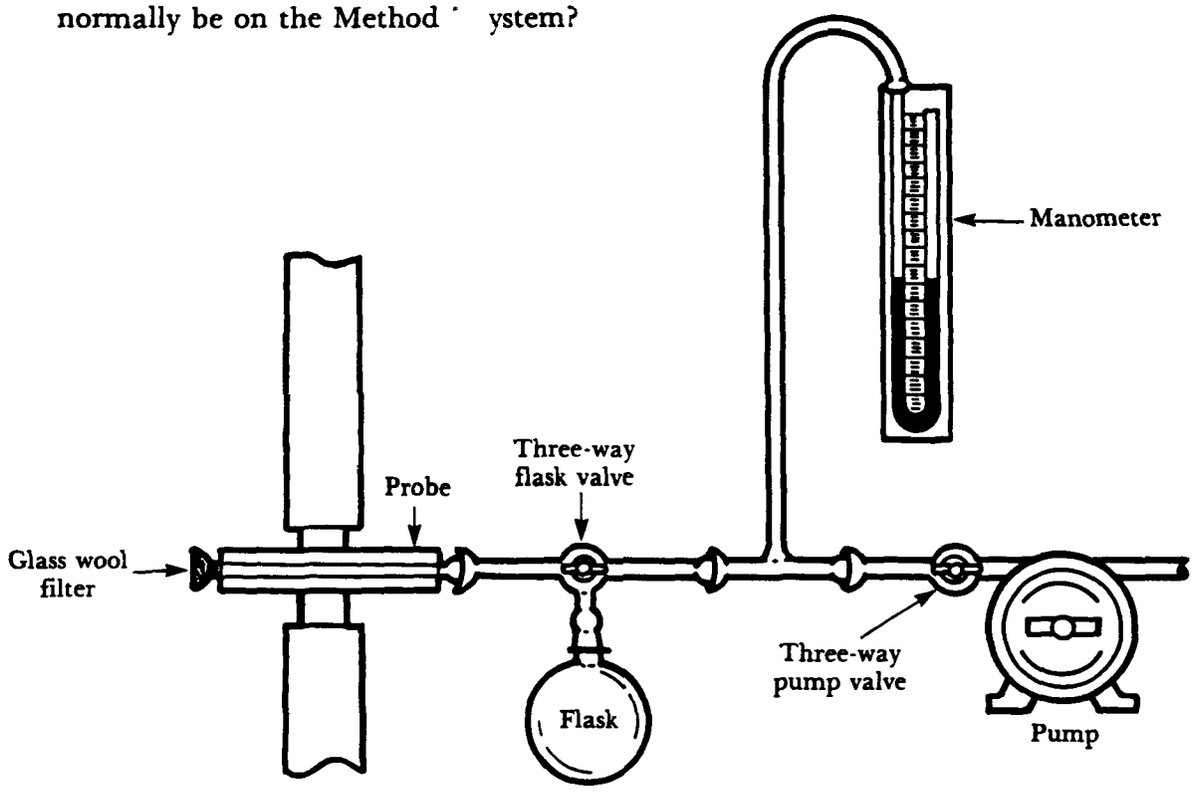
3. On a field test, Mike realized that he had not prepared the isopropanol solution for the Method 6 impingers. He did have a bottle of ACS grade 100% isopropanol, so he quickly made the 80% solution by diluting with tap water. Was this a good thing to do?
   Yes _____
   No _____
   Why or why not?

   If your answer was "no", what would you have done instead?

4. What types of reagents are to be used in Method 6?
   a. ACS grade
   b. PBS grade
   c. NBS grade
   d. C.P. grade

(Review exercises continue on page 120.)

5. The figure below shows a Method 7 train assembly. What is missing that would normally be on the Method ' ystem?



   a. orifice meter
   b. thermometer
   c. flask shield
   d. pitot tube

6. How many volumetric flasks, and what sizes, would have to be ordered for a Method 7 experiment?
   a. one 100-ml flask; two 1000-ml flasks; several 50-ml flasks
   b. ten 100-ml flasks; five 1000-ml flasks
   c. three 1-L flasks

7. What do you need to make phenoldisulfonic acid solution?
   a. phenol, water, and disulfonic acid
   b. phenol and sulfuric acid
   c. sulfone, phenolidene, and distilled water

8. In what wavelength range must a spectrophotometer used in Method 7 be capable of measuring?
   a. 210 to 214 nm
   b. 3.5 to 4.6 μm
   c. 2000 Å to 3000 Å
   d. 400 to 415 nm

Answers to
Reading Assignment 8 Review Exercises

1. a b c (d)
2. a (b) c d
3. Yes_____ No__x__
   Sulfates may be introduced from the tap water.
   Purchase distilled water in a supermarket, or better yet, go to a local university or hospital and ask for some distilled water from a laboratory. A sample of the water should be retained for analysis as a blank.
4. (a) b c d
5. a (b)(c) d
6. (a) b c d
7. a (b) c d
8. a b c (d)

Lesson D

Calibration of Equipment

Lesson Goal

The goal of this lesson is for you to understand the procedures that are to be followed when calibrating equipment used in source sampling and analysis.

Lesson Objectives

After completing this lesson, you should be able to:

1. describe how to set up a pitot tube calibration apparatus.
2. perform the calculations used in reporting the pitot tube calibration coefficient, C_p.
3. tell how to calibrate a thermometer, barometer, and differential pressure gauge.
4. explain the procedure that is to be followed when checking the Orsat apparatus.
5. explain, using a diagram, how to calibrate a dry gas meter with a wet test meter.
6. explain how to check the calibration of a wet test meter.

Materials

Assignment 9

- Section 3.1.2, Calibration of Apparatus in Section 3.1
  Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate
- Section 3.2.2, Calibration of Apparatus in Section 3.2
  Method 3—Determination of CO_2, O_2, Excess Air, and Dry Molecular Weight

Assignment 10

- Section 3.3.2, Calibration of Apparatus in Section 3.3
  Method 4—Determination of Moisture in Stack Gases
- Section 3.5.2, Calibration of Apparatus in Section 3.5
  Method 6—Determination of Sulfur Dioxide Emissions from Stationary Sources

Reading Guidance—Assignment 9

It is very important to use properly calibrated equipment when stack sampling. Lesson D will emphasize the procedures that can be used to calibrate and check the following types of equipment:

1. pitot tubes
2. dry gas meters and wet test meters
3. thermometers, barometers, differential pressure gauges
4. spectrophotometers

The first reading assignment covers the calibration of the pitot tube, a check of the Orsat apparatus, and methods for calibrating ancillary source sampling equipment such as temperature sensors and barometers.

> Begin by reading Section 3.1.2—Calibration of Apparatus, Method 2. Read pages 1 of 21 through 21 of 21.

In Lesson C, you reviewed the design criteria for assigning a C_p value of 0.84 to a pitot tube. Section 3.1.2, pages 1 of 21 to 13 of 21, provides a method for obtaining a laboratory-referenced value for C_p. Considerable experimental work has shown that by placing a pitot tube on a probe-nozzle assembly, the C_p value can decrease. The interference-free criteria of ¾ in. between pitot tube and nozzle may not in all cases guarantee that the C_p will still be equal to 0.84 in this assembly. A review of interference effects for several types of pitot-tube—probe assemblies is given in Williams, J.C. and DeJarnette, F.R., *A Study on the Accuracy of Type-S Pitot Tubes* EPA 600/4-77-030, June 1977.

Note that if the true value of C_p is equal to 0.80 (for example), then when the tube is attached to a probe, if a value of 0.84 is assumed instead, the following error arises when determining the stack gas velocity:

$$v_s = C_p K_p \sqrt{\frac{T_s \Delta p}{P_s M_s}}$$

$$v_s(\text{true}) = 0.80\ K_p \sqrt{\frac{T_s \Delta p}{P_s M_s}}$$

$$v_s(\text{assumed}) = 0.84\ K_p \sqrt{\frac{T_s \Delta p}{P_s M_s}}$$

$$\frac{v_s(\text{true})}{v_s(\text{assumed})} = \frac{0.80}{0.84}$$

$$v_s(\text{true}) = 0.95\ v_s(\text{assumed})$$

The true velocity would therefore be lower than that obtained if the tester had assumed a value of 0.84. This is an example of *positive bias* in stack sampling. If the pollutant emissions were to be reported on a lbs per hour basis, i.e.,

$$\text{pollutant mass rate} = c_s v_s A_s$$

Where: c_s = the concentration of the pollutant
A_s = the area of the stack or duct

then the reported values would be higher than true and could possibly indicate a violation when in fact there was none.

The above illustration was given to underscore the importance of calibration in obtaining accurate test data. It is often worth the effort to recheck calibrations if there is a question about their validity.

Paragraph 2.1.2 shows how you can construct a test setup for calibrating a pitot tube. The major expense is the fan. You should use a fan large enough to reach representative stack velocities (see page 4 of 21—bottom of page).

When performing this calibration, it is very important to properly align the standard and Type S pitot tubes, as discussed on page 6 of 21.

Read the calibration procedure of paragraph 2.1.3 carefully. Note that standard and Type S tube readings are taken alternately.

Paragraph 2.1.4 points out problems that can occur if you attempt to calibrate a probe assembly if the test duct area is small relative to the probe. Correction factors for blockage effects are given in Figure 2.9 on page 14 of 21.

Subsection 2.2 gives simple and straightforward procedures for calibrating temperature sensors. Keep in mind that when your temperature readings are used in sampling calculations, they must be converted into absolute temperatures. Small errors in degrees Celsius or degrees Fahrenheit may be relatively unimportant once values are converted to kelvin or degrees Rankine.

Turn to Section 3.2.2, Calibration of Apparatus used for Method 3—Determination of CO_2, O_2, Excess Air, and Dry Molecular Weight. Read Subsection 2.1 which gives a procedure for checking the performance of both the operator and the Orsat analyzer.

You have completed your reading for Assignment 9. Do the review exercises which follow; then check your answers. The correct answers are given on the page following the review exercises.

Reading Assignment 9 Review Exercises

1. Carl obtained the following data in the calibration of Fast-Weld's No. 68 Type S pitot tube in a wind tunnel. Calculate the $C_{p(s)}$ value where $C_{p(std)} = 0.99$ (use form M2-2.5 in Section 3.1.12).

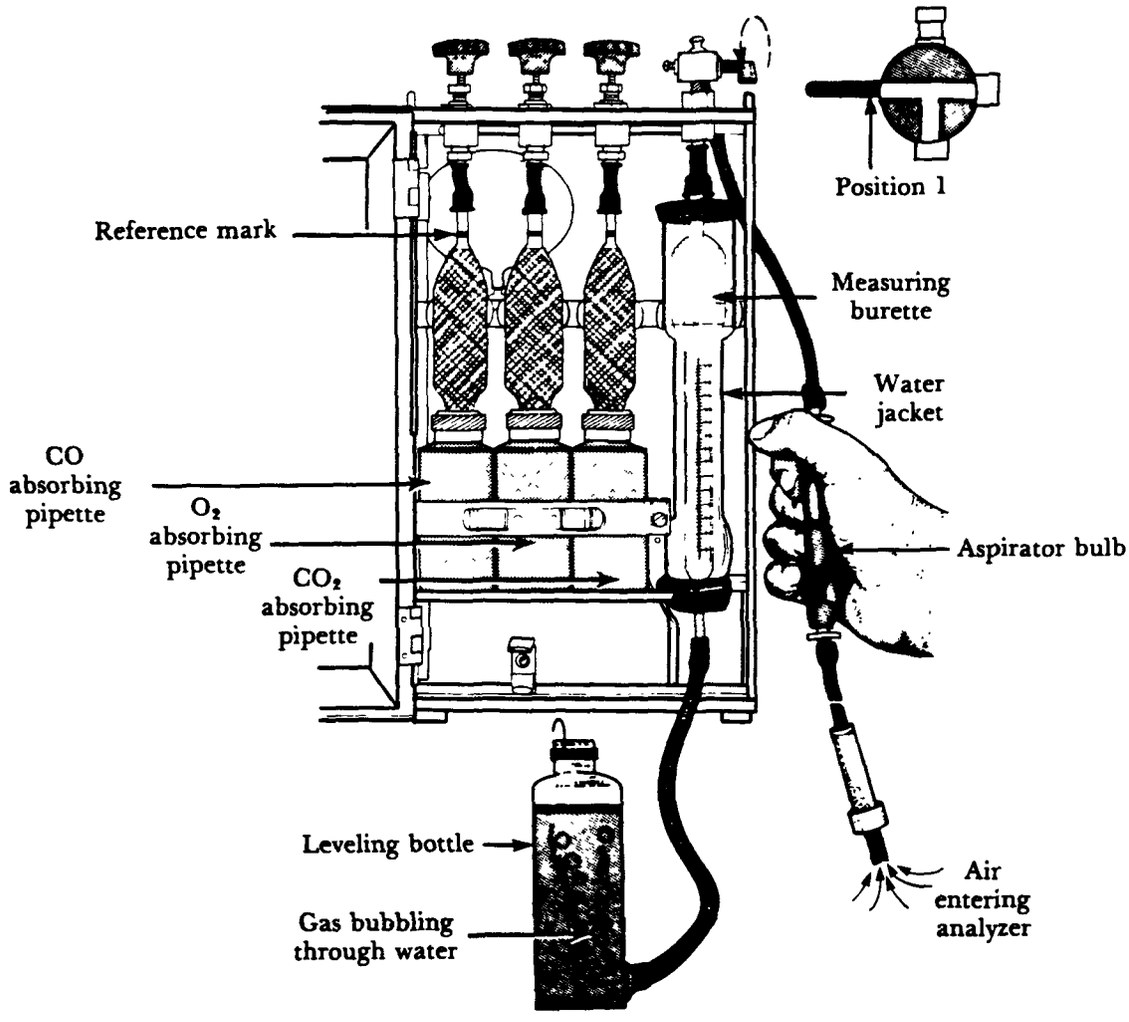
A-side calibration	
Δp_{std}	Δp_s
0.072 in. H_2O	0.116 in. H_2O
0.065	0.120
0.070	0.118

B-side calibration	
Δp_{std}	Δp_s
0.060 in. H_2O	0.120 in. H_2O
0.060	0.122
0.065	0.118

2. Calculate the average DEV between $C_{p(s)}$ and $\overline{C}_p$ for both the A and the B sides of the pitot tube. Does the average deviation come within the Volume III criteria?
3. From the data in 1, calculate $\overline{C}_p(A) - \overline{C}_p(B)$. Does the value meet the Volume III requirements?
4. It is highly recommended in Volume III that an ASTM reference thermometer be purchased for thermometer and thermocouple calibrations. What would be the number of an ASTM thermometer that could be used for such calibrations?

   _____ or _____
5. The calibration of temperature sensors is a relatively simple task, but Bob often neglected it. How would he compare a thermocouple's response to that of an ASTM reference thermometer at a temperature of 230 °C?
   a. use boiling water
   b. use crushed ice in Dewar flask
   c. calibrate it at the stack
   d. use cooking oil
6. Orsat values that are high compared to a standard indicate:
   a. leaking valves.
   b. spent absorbing reagent.
   c. poor operator technique.
   d. a worsening greenhouse effect.

7. In the analysis being performed in the figure below, what should the average O_2 value for three replicates be?



a. $20.9 \pm 2\%$
b. $20.8 \pm 0.7\%$
c. $21.5 \pm 0.9\%$
d. $20.1 \pm 0.1\%$.

Answers to
Reading Assignment 9 Review Exercises

1, 2, and 3.

Pitot tube calibration data

Calibration pitot tube: type _____ size (OD) _____ ID number _____

Type S pitot tube ID number 68 $C_{p(std)}$ = 0.99

Calibration: date _____ performed by Carl

A-side calibration			
Δp_{std}, cm (in.) H_2O	Δp_s, cm (in.) H_2O	$C_{p(S)}$[a]	DEV[b]
0.072 in.	0.116 in.	0.780	0.023
0.065	0.120	0.729	0.028
0.070	0.118	0.762	0.005
	Average	0.757	0.019

B-side calibration			
Δp_{std}, cm (in.) H_2O	Δp_s, cm (in.) H_2O	$C_{p(S)}$[a]	DEV[b]
0.060 in.	0.120 in.	0.700	0.010
0.060	0.122	0.694	0.016
0.065	0.118	0.735	0.025
	Average	0.710	0.017

[a] $C_{p(S)} = C_{p(std)} \frac{\Delta p_{std}}{\Delta p_s}$ = _____

[b] $DEV = C_{p(S)} - \overline{C}_p$ (must be ≤ 0.01)

$\overline{C}_p(A) - \overline{C}_p(B) = 0.047$ (must be ≤ 0.01).

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The average values of DEV for both the A and the B sides exceed the recommended value of 0.01. The value of $\overline{C}_p(A) - \overline{C}_p(B) = 0.047$, exceeds the recommended value of 0.01. The calibration requirements for the pitot tube are not met and the pitot tube should not be used.

4. ASTM 63C or ASTM 63F
5. a b c (d)
6. a b (c) d
7. a (b) c d

Reading Guidance—Assignment 10

EPA Methods 4, 5, and 6 use dry gas meters for determining volumetric flow through the sampling train. The discussion on the calibration of the dry gas meter is the same for Methods 4 and 5 (Sections 3.3.2 and 3.4.2 in Volume 3). Section 3.5.2 contains some additional information concerning the calibration of the wet test meter.

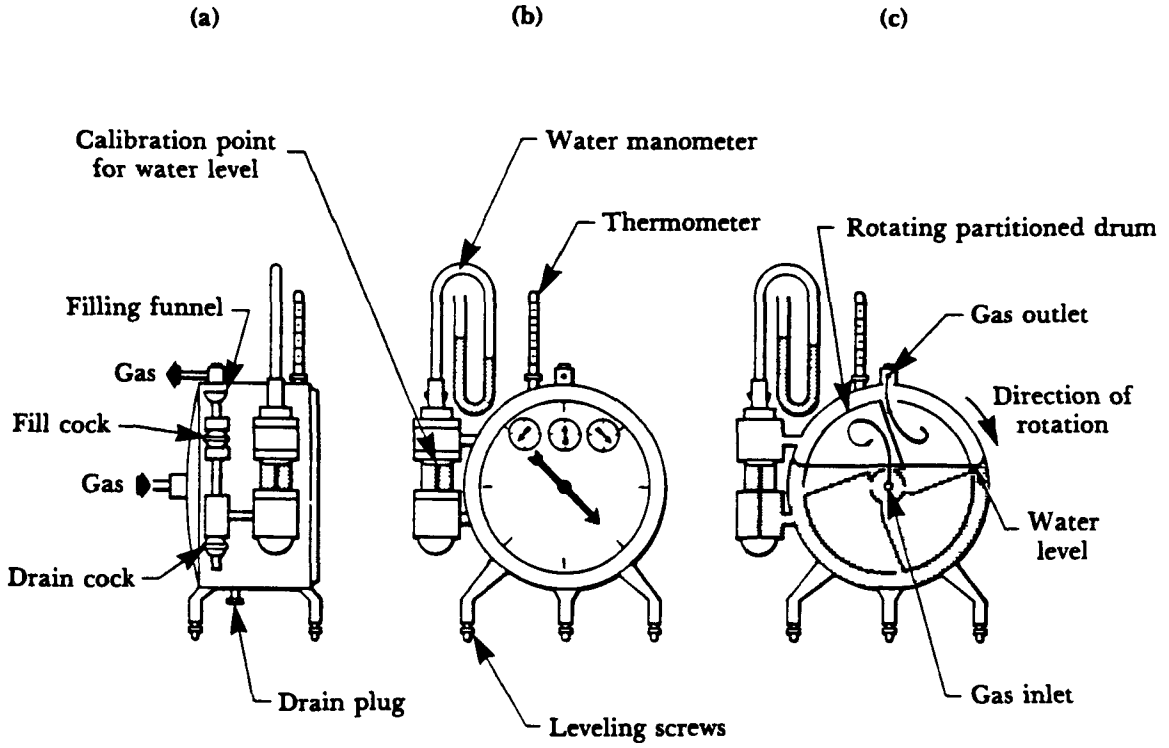
The wet test meter is an intermediate standard commonly used to calibrate dry gas meters. Wet test meters are used for this purpose because of their high accuracy (better than ±1%). However, because of their bulk, weight, and equilibration requirements, they are seldom used outside a laboratory setting.

Since it is often difficult to locate information about wet test meters, a brief review of their operation is given here (abstracted from APTI Course 435 *Atmospheric Sampling—Student Manual* EPA 450/2-80-004, September 1980).

Wet Test Meter

The wet test meter consists of a series of inverted buckets or traps mounted radially around a shaft and partially immersed in water (c). The location of the entry and exit gas ports is such that the entering gas fills a bucket, displacing the water and causing the shaft to rotate because of the lifting action of the bucket full of air. The entrapped air is released at the upper portion of the rotation and the bucket again fills with water. As it turns, the drum rotates index pointers that register the volume of gas passed through the meter (b).

After the meter is leveled, the proper water level is achieved by using the filling funnel, fill cock, and drain cock (a) to bring the meniscus of the water in touch with the tip of the calibration index point. The calibration gas should be passed through the meter for one hour to saturate the water with the gas. The water in the meter should be at the same temperature as the surrounding atmosphere. If any water is added, sufficient time must be allowed for complete equilibration.



If a wet test meter is used to measure a dry gas stream, a significant error is introduced if the measured volume is not corrected to dry conditions. The correction can be made by using the following expression:

$$V_c = V_{meas} \frac{P_b - P_w}{P_b}$$

Where: P_b = atmospheric pressure
P_w = vapor pressure of water at room temperature
V_c = corrected volume at room temperature
V_{meas} = measured volume at room temperature

It may be easier, however, to first saturate the test gas stream by passing it through a bubbler before it enters the wet test meter.

Begin by reading Section 3.3.2—Calibration of Apparatus, Method 4. Read pages 1 of 19 through 19 of 19.

Four methods of checking the initial calibration of a wet test meter are given in paragraph 2.1.1 on page 1 of 19, Section 3.3.2. The second method suggests using any primary air or liquid displacement method. A spirometer, mercury-sealed piston, or simple displacement bottle all can be used for this purpose. It may be difficult, though, to find a large enough displacement bottle to handle the volume requirements of the wet test meter. The calibration section for Method 6, given in Section 3.5.2, gives a step-by-step procedure for the displacement bottle method.

Read paragraph 2.1.1 of Section 3.5.2 at this point, before proceeding with the actual calibration procedures for the sample train. We would like to round out the discussion of wet test meters by including that part of the Method 6 discussion here.

Calibrating the Sample Meter System

Now read paragraph 2.1.2, pages 2 of 19 through 12 of 19 of Section 3.3.2—Calibration of Apparatus, Method 4.

The leak-check procedures are, of course, important. Both positive pressure and vacuum leak checks should be performed before proceeding with the calibration.

Note that a needle valve will be needed for this calibration method.

Note that the forms of Figure 2.3A provide for taking data from ΔH values of 0.5 to 4.0 in. H_2O. Higher values of ΔH are not routinely encountered in source testing so a calibration ranging to 4.0 in. H_2O will generally be adequate.

Note on page 11 of 19 of Section 3.3.2, that from steps 7 and 8, each calibration factor must be within $\pm 2\%$ of the average Y value. The average Y value does not necessarily have to be equal to 1.0.

Note that metric data sheets are provided on page 9 of 19 of Section 3.3.2.

A post-test calibration check is given on page 11 of 19 of Section 3.3.2.

Temperature gauges: Briefly review the discussion of calibration techniques for thermometers used in Method 4. Much of this material is similar to that reviewed in the discussion of temperature sensors for Method 2.

Continue your reading with Section 3.4.2—Calibration of Apparatus for Method 5.

This section contains almost identical information to that given in the calibration section for Method 4. It is not necessary to reread all the material, but do note on pages 18 and 19 of 22, Section 3.4.2, calibration procedures specific to Method 5 (heater and nozzle calibrations).

You have completed your reading for Assignment 10. Do the review exercises which follow; then check your answers. The correct answers are given on the page following the review exercises.

Reading Assignment 10 Review Exercises

1. In performing a positive leak check of the *metering* system of a Method 5 train before calibrating the dry gas meter, which one of the following preparatory steps **would not** be done?
   a. plug downstream tap of orifice meter
   b. plug inlet to vacuum pump
   c. vent positive side of inclined manometer attached to the orifice meter
   d. place a one-hole stopper with attached tubing to end of orifice meter

2. How does one perform a negative leak check on the metering system?
   a. by observing the leakage rate on the DGM at a vacuum of 75 mm Hg
   b. by observing the leakage rate on the orifice meter at a vacuum of 75 mm Hg
   c. by observing the leakage rate on the vacuum gauge at a vacuum of 75 mm Hg

3. When calibrating a Method 5 metering system against a wet test meter, Carl simultaneously calibrated the dry gas meter and orifice meter. When performing the calibration, what should Carl have set and what should he have observed?
   a. He should have set V_d and observed ΔH and V_w.
   b. He should have set ΔH and observed V_d and V_w.
   c. He should have set ΔH and V_w and observed V_d.
   d. He should have set V_w and observed ΔH and V_d.

4. When calibrating a dry gas meter with a wet test meter the inlet air should be:
   a. at projected stack temperature.
   b. saturated.
   c. the same molecular weight as the projected stack gas.
   d. free of oxygen.

5. Calculate a value for Y_i, given the following data:

$V_w = 5$ ft^3	$V_d = 5.036$ ft^3
$P_b = 28.63$ mm Hg	$\Delta H = 0.5$ in. H_2O
$t_d = 74$ °F	$t_w = 73$ °F

   $Y_i =$ _____.

6. In order to calculate the value of $\Delta H_@$ for an orifice meter, what additional information is needed beyond that given in exercise 5?
   a. K_m
   b. Θ
   c. Y_i
   d. P_s

7. Where in Volume III can you find a detailed procedure for calibrating a *wet* test meter?
   a. Section 3.3.2
   b. Section 3.4.2
   c. Section 3.5.2
   d. Section 3.6.2

8. Calibration values for Method 5 probe nozzles are obtained by:
   a taking vendor certified values.
   b. measuring with a ruler.
   c. measuring with a micrometer caliper.
   d. projecting the nozzle area on graph paper and weighing the cut-out projected area.

Answers to
Reading Assignment 10 Review Exercises

1. a b (c) d
2. (a) b c
3. a b (c) d
4. a (b) c d
5. $$Y_i = \frac{V_w P_b (t_d + 460)}{V_d \left(P_b + \frac{\Delta H}{13.6}\right)(t_w + 460)} = \frac{5 \times 28.63 \times (74 + 460)}{5.036 \times \left(28.63 + \frac{0.5}{13.6}\right)(73 + 460)}$$

   $$= \frac{5 \times 28.63 \times 534}{5.036 \times 28.67 \times 533} = 0.993$$
6. a (b) c d
7. a b (c) d
8. a b (c) d

You have now completed the material for **Quiz 1.**

Take **Quiz 1** under the direction of your test supervisor. (See page 5 of this guidebook for more detailed instructions.)

Lesson E

Presampling Operations

Lesson Goal

The goal of this lesson is for you to be able to properly prepare and pack calibrated equipment and supplies before conducting a source test.

Lesson Objectives

After completing this lesson, you should be able to:

1. inspect a Type S pitot tube before taking it into the field.
2. describe the procedures for checking the Orsat analyzer before a field test.
3. describe proper packing methods for source sampling equipment, including probes, glassware, and chemicals.
4. consider the utility of using a detailed packing list.
5. describe the procedure for checking filters used in Method 5.
6. effectively use an activity matrix for presampling preparation.

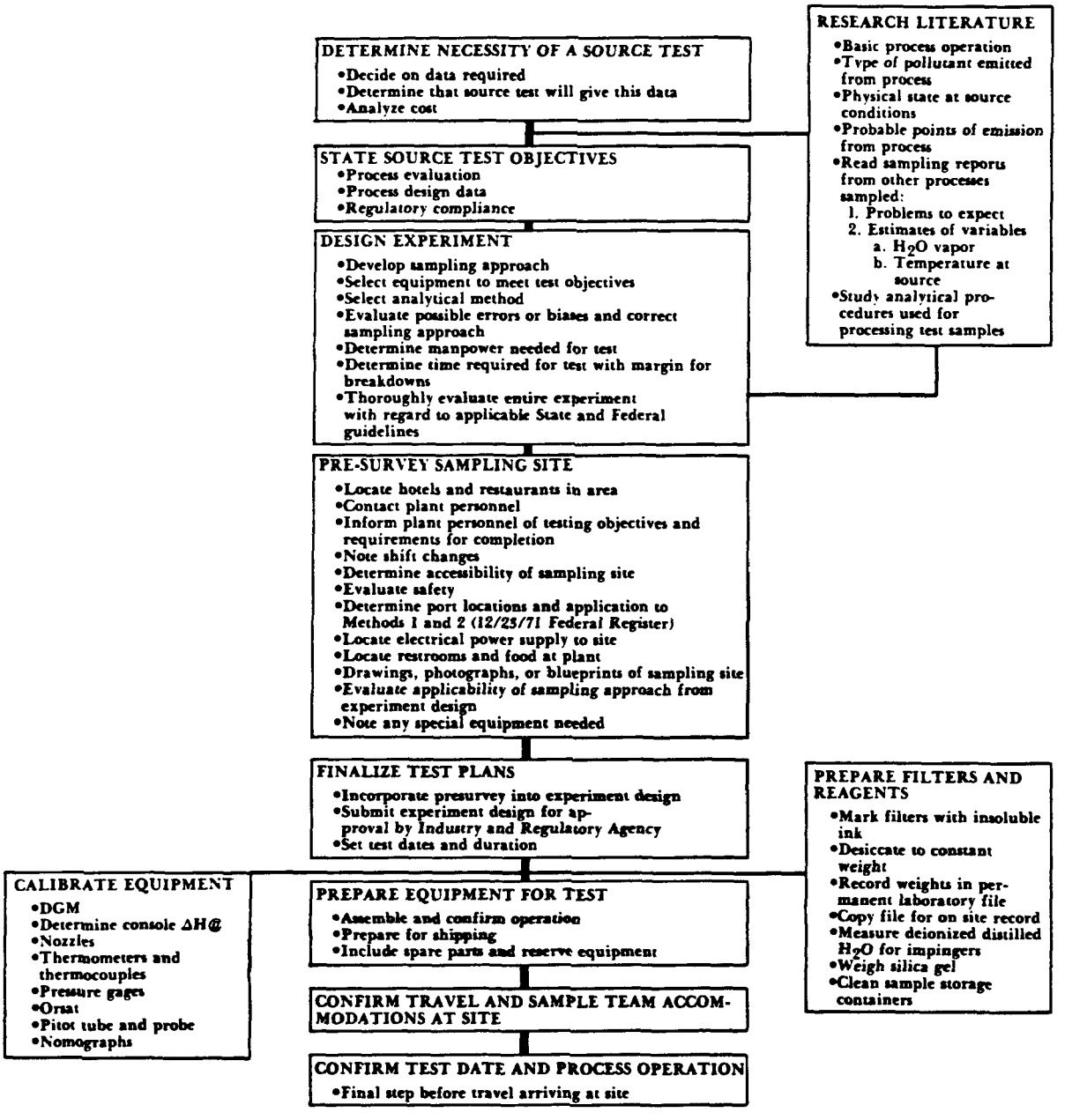
Materials

Assignment 11

- Section 3.1.3—Presampling Operations in Section 3.1, Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate
- Section 3.2.3—Presampling Operations in Section 3.2, Method 3—Determination of CO_2, O_2, Excess Air, and Dry Molecular Weight
- Section 3.3.3—Presampling Operations in Section 3.3, Method 4—Determination of Moisture in Stack Gases
- Section 3.4.3—Presampling Operations in Section 3.4, Method 5—Determination of Particulate Matter from Stationary Sources
- Activity matrix given on page 5 of 6, Section 3.5.3 in Section 3.5—Presampling Operations, Method 6—Determination of Sulfur Dioxide Emissions from Stationary Sources
- Activity matrix given on page 8 of 9, Section 3.6.3 in Section 3.6—Presampling Operations, Method 7—Determination of Nitrogen Oxide Emissions from Stationary Sources

Reading Guidance—Assignment 11

The figure below is taken from APTI Course 450 *Source Sampling for Particulate Pollutants—Student Manual,* Aldina, G.J. and Jahnke, J.A., December 1979 EPA 450/2-79-006. It provides an overview of much of what we have studied in Volume III so far.



Remember that Section 3.0 (Sections 3.0.1 and 3.0.2) in the beginning of Volume III discusses planning the test program and general factors involved in source testing. A number of points discussed there bear on pretest preparation discussions for the individual methods. You may wish to go back and briefly review the contents of Section 3.0 before proceeding with this reading assignment.

> Read the Presampling Operations sections for Methods 2 through 5. Since much of the material repeats, just review the Activity Matrices for Presampling Operations for Methods 6 and 7. Begin your reading with Section 3.1.3—Presampling Operations, Method 2. Read pages 1 of 7 through 3 of 7.

Note the various pretest preparation check forms provided for each of the reference methods. The preparedness of a test team can be evaluated by noting how completely these forms are filled out.

> Continue your reading with Sections 3.2.3, 3.3.3, and 3.4.3—Presampling Operations for Methods 3, 4, and 5.

Paragraph 3.1.3 on page 3 of 6 of Section 3.2.3 is important. Before going to the field, care should be taken that the absorbing solutions have not deteriorated.

The presampling operations section for Method 5 contains a packing list starting on page 3 of 20, Section 3.4.3. This listing points out the many tools and spare parts that a test team may need under the actual conditions at a source. The listing has been derived from the experience of many stack testers over a period of several years. It is inclusive for the reference methods performed by the professional stack tester. Although all of the materials listed will not be needed for any one test, the pack sheet allows you to consider taking the items which might otherwise be forgotten.

Carefully read the discussion on Method 5 filters, on page 16 of 20, Section 3.4.3.

> Since you have already read information on presampling operations for probes, glassware, etc., for Methods 6 and 7, just review the activity matrices. For Method 6, the activity matrix is given on page 5 of 6, Section 3.5.3, Table 3.1. For Method 7, the activity matrix is given on page 8 of 9, Section 3.6.3, Table 3.1.

If you are not familiar with the procedures for the preparation of reagents used in Methods 6 and 7 sampling, read Subsection 3.2 *Reagents* on page 3 of 6, Section 3.5.3 for Method 6 preparations and Subsection 3.2 *Reagents* on page 4 of 9, Section 3.6.3.

You have completed your reading for Assignment 11. Do the review exercises which follow; then check your answers. The correct answers are given on the page following the review exercises.

Reading Assignment 11 Review Exercises

1. An inspection of a Type S pitot tube before a test primarily incorporates:
   a. a visual check for damage and misalignment.
   b. the measurement of P_A, P_B, and A.
   c. the measurement of γ, θ, and w.
   d. a check of vendor's C_p certification form.
2. True or False? Since pitot tubes and thermocouples are not fragile like glass probes are, they can just be thrown in back of the van when going out on a test.
3. List the four steps that should be taken in preparing an Orsat analyzer before use.
   a. _____
   b. _____
   c. _____
   d. _____
4. SST lucked out and got a job in Hawaii. How should Mike ship the Orsat?
   a. He should put it assembled in a cardboard box and stuff paper around it.
   b. He should ship it by freighter.
   c. He should disassemble it and pack each item individually into a rigid container.
   d. He should carry it (assembled) with him and put it under the seat of the airplane.

5. Indicate whether or not each of the following items is noted in the packing list in Section 3.4.3 (Method 5)?

	Yes	No	
a.	____	____	first aid kit
b.	____	____	ice chest
c.	____	____	tool box
d.	____	____	calculator
e.	____	____	glass wool
f.	____	____	duct tape
g.	____	____	Method 5 meter box
h.	____	____	hard hat
i.	____	____	500-ml graduated cylinder

6. The procedure for checking Method 5 filters is to:
   a. perform an ASTM burst test on the filter material.
   b. determine the tensile strength of 1 in. × 5 in. strips.
   c. visually check each filter for flaws or leaks.
   d. use them and see if they work.

7. True or False? It is **not** necessary to desiccate filters before a test since they will be measured at ambient conditions anyway.

8. Turn to page 5 of 6, Section 3.5.3—The Activity Matrix for Presampling Operations, Method 6. At this stage of the source test, what is the predominant activity if acceptance limits are not met?
   a. repair or replace
   b. recalibrate
   c. return to manufacturer
   d. repeat the procedures

Answers to
Reading Assignment 11 Review Exercises

1. (a) b c d

2. False. Care must be taken not to damage pitot tube or thermocouple tips.

3. a. check fluid levels
   b. clean stopcocks
   c. change absorbing solutions if necessary
   d. check for leaks

4. a b (c) d

5.

	Yes	No
a.	x	
b.	x	
c.	x	
d.		x
e.	x	
f.	x	
g.	x	
h.	x	
i.	x	

6. a b (c) d

7. False. Must desiccate to a consistent moisture level.

8. (a) b c d

Sampling and Analysis

Lesson F

On-site Measurements

Lesson Goal

The goal of this lesson is for you to understand the quality assurance checks and procedures that can be followed while conducting a source test.

Lesson Objectives

After completing this lesson, you should be able to:

1. use an on-site checklist to see if proper procedures are followed in a test method.
2. list at least three special precautions that should be taken when using a Type S pitot tube.
3. state when an Orsat analyzer is to be leak checked and state the criterion for repeating analyses.
4. list at least three special precautions that should be taken when performing Reference Method 3 for emission rate factor or excess air calculations.
5. state the conditions for an acceptable Reference Method 4 sample run.
6. discuss precautions that should be observed when determining the isokinetic sampling rate using a nomograph.
7. explain how filter blanks are used in Reference Method 5.
8. describe the leak-check procedure for a Reference Method 5 sampling train.
9. describe the probe-rinsing procedure outlined in Volume III—Section 3.4.4.
10. describe the sample-recovery procedure used in Reference Method 6, including the handling of blanks.
11. outline the evacuation-purge-sampling procedure followed in Reference Method 7, and state the leak-rate requirements.

Materials

Assignment 12

- Section 3.1.4, On-site Measurements in Section 3.1
  Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate
- Section 3.2.4, On-site Measurements in Section 3.2
  Method 3—Determination of CO_2, O_2, Excess Air, and Dry Molecular Weight
- Section 3.3.4, On-site Measurements in Section 3.3
  Method 4—Determination of Moisture in Stack Gases

Assignment 13

- Section 3.4.4, On-site Measurements in Section 3.4
  Method 5—Determination of Particulate Matter from Stationary Sources
- Section 3.5.4, On-site Measurements in Section 3.5
  Method 6—Determination of Sulfur Dioxide Emissions from Stationary Sources
- Section 3.6.4, On-site Measurements in Section 3.6
  Method 7—Determination of Nitrogen Oxide Emissions from Stationary Sources
- Optional: Section 3.7.4, On-site Measurements in Section 3.7
  Method 8—Determination of Sulfuric Acid Mist and Sulfur Dioxide Emissions from Stationary Sources

Reading Guidance—Assignment 12

Begin by reading Section 3.1.4—On-site Measurements for Method 2. Read pages 1 of 12 through 12 of 12.

Paragraph 4.2 outlines Reference Method 2 procedures. Note that the data form of Figure 4.1 provides spaces for each parameter that will be needed to determine the stack gas velocity, v_s. Also note the section of the form used for recording data from the cyclonic flow determination.

The material in paragraph 4.2.1 on page 3 of 12 was not discussed in this amount of detail in the explanation of Reference Method 1 (Volume III—Section 3.0 *General Aspects,* page 10 of 19). Note the two methods of measuring stack dimensions, and the problems that can occur with non-uniform dimensions and the buildup of particulate matter in ducts.

On page 5 of 12, paragraph 4.2.3, note the procedures given for checking for plugging of pitot tube openings.

Paragraph 4.2.5 on page 7 of 12 gives a number of common sense techniques that can be followed to help you perform Reference Method 2. Item 2 of this paragraph is particularly important, since a maximum response does not correspond to the pitot tube impact opening being aligned with the direction of flow ($\theta = 0°$ yaw). See Figure 2.6 in Section 3.1.2, page 10 of 21.

Paragraph 4.2.6 gives a number of methods for measuring static pressure. You should be aware that in all Reference Method calculations, the static pressure is used on an absolute basis. For example, if the barometric pressure is 29.82 mm Hg and the stack static pressure was found to be -2.0 in. of H_2O, then the value used in subsequent calculations would be

$$P_{s_{abs}} = P_{bar} + \frac{P_s}{13.6} =$$

$$= 29.82 - \frac{2.0}{13.6}$$

$$= 29.67$$

You can see that for the small static pressures normally encountered in stack tests (except near fans, venturis, etc.), errors in this measurement have limited effect on final calculations.

The on-site measurements checklist shown in Figure 4.2 (page 10 of 12) can be used to see if proper procedures are followed when performing the reference method. This form, used in conjunction with the Activity Matrix of Table 4.1, can help to check the performance of the tester.

Continue your reading with Section 3.2.4—On-site Measurements for Method 3. Read pages 1 of 12 through 12 of 12.

Note in paragraph 4.1, the three methods of obtaining a sample for subsequent Orsat analysis:

- single-point grab sampling and analysis
- single-point integrated sampling and analysis
- multipoint integrated sampling and analysis

Each method has its purpose. The significant point is that the Orsat method can be used to determine

1. dry molecular weight
2. O_2 concentrations for F factor emission rate calculations

Standards requiring an F factor emission rate calculation generally specify that a multipoint integrated sample be obtained for Orsat analysis. For example, in 40 CFR 60 Subpart D for fossil-fuel-fired steam generators,

> "the oxygen sample shall be obtained simultaneously by traversing the duct at the same sampling location used for each run of Method 5" (paragraph 60.46 f3i)

When reading paragraph 4.1, pay particular attention to the leak-check and purging procedures for the sample bags.

Read paragraph 4.2.2 carefully. Note the leak-check requirements and the conditions for accepting the data. When obtaining an integrated, multipoint sample, the source tester should be sure to turn off the sample pump to the bag (if one is used) before switching ports. Not doing this is a common blunder and leads to the dilution of the sample with ambient air.

Note the special precautions that should be taken with the Orsat analyzer. These are discussed in paragraph 4.3. An article has been written on collaborative field studies using the Orsat analyzer. The article contains points that can supplement Section 3.2.4. See Mitchell, W.J. and Midgett, M., "Field Reliability of the Orsat Analyzer" *J. Air Pol. Control Assoc.* 21:491-495. 1976.

Many of the problems associated with the Orsat analyzer stem from operator error. Proper training and experience are essential if one is to obtain quality data from this deceptively simple analytical method.

Continue your reading with Section 3.3.4—On-site Measurements for Method 4. Read pages 1 of 10 through 10 of 10.

Note again the on-site measurements checklist. This is given on page 2 of 10 and provides a handy means of seeing if proper procedures were followed during the test.

Note, in paragraph 4.2, the specification of the sampling time and sampling rate. A sampling rate of 0.75 ft^3/min corresponds merely to maintaining the system at a ΔH value near the $\Delta H_@$ of the meter box.

Paragraph 4.2.4 gives the procedures for sampling at a constant rate. Note that item 12 on page 7 of 10 recommends that the volume taken at each point not differ by more than $\pm 10\%$ from the average sample volume for all points.

> You have completed your reading for Assignment 12. Do the review exercises which follow and check your answers after you complete them. The correct answers are given on the page following the review exercises.

Reading Assignment 12 Review Exercises

1. SST got a job to do a stack test at Superior Light and Power Co. The ports were located on the stack, which had a diameter of about 40 feet at that point. Jeff, a new member of the team, was given the job of determining the stack dimensions. What would be the best way of doing this?
   a. Get a 40-ft-long piece of Unistrut® insert it into the port, and mark it off.
   b. Go to the woods next to the plant, get a 40-ft-long tree, and do the same thing.
   c. Measure the outside circumference of the stack and correct for the wall/ insulation thickness.
   d. Obtain stack blueprints from the plant engineer and read the dimensions from them.

2. Jeff inserted the pitot tube into one of the four ports at the sampling location. In starting the velocity traverse, he rotated the probe (yaw axis) and found that he could get a maximum reading on his manometer by doing this. How should he proceed?
   a. He should obtain the maximum Δp value at all points of the traverse because Δp is at a maximum at 0° yaw angle.
   b. He should align the probe with reference to the stack geometry if it is determined that cyclonic flow is absent.
   c. He should align the probe to obtain maximum Δp values since the flow would be cyclonic.
   d. He should not use the Type S probe. He should use a Fecheimer probe for this particular condition.

3. Jeff needed to determine the stack static pressure. The static pressure was obviously negative because a piece of paper would quickly be sucked into the stack. However, when he stuck a straight tube into the port to determine p_s, he found a pressure differential fluctuating around zero, with no more of a meter reading than -0.5 in. H_2O. What could have been the problem?
   a. He didn't stick the tube in far enough.
   b. He didn't plug the port.
   c. The cyclonic flow was creating the condition.
   d. No problem. That's the way it was and should be recorded as such.

4. The Orsat analyzer must be leak checked before and after analysis for which method(s) listed below?
   a. determination of gas composition for emission rate factor determinations
   b. determination of gas composition for stack gas molecular weight calculations
   c. determination of gas composition for excess air correction factors

5. When obtaining the integrated sample for subsequent Orsat analysis, Jeff had to change ports four times. He hooked up a separate probe on the side of the Method 5 probe and used a separate pump to obtain the Orsat sample during the traverse. When switching ports, he turned off the pump for the meter box, but forgot about the pump for the Orsat sample. How would this affect the data?
   a. The emission rate data would be higher than true.
   b. The emission rate data would be lower than true.
   c. There would be no effect.

6. Three analyses are to be made of each Orsat sample. Which of the following would be true if the data were to be used in an F factor calculation?
   a. repeat until any three CO_2 analyses differ by $\leq 0.3\%$ (absolute) when the mean CO_2 reading is $\leq 4\%$
   b. repeat until any three O_2 analyses differ by $\geq 0.3\%$ when the mean O_2 reading is $\leq 15\%$
   c. repeat until any three mean O_2 analyses differ by $\leq 0.2\%$ when the mean O_2 reading is $> 15\%$
   d. repeat until any three molecular weight mean values differ by ≤ 0.3 g/mol

7. List at least four special precautions that should be taken when performing an Orsat analysis.
   - _____
   - _____
   - _____
   - _____

8. A Method 5 meter box was used for a stack gas moisture determination. The $\Delta H_@$ of the orifice meter was 1.83 in. H_2O. What should the ΔH of the system be while running Method 4?
   a. variable—adjusted to the isokinetic rate
   b. variable—proportional to stack gas velocity
   c. constant at any arbitrary value
   d. constant near 1.83 in. H_2O

9. The volume taken at each point of a Method 4 traverse should be within $\pm$____ of the average sample volume for all points.
   a. 2%
   b. 5%
   c. 10%
   d. 20%

10. What are the leak-rate requirements for a Reference Method 4 test?
   a. The leak rate must be less then 2% of the average sample rate.
   b. There are no leak-rate requirements, since sample dilution will not affect the results.
   c. The leak rate must use less than 4% of the average sample rate.
   d. The leak rate must be greater than 4% of the average sample rate.

Answers to
Reading Assignment 12 Review Exercises

1. a b (c) d
2. a (b) c d
3. (a) (b) c d
4. (a) b (c)
5. (a) b c
6. a b (c) d

since $E = F_d c_s \dfrac{20.9}{20.9 - \%O_2}$

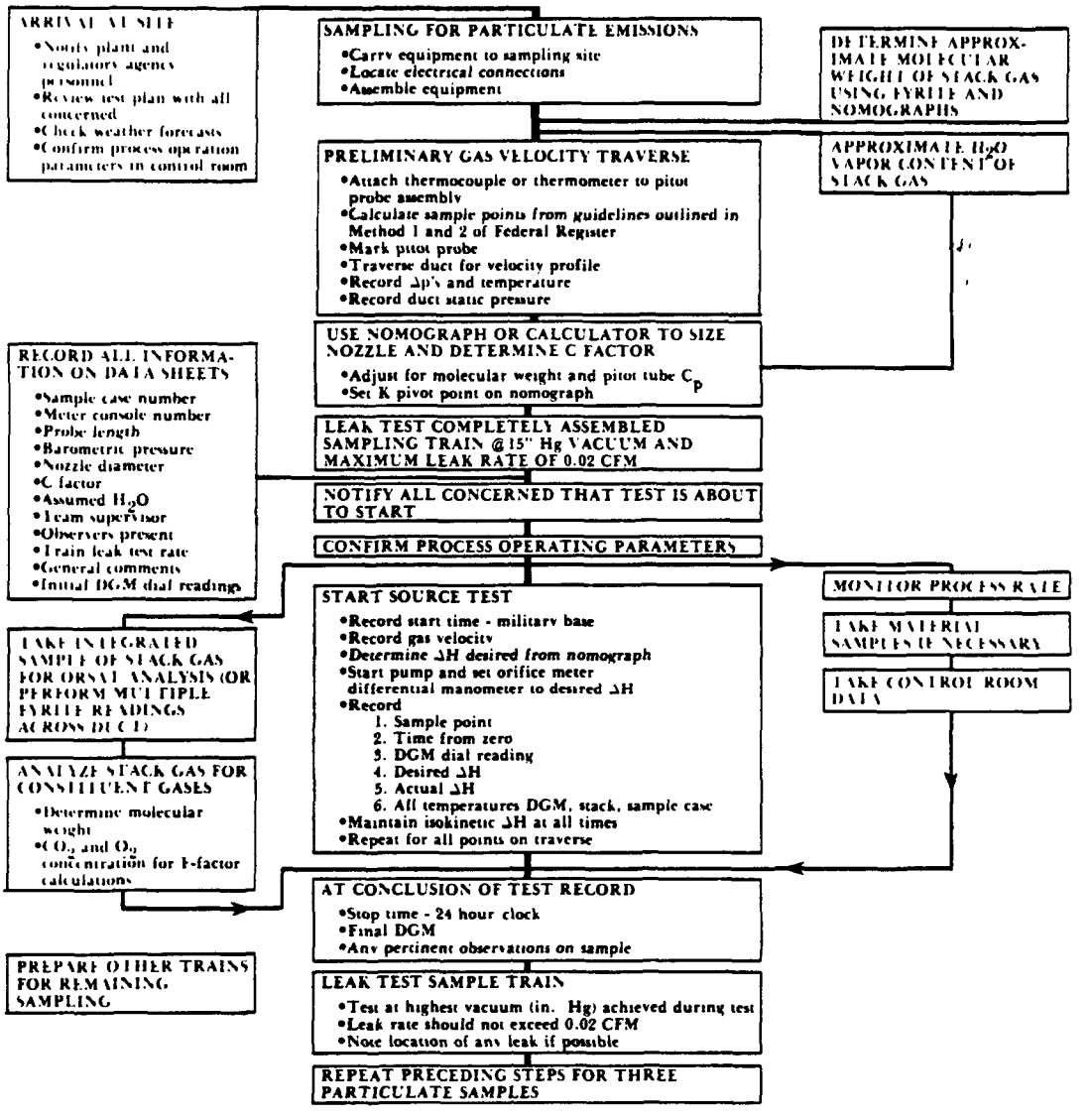
$\%O_2$ would be higher than true,
$(20.9 - \%O_2)$ would be too low, and
therefore E would be higher than true

7. • Do not allow ambient air to enter analyzer.
   • Follow proper sequence—O_2, CO_2, CO.
   • Saturate indicating solution with salt.
   • Keep absorber solution from manifold.
   • Allow 5 minutes for samples to come to temperature.
   • Operate at constant temperature and pressure.
8. a b c (d)
9. a b (c) d
10. a b (c) d

Reading Guidance—Assignment 13

Begin by reading Section 3.4.4—On-site Measurements for Method 5. Read pages 1 of 19 through 19 of 19.

Performing a source test for particulate matter involves the techniques of Reference Methods 1-4. The many procedures to be done require both planning and coordination. The figure below* shows how these procedures are interrelated, and may help you review Section 3.4.4, On-site Measurements for Reference Method 5.



*Abstracted from Aldina, G.J. and Jahnke, J.A., 1979, APTI Course 450 *Source Sampling for Particulate Pollutants—Student Manual.* EPA 450/2-79-006. Pages 5.4 to 5.5

Preliminary data on temperatures, pressures, etc. are used to set up the nomograph. A data form that can aid in this set-up is given on page 8 of 19. The nomograph for Reference Method 5 was developed in the early 1970s—before hand-held calculators were widely available. At that time, it enabled the stack tester to quickly perform the lengthy calculations for the nozzle diameter, D_n, and the isokinetic sampling rate.

Today, hand-held programmable calculators are available and relatively inexpensive (although a simple calculator with the basic math functions and a square root function is all that is actually needed). Numerous types of plastic slide-rule calculators that will do these two calculations have also been developed. The reference method does not specify that the Method 5 nomograph is required. The tester may use either a calculator, a nomograph, or a slide rule—it is most often a matter of personal preference. However, it should be determined that the tester knows how to use the calculation instrument that he chooses. The test observer or auditor should ask the person doing these calculations to work an example problem before the test. Any discrepancies between the known answer and the one calculated should be resolved. Such example problems are given in APTI Course 450 *Source Sampling for Particulate Pollutants—Student Workbook,* 1979. EPA 450/2-79-007, pages 53 and 57.

Again, leak-check procedures are very important. Read paragraph 4.2.5 carefully (pages 5 of 19 through 7 of 19). A common blunder of the novice stack tester is improperly releasing the vacuum at the end of the leak test. If care is not taken, water from the impingers will back up onto the filter and the system will have to be set up again.

Note that a leak check is mandatory only at the end of the test. However, the pretest leak check is recommended, since it might save a test if a major leak can be detected before the testing is started.

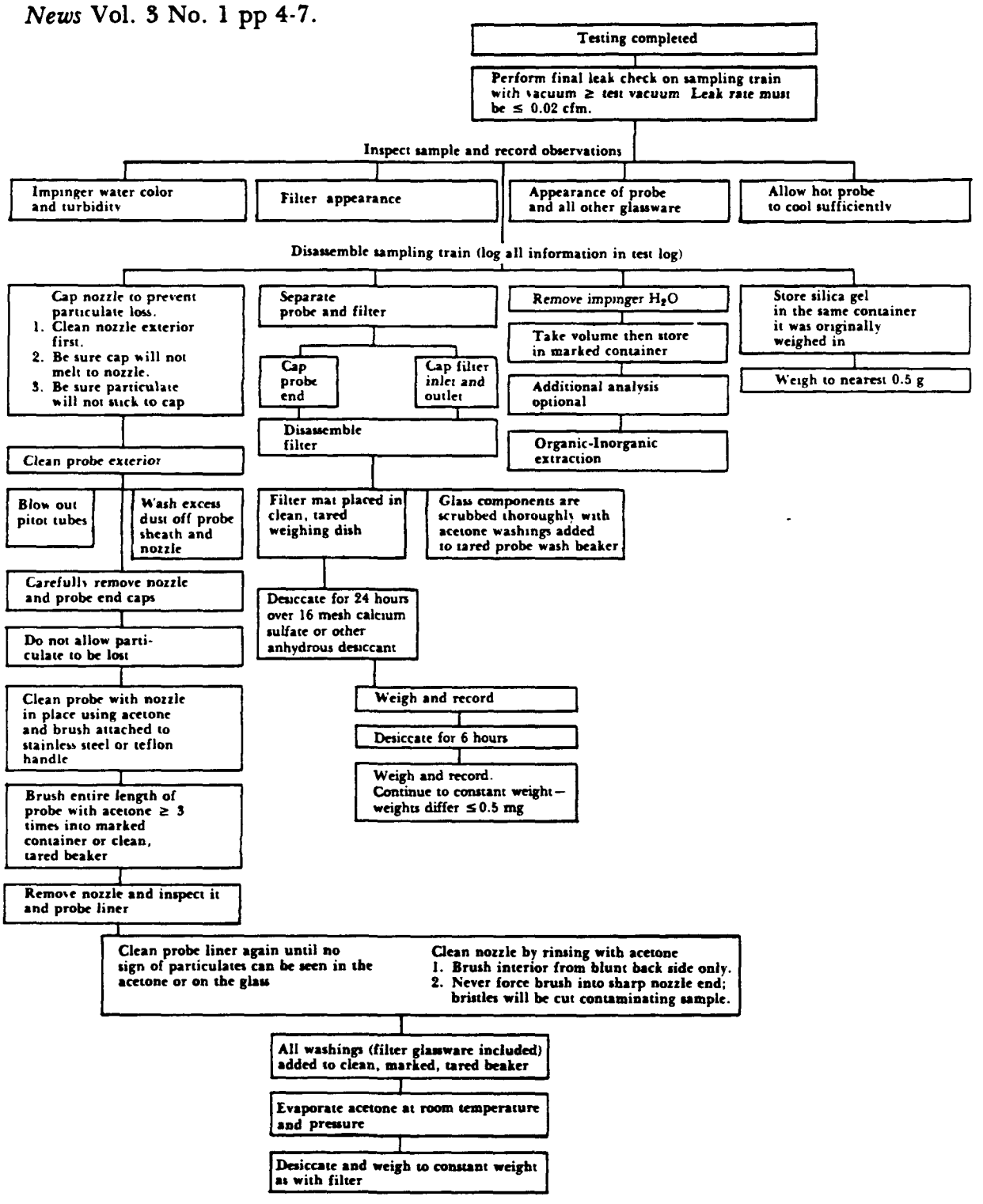
On page 9 of 19, paragraph 4.2.6, the nomograph is again discussed. Note the assumptions inherent in the nomograph. If the C_p and M_s values are beyond the limits recommended here, corrections to the nomograph values can be made. Equations for correction factors are given in APTI Course 450 *Source Sampling for Particulate Pollutants—Student Workbook,* 1979. EPA 450/2-79-007, page 56.

Paragraph 4.3.1 on page 10 of 19 contains a very important point which should be included in any quality assurance program. This is the use of the filter blank. The reference method itself does not require that filter blanks be saved and this has normally not been a procedure followed by testing teams. It might be thought that weighing filters is a simple procedure and that errors from the analytical balance determinations would be small. Weighing, however, is a tedious proposition and mistakes in weighing frequently occur.

Also, problems with high humidity or with the transport of the filters may raise questions about the actual mass determination of the Reference Method 5 sample. Setting aside three tared filters at the beginning of the test, and treating them as actual sample filters throughout the test, provides an excellent quality control check of the gravimetric procedures.

Cleaning the sample train is a very important part of Reference Method 5. Recent studies indicate that over 50% of the particulate catch can end up in the probe and not on the filter. For this reason, as much care should be taken with the probe-washing procedures as with the handling of the filters. An outline of the cleaning procedure is provided below to supplement the discussion of paragraph 4.3, pages 10 of 19 through 14 of 19.

A good article on Method 5 clean-up procedures has been written—see Riley, C.E., July 1975. EPA Method 5 Sample Clean-up Procedures. *Stack Sampling News* Vol. 3 No. 1 pp 4-7.

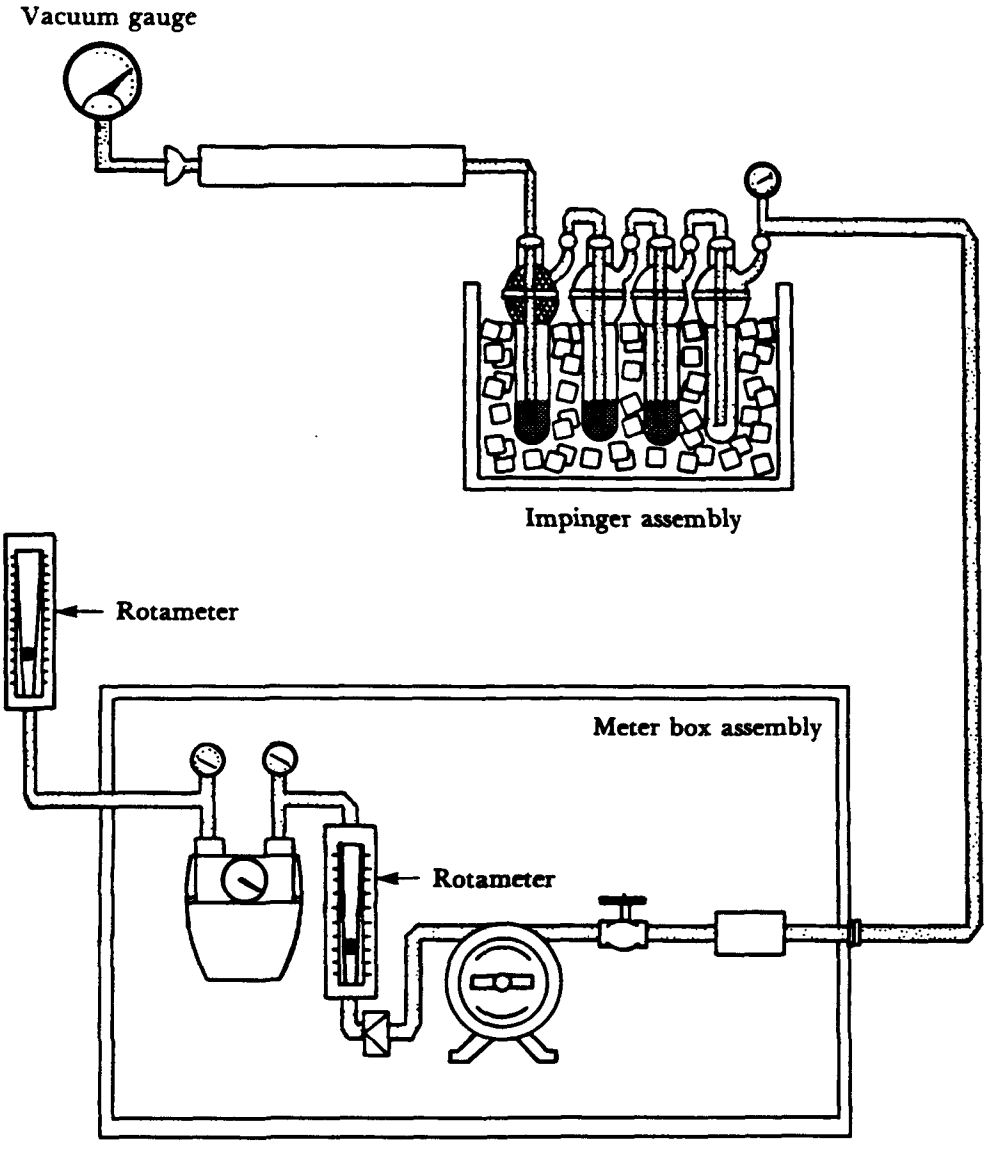


The on-site measurements checklist provides a convenient means of seeing if many of the procedures involved in a Reference Method 5 test were performed. This may be useful for the test observer or for the test team leader checking the performance of his crew.

> Continue your reading with Section 3.5.4—On-site Measurements for Method 5. Read pages 1 of 12 through 12 of 12.

Paragraphs 4.1 and 4.2 of Section 3.5.4 are fairly straightforward. The notes and comments given in paragraph 4.3, *Sampling,* should be read carefully.

A leak-check procedure for the assembled sampling train is given in paragraph 4.3.2. The figure below is provided to assist you in your reading.



Reference Method 6: sample train leak check.

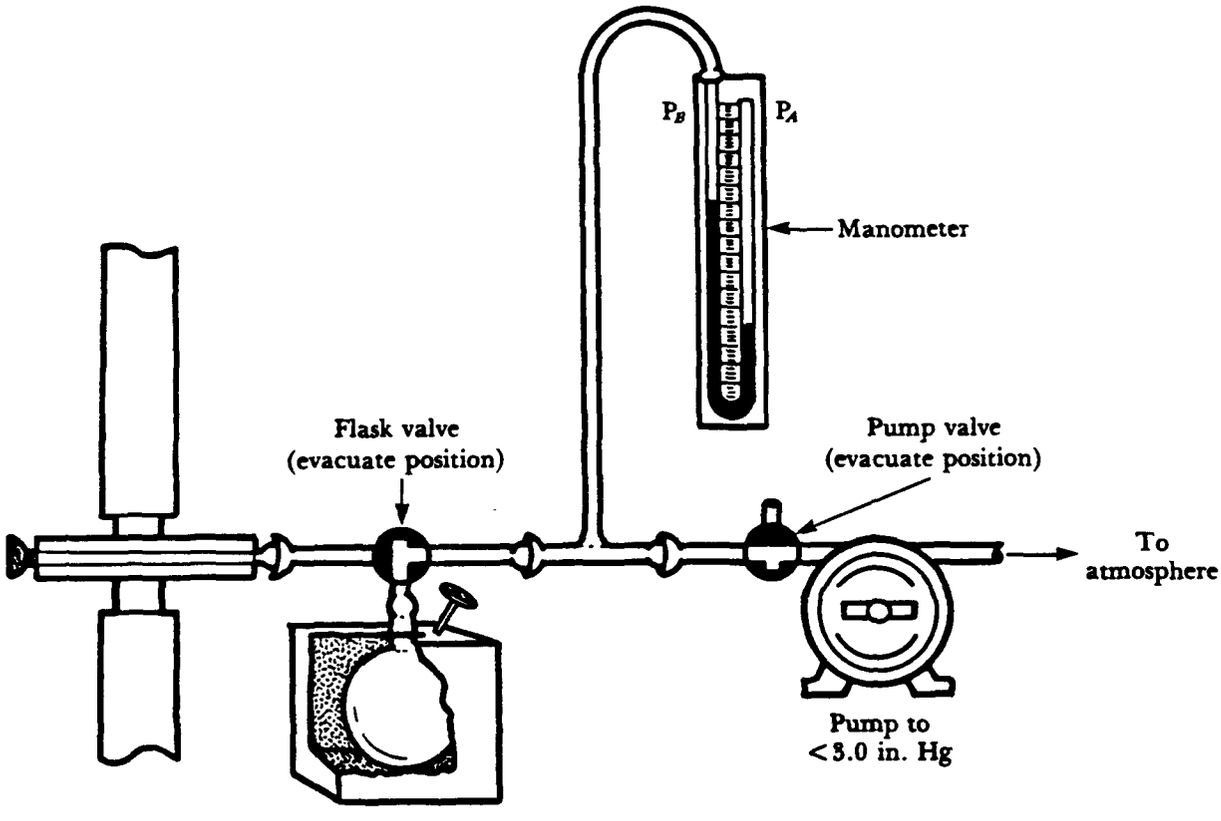
Paragraph 4.3.3 discusses sampling at a constant rate. Sampling for SO_2 is conducted at a constant rate—not proportionally or isokinetically. The sample volume at each reading interval should be within ± 10% of the average sample volume for each interval.

During sampling, SO_3 in the flue gas is scrubbed from the gas stream by the 80% isopropyl alcohol in the first midget impinger. The SO_2 passes through the first impinger and is oxidized by the H_2O_2 in the second and third impingers by the following reaction:

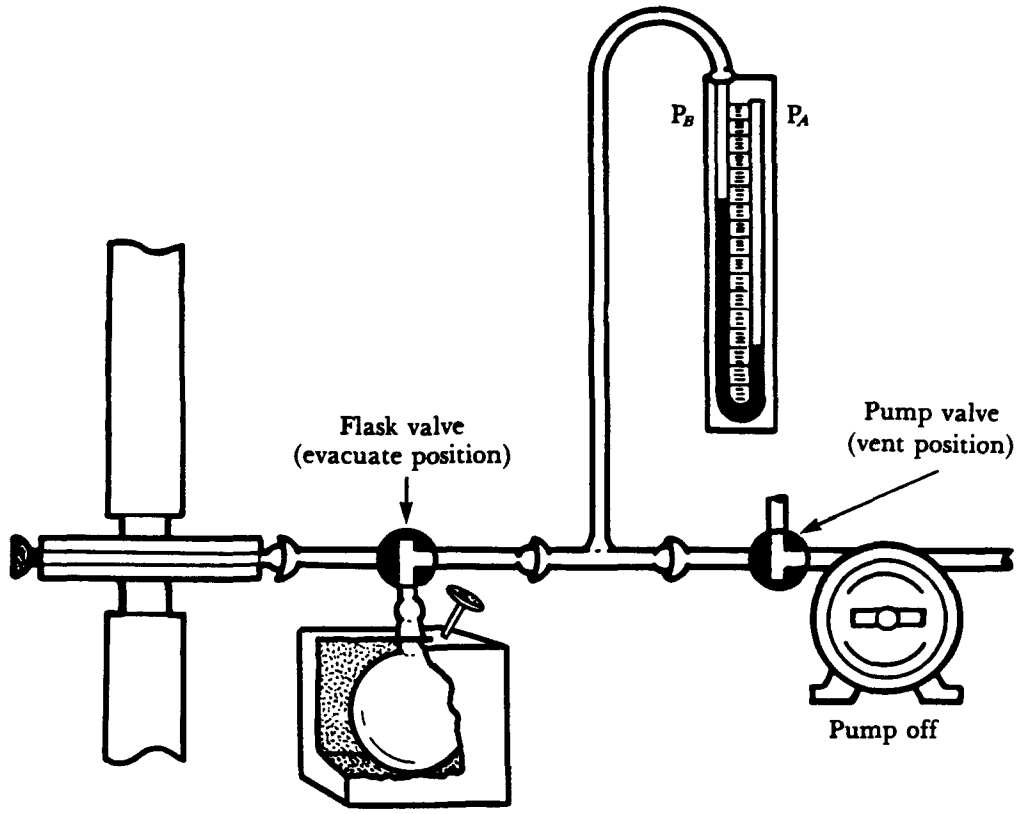
SO_2	+	H_2O_2	→	H_2SO_4
Sulfur dioxide		Hydrogen peroxide		Sulfuric acid

Continue your reading with Section 3.6.4—On-site Measurements for Method 6. Read pages 1 of 11 through 11 of 11.

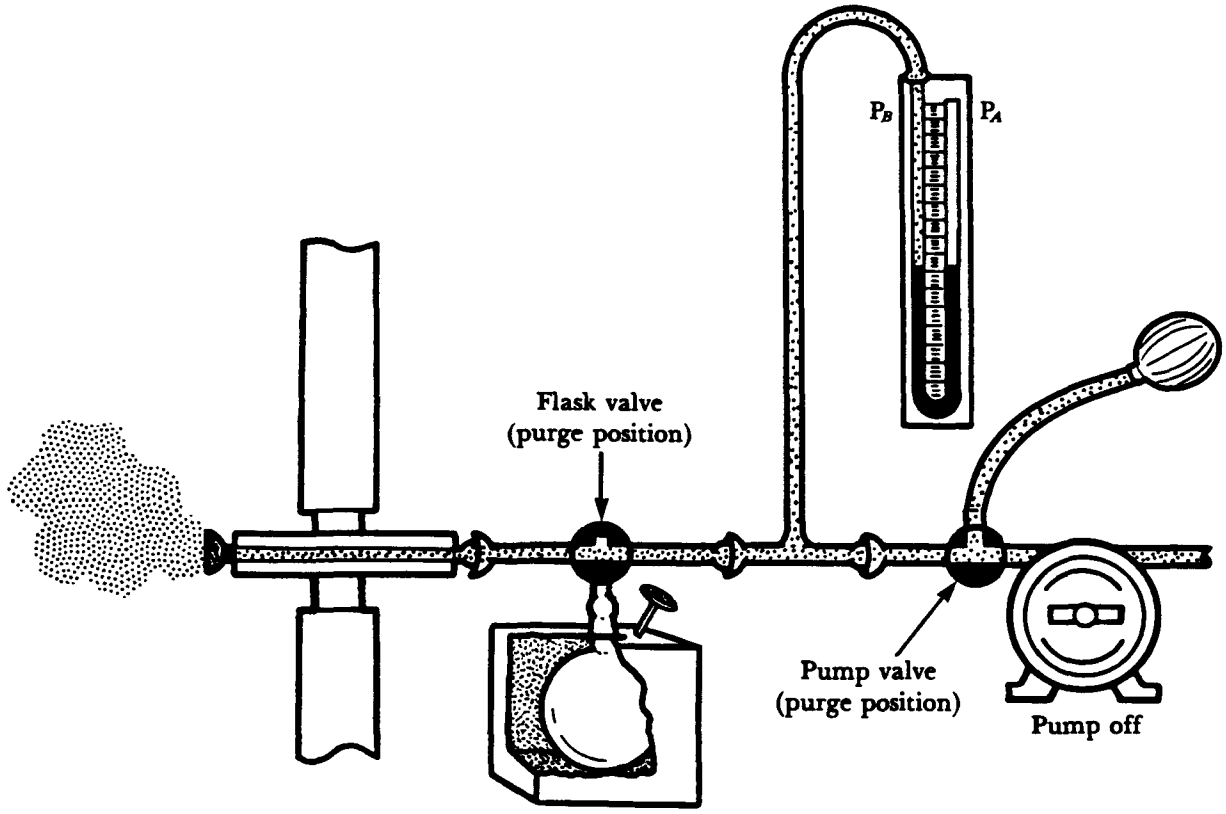
Reference Method 7 requires careful attention to both sampling and analytical details. The following seven figures will help you follow the discussion on sampling procedures given on pages 2 of 11 through 6 of 11, paragraph 4.3.3.



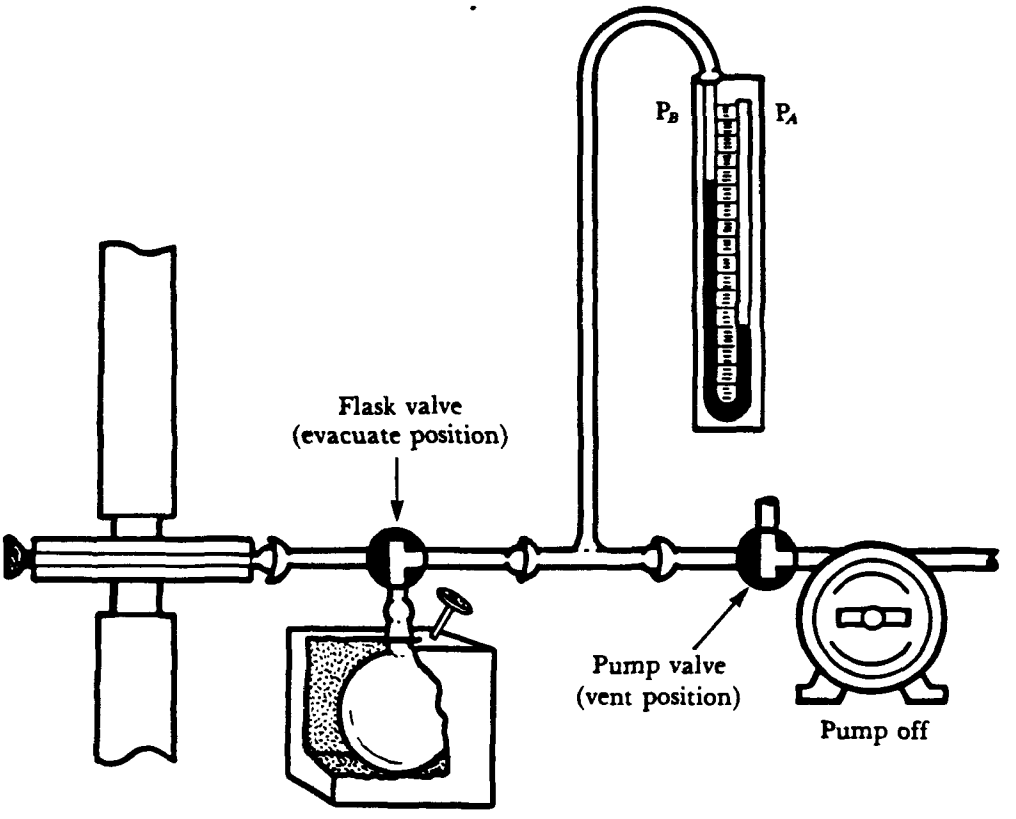
Reference Method 7: evacuate flask. Paragraph 4.3.3, subparagraph 1.



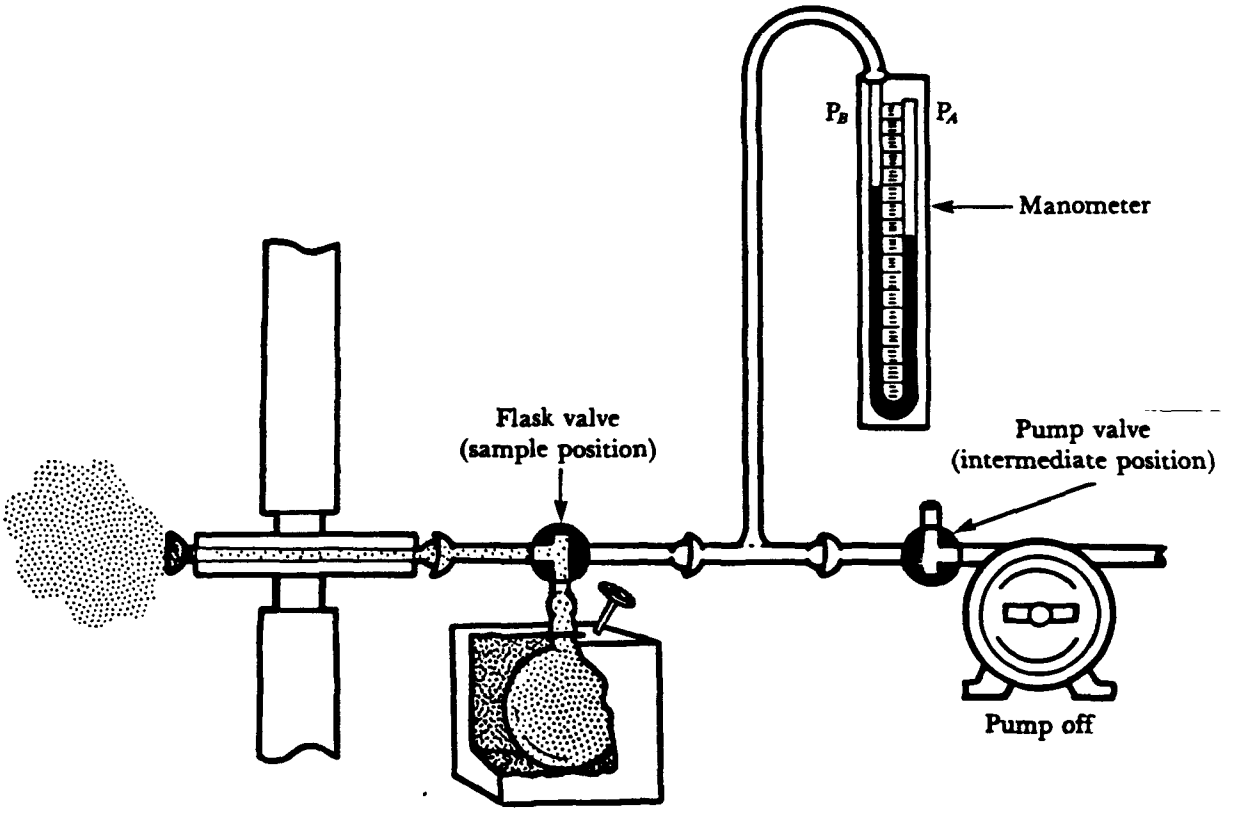
Reference Method 7: initial leak check. Paragraph 4.3.3, subparagraph 2.



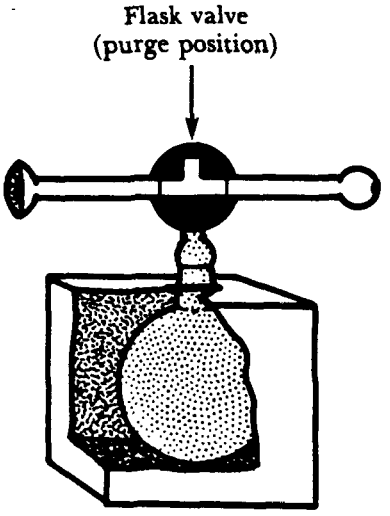
Reference Method 7: purge. Paragraph 4.3.3, subparagraphs 4, 5, and 6.



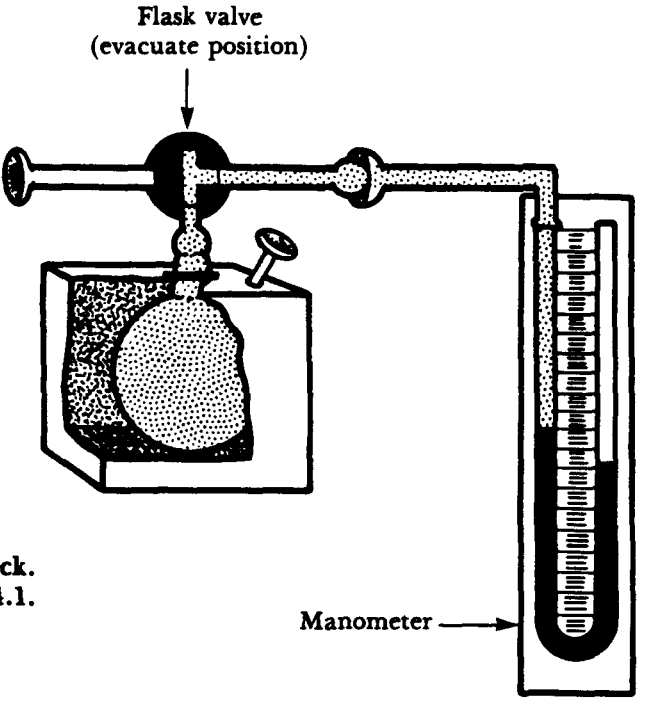
Reference Method 7: final leak check. Paragraph 4.3.3, subparagraphs 8 and 9.



Reference Method 7: sampling. Paragraph 4.3.3, subparagraph 10.



Reference Method 7: sample recovery. Paragraph 4.3.3, subparagraphs 11 and 12.



Reference Method 7: final pressure check. Paragraph 4.4.1.

Optional Assignment

The procedures for Method 8 are similar to those for Method 5. You may continue reading Section 3.7.4, On-site Measurements—Determination of Sulfuric Acid Mist and Sulfur Dioxide Emissions from Stationary Sources on pages 1 of 18 through 18 of 18.

You have completed your reading for Assignment 13. Do the review exercises which follow and check your answers after you complete them. The correct answers are given on the page following the review exercises.

Reading Assignment 13 Review Exercises

1. Using your Reference Method 5 nomograph, Method 5 slide rule, or calculator, compute the isokinetic sampling rate (ΔH), if the Δp at the first test point is found to be 1.0 in. H_2O. The following calibration factors and stack parameters were determined before sampling:

   $Q_m = 0.75$ cfm
   $\Delta H_@ = 1.85$
   Pitot tube $C_p = 0.85$
   $t_m = 80\,°F$
   $P_m = 30.0$ in. Hg
   $P_s = 29.6$ in. Hg
   $B_{wm} = 0$
   $B_{ws} = 0.12$
   $t_s = 280\,°F$
   $M_d = 29$ lb/lb•mole
   Average $\overline{\Delta p} = 0.80$ in. H_2O

   Note: The following expressions can be used in this problem:

   $$M_s = M_d(1 - B_{ws}) + 18B_{ws}$$

   Nozzle Diameter Selection Equation

   $$D_n = \sqrt{\left(\frac{0.0358 Q_m P_m}{T_m C_p (1 - B_{ws})}\right)\sqrt{\frac{T_s M_s}{P_s(\Delta p)}}}$$

   Isokinetic Rate Equation—Working Form

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

   Answer: ΔH = _____ in. H_2O

2. Mike assembled the sampling train, but didn't bother to perform a leak check until after the test. After the test, he found a leak rate of 0.10 ft^3/min. Answer the following questions:
   a. Is the test acceptable? ____yes ____no
   b. Was the actual test performed isokinetically if the calculated ΔH values were set at each point? ____yes ____no
   c. Would he be able to correct the final metered volume for the leak? ____yes ____no
   d. Would he be able to correct the weight of the collected particulate matter for the leak? ____yes ____no

3. Under which of the following conditions is the use of the nomograph valid without compensating for nomograph assumptions?
   a. $C_p = 0.79$
   b. $C_p = 0.84$
   c. $M_d = 27.3$
   d. $M_d = 24.1$
   e. $C_p = 0.87$

4. The three filter blanks mentioned in paragraph 4.3.1 on page 10 of 19 (Section 3.4.4) should be:
   a. saved so that they can be used to replace a filter if it gets wet during the leak check.
   b. left in the desiccator in the lab.
   c. transported along with the sample filters and weighed again at the end of the test.
   d. weighed only at the end of the test, so the balance can be checked.

5. In the Method 5 sample recovery procedures, the probe should be:
   a. rinsed only once since more rinsings waste time and contribute to greater error because of increased amounts of solvent.
   b. rinsed only once since the bulk of the particulate matter is caught on the filter and any probe losses would be unimportant.
   c. rinsed with soap and water so that it will be really clean before the next test.
   d. rinsed with acetone more than three times so that all the particulate matter can be collected.

6. Look at Figure 4.5 on pages 15 of 19 through 17 of 19 in Section 3.4.4 (or Figure 4.5 on pages 6 of 15 through 8 of 15 in Section 3.4). An agency observer noticed a number of things listed below. Make believe that you are the observer, and check off the points in Figure 4.5.
   a. A glass liner was used.
   b. The pitot tube coefficient was assumed to be 0.84.
   c. One of the filters used had a tear.
   d. The test team had only a ¼-in. nozzle at the site.
   e. The pitot tube lines were checked for leaks.
   f. A calculator was used for the isokinetic rate determinations.
   g. Only time and volume data were put on the sampling form (Figure 4.2); other data were written on a note pad.
   h. Clean-up was performed at the sampling site.
   i. Some filter paper was left on the silicone gasket.
   j. Probe-wash bottles were labeled with labels similar to Figure 4.3.

7. In the leak-check procedure for the Reference Method 6 assembled sampling train:
   a. the leak rate is determined by the amount of bubbling in the impingers.
   b. the end of the probe is capped and the leak rate determined from the dry gas meter.
   c. the end of the probe is capped and the leak rate determined from a rotameter attached to the outlet of the dry gas meter.
   d. the end of the probe is capped with a vacuum gauge and a rotameter is attached to the outlet of the dry gas meter.

8. Method 6 sampling is performed:
   a. at a constant rate.
   b. isokinetically.
   c. proportionally.
   d. by obtaining a grab sample.

9. The contents of the first Method 6 impinger should be:
   a. discarded.
   b. retained for checking sampling errors.
   c. retained for $SO_4^=$ analysis.
   d. retained for H_2SO_4 analysis.

10. Data taken during the Method 6 test should be:
   a. packed with the sample bottles and shipped with them.
   b. recorded in duplicate—one set mailed to the lab, one set hand carried.
   c. photocopied at the base lab only.
   d. given, for safe-keeping, to the test observer.

11. The following true-false statements address the Reference Method 7 leak-check and sampling procedures.
   a. True or False? When the flask is first evacuated, the flask valve is set at the evacuate position and the pump valve is set at the evacuate position.
   b. True or False? In the initial leak check, the flask valve is in the evacuate position and the pump valve is in the vent position.
   c. True or False? In the purge procedure, the manometer fluid is level in both legs.
   d. True or False? In the final leak check, the flask valve is in the evacuate position and the pump valve is in the purge position.
   e. True or False? The flask is removed when the valve is in the evacuate position.
   f. True or False? The flask valve is in the purge position during the final pressure check.

12. The pH of the Method 7 flask sample is adjusted to a value of:
   a. 7.0 to 9.
   b. 9 to 12.
   c. 5 to 7.
   d. It is not adjusted.

Answers to
Reading Assignment 13 Review Exercises

1. $M_s = M_d(1 - B_{ws}) + 18B_{ws}$

   $M_s = 29(1 - 0.12) + 18(0.12) = 27.7$

$$D_n = \sqrt{\left(\frac{0.0357 Q_m P_m}{T_m C_p}\right) \frac{1}{(1 - B_{ws})} \sqrt{\frac{T_s M_s}{P_s \Delta p}}}$$

$$= \sqrt{\frac{(0.0357)(0.75)(30.0)}{(540)(0.85)} \frac{1}{0.88} \sqrt{\frac{(740)(27.7)}{(29.6)(0.80)}}}$$

$$= 0.241 \text{ in.}$$

   Choose a nozzle close to 0.241 in., e.g., 0.25 in.
   then:

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

$$= \left\{846.72(0.25)^4 1.85(0.85)^2(0.88)^2 \frac{29}{27.7} \left(\frac{540}{740}\right) \left(\frac{29.6}{30.0}\right)\right\} \Delta p$$

$$= 2.59 \Delta p$$

2. a. no
   b. no
   c. yes
   d. no

3. a (b) c d

4. a b (c) d

5. a b c (d)

6. Answers on pages 167 through 169

7. a b c (d)

8. (a) b c d

9. a (b) c d

10. a (b) c d

11. a. True d. False
    b. True e. False
    c. True f. False

12. a (b) c d

6.

ON-SITE MEASUREMENTS CHECKLIST
(Method 5, Figure 4.5)

Sampling Train Schematic Drawing

Apparatus

Probe nozzle: stainless steel _____ glass _____
Button-hook _____ elbow _____ size _____
Clean? _____
Probe liner: borosilicate ✓(a) quartz _____ other _____
Clean? _____
Heating system* _____
Checked? _____
Pitot tube: Type S _____ other _____
Properly attached to probe?* _____
Modifications _____
Pitot tube coefficient ✓ 0.84 (assumed) (b)
Differential pressure gauge: two inclined manometers _____
other _____ sensitivity _____
Filter holder: borosilicate glass _____ glass frit _____
filter support _____ silicone gasket _____ other _____
Clean? _____
Condenser: number of impingers _____
Clean? _____
Contents: 1st _____ 2nd _____ 3rd _____ 4th _____
Cooling system _____
Proper connections? _____
Modifications _____

Barometer: mercury _____ aneroid _____ other _____
Gas density determination: temperature sensor type _____
pressure gauge _____
temperature sensor properly attached to probe?* _____

Procedure

Recent calibration: pitot tubes* _____
meter box* _____ thermometers/thermocouples* _____
Filters checked visually for irregularities?* tear in filters (c)
Filters properly labeled?* _____
Sampling site properly selected? _____
Nozzle size properly selected?* ¼ in. nozzle (only nozzle available) (d)

(continued)

(continued)

Selection of sampling time? _____
All openings to sampling train plugged to prevent pretest contamination? _____
Impingers properly assembled? _____
Filter properly centered? _____
Pitot tube lines checked for plugging or leaks?* ✓ (e)
Meter box leveled? _____ Periodically? _____
Manometers zeroed? _____
ΔH@ from most recent calibration _____
Nomograph set up properly? ✓ not applicable - calculator used (f)
Care taken to avoid scraping nipple or stack wall?* _____
Effective seal around probe when in-stack? _____
Probe moved at proper time? _____
Nozzle and pitot tube parallel to stack wall at all times?* _____
Filter changed during run? _____
Any particulate lost? _____
Data forms complete and data properly recorded?* ✓ not complete (g)
Nomograph setting changed when stack temp changed significantly? _____
Velocity pressure and orifice pressure readings recorded accurately?* _____
Posttest leak check performed?* _____ (mandatory)
Leakage rate _____ @ in. Hg _____
Orsat analysis _____ from stack _____ integrated _____
Fyrite combustion analysis _____ sample location _____
Bag system leakchecked?* _____
If data forms cannot be copied, record:
approximate stack temp _____ volume metered _____
% isokinetic calculated at end of each run _____

SAMPLE RECOVERY

Brushes: nylon bristle _____ other _____
Clean? _____
Wash bottles: glass _____
Clean? _____
Storage containers: borosilicate glass _____ other _____
Clean? _____ Leakfree? _____
Petri dishes: glass _____ polyethylene _____ other _____
Clean? _____
Graduated cylinder/or balance: subdivisions ≤2 ml?* _____
other _____
Balance: type _____
Plastic storage containers: airtight? _____
Clean? _____
Probe allowed to cool sufficiently? _____
Cap placed over nozzle tip to prevent loss of particulate?* _____
_____

(continued)

(continued)

During sampling train disassembly, are all openings capped? _____
Clean-up area description: ✓ at site (h)
Clean? _____ Protected from wind? _____
Filters: glass fiber _____ type _____
Silica gel: type (6 to 16 mesh)? new? _____ used? _____
Color? _____ Condition? _____
Filter handling: tweezers used? _____
surgical gloves? _____ other _____
Any particulate spilled?* _____
Water distilled? _____
Stopcock grease: acetone-insoluble? _____
heat-stable silicone? _____ other _____
Probe handling: acetone rinse _____
distilled water rinse _____
Particulate recovery from: probe nozzle _____
probe fitting _____ probe liner _____
front half of filter holder ✓ (some paper remaining) (i)
Blank: acetone _____ distilled water _____
Any visible particles on filter holder inside probe?:* _____
All jars adequately labeled? ✓ (j) Sealed tightly? _____
Liquid level marked on jars?* _____
Locked up? _____
Acetone reagent: <0.001% residue? _____
glass bottles _____ (required)
acetone blanks? _____

*Most significant items/parameters to be checked.

Lesson G

Postsampling Operations

Lesson Goal

The goal of this lesson is for you to understand the quality assurance checks and procedures that can be followed in the postsampling/analytical operations of the reference methods.

Lesson Objectives

After completing this lesson, you should be able to:

1. state the procedures outlined in Volume III for the posttest checks of temperature sensors, barometers, and dry gas meters.
2. compare Orsat data with estimated source combustion data as a quick check for gross measurement errors.
3. use the Volume III checklists:
   Procedure for Weighing Filters (Figure 5.5)
   Procedure for Analysis of Acetone Rinse Samples (Figure 5.6)
4. list the standardization activities that are conducted as part of the Reference Method 6 analytical procedures.
5. describe how blanks and control samples are used in the Reference Method 6 analytical procedures.
6. describe how control samples are used in the analytical procedures of Reference Method 7.
7. describe how working standards are used in the analytical procedures of Reference Method 7.

Materials

Assignment 14

- Section 3.1.5, Postsampling Operations in Section 3.1
  Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate
- Section 3.2.5, Postsampling Operations in Section 3.2
  Method 3—Determination of CO_2, O_2, Excess Air, and Dry Molecular Weight
- Section 3.3.5, Postsampling Operations in Section 3.3
  Method 4—Determination of Moisture in Stack Gases
- Section 3.4.5, Postsampling Operations in Section 3.4
  Method 5—Determination of Particulate Matter from Stationary Sources

Assignment 15

- Section 3.5.5, Postsampling Operations in Section 3.5
  Method 6—Determination of Sulfur Dioxide Emissions from Stationary Sources
- Section 3.6.5, Postsampling Operations in Section 3.6
  Method 7—Determination of Nitrogen Oxide Emissions from Stationary Sources
- Optional: Section 3.7.5, Postsampling Operations in Section 3.7
  Method 8—Determination of Sulfuric Acid Mist and Sulfur Dioxide Emissions from Stationary Sources

Reading Guidance—Assignment 14

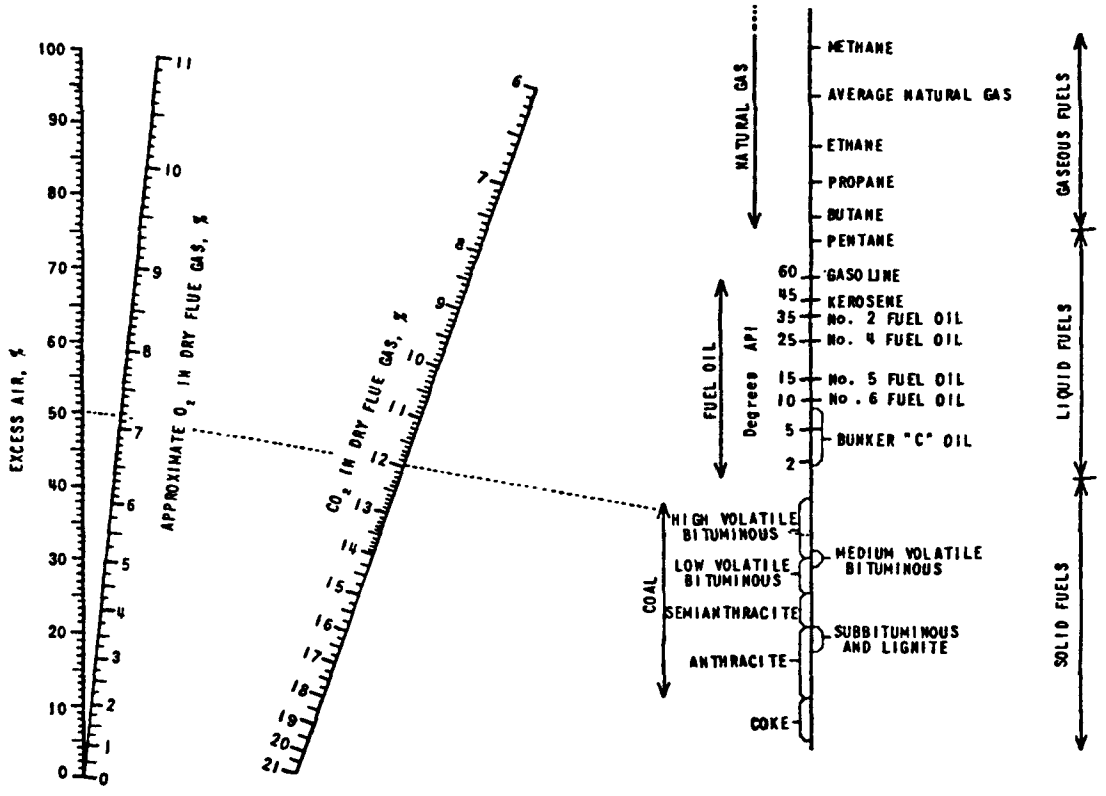
Begin by reading Section 3.1.5—Postsampling Operations for Method 2. Read pages 1 of 3 through 3 of 3.

Checking equipment after a test often seems unnecessary and tedious to someone who is eager to get home or proceed with an analysis. The postsampling checks of thermometers, barometers, etc., have been specified so that they can be done quickly, but yet indicate whether or not there might be a problem. For example, temperature sensor checks merely specify comparing the stack or meter thermometer readings to the ambient temperature.

Note that if the pitot tube is damaged and recalibrated, corrections can be made for the velocity determinations made after the damage occurred. This is true for velocity traverses. If the pitot tube of a Method 5 probe is damaged, the isokinetic rate calculations would be in error and the tester would not have been sampling isokinetically after the damage occurred.

Continue your reading with Section 3.2.5—Postsampling Operations for Method 3. Read pages 1 of 2 through 2 of 2.

There are a number of ways of estimating theoretical CO_2 or O_2 emission concentrations from combustion sources. A convenient nomograph that can be used for this purpose is shown below (Smith, W.S. and Gruber, C.W. *Atmospheric Emissions from Coal Combustion—An Inventory Guide.* Springfield, Va. NTIS No. PB-170-851, 1966).



If the fuel type and one other parameter are known (such as $\%O_2$, $\%CO_2$, or %excess air), a line can be drawn between them and approximate values for the other parameters determined.

Another convenient way of checking Orsat data is by using F factor relationships. The F_d factor represents the ratio of the volume of dry flue gases generated in a combustion process, to the calorific value of the fuel combusted. The F_c factor represents the ratio of the volume of carbon dioxide generated to the calorific value of the fuel combusted.

The F_o factor is the ratio

$$F_o = \frac{20.9}{100} \frac{F_d}{F_c}$$

and is equal to

$$F_o = \frac{20.9 - \%O_2}{\%CO_2}$$

with the $\%O_2$ and $\%CO_2$ being obtained on or adjusted to a dry basis. F_o factors for various fuels are given in the table below.

Fuel type	F_o	Maximum deviations from midpoint F_o value (%)
Coal		
Anthracite	1.070	2.9
Bituminous	1.140	4.5
Lignite	1.076	2.8
Oil		
Gas		
Natural	1.79	2.9
Propane	1.51	1.2
Butane	1.479	0.9

To use this technique, Orsat values for $\%O_2$ and $\%CO_2$ can be substituted into the equation above. If the resultant value differs from the midpoint F_o values by more than the range given in the table, the Orsat analysis should be rechecked or redone (Aldina, G.J. and Jahnke, J.A., 1979. APTI Course 450 *Source Sampling for Particulate Pollutants—Student Manual.* EPA 450/2-79-006, pages 9-5 through 9-15).

Continue your reading with Section 3.3.5—Postsampling Operations for Method 4. Read pages 1 of 4 through 4 of 4.

Note that the dry gas meter is to undergo a posttest check. Refer back to page 11 of 19 of Section 3.3.2 to review this procedure. Note again that these procedures are just data checks and not calibrations.

The dry gas meter is initially calibrated to a precision of 2%. On the posttest check, however, a 5% difference from the calibrated value is allowed before a recalibration is necessary.

If a recalibration is done, the lower calibration factor is then used in the calculations. This results in a lower gas volume collected and a higher particulate concentration.

The dry gas meter thermometer is calibrated to within 3°C initially, but a 6°C deviation is allowed before recalibration is required. In this case, if recalibration is necessary, the higher values are used in the calculations.

Continue your reading with Section 3.4.5—Postsampling Operations for Method 5. Read pages 1 of 15 through 15 of 15.

Postsampling checks of the dry gas meter, thermometers, and barometers are similar to the procedures which you have read about for Methods 2, 3, and 4. Do, however, read Subsection 5.2—Analysis (Base Laboratory) carefully. Figure 5.5 on pages 10 of 15 and 11 of 15 and Figure 5.6 on pages 12 of 15 through 14 of 15 give step-by-step procedures for the handling and analysis.

The filter blanks should be treated the same as the sample filters—from the beginning through the final weighing. Weighing filters can be a tedious procedure. The use of blanks can help uncover inadvertant errors which might not otherwise be detected.

The evaporation of acetone rinses may seem like a simple procedure. But many test teams have often had difficulty meeting the residue requirements for the acetone blanks ($<0.001\%$ of total acetone weight). This problem may not be due to contaminated acetone, but rather, to dust from the air settling into the beakers. Watch glasses don't always help eliminate the problem.

Some testing organizations have found it necessary to evaporate the acetone in glove boxes, with filtered air intakes.

The volume of the acetone blank should be approximately the same as that of the sample rinses. If they are not the same, an error could result. Consider the following case: compare two 250-ml beakers. Also compare 50 ml of acetone blank in one beaker to 200 ml of sample rinse in the other. Assume equal amounts of dust fall into each beaker. If you scaled up the blank reading to correspond to 200 ml, an error would result because you would be scaling up the dustfall in addition to any actual residue in the acetone.

> You have completed your reading for Assignment 14. Do the review exercises which follow and check your answers after you complete them. The correct answers are given on the page following the review exercises.

Reading Assignment 14 Review Exercises

1. A pitot tube used in a Method 2 traverse was damaged just before the test. Mike told Jeff to go ahead and traverse anyway, since the data could be corrected later on. If the original calibration value were $C_p = 0.82$, the recalibrated value were $C_p = 0.78$, and the average stack gas velocity were 41.2 ft/sec, what would the corrected velocity be?
   a. 39.2 ft/sec
   b. 43.3 ft/sec
   c. 32.1 ft/sec
   d. 33.7 ft/sec

2. Jeff did the Orsat analysis for the second time in his life. He told Mike that he got a value of .4% for CO_2 and 6% for O_2. The plant was burning anthracite coal. Was Jeff correct?
   Yes _____
   No _____
   How do you know? _____

3. Jeff did the Orsat experiment over again and this time got a value of 14% for CO_2 and 5% for O_2. What would the F_o factor be from this data?
   a. 1.070
   b. 1.380
   c. 1.492
   d. 1.136

4. Is the F_o factor calculated within ±2.9% of the midpoint F_o value for anthracite?
   a. No. It is 6%; but its close enough, so the data should be accepted.
   b. Yes. It is within the tabulated range; the data should be accepted.
   c. No. It is 6%; the data should be checked and/or the test redone.
   d. No. It is 6%; the data should be rejected and the value estimated from a nomograph.

5. What are the precision limits and posttest deviations for the following pieces of equipment?

	Pretest calibration precision	Posttest deviation limit
Dry gas meter thermometer	± 5.4°F (3°C)	
Dry gas meter correction factor	± 0.02Y	
Field barometer		± 5 mm
Stack temperature sensor		

6. In the treatment of Method 5 acetone rinses, in no case should a blank residue greater than ____% of the blank weight be subtracted from the sample weight.
   a. 1
   b. 0.1
   c. 0.01
   d. 0.001

7. Is each of the following true or false?
   a. Acetone rinses may be evaporated at room temperature only.
   b. Sample filters may be oven dried at 220°F for 2 to 3 hours.
   c. Acetone leakage can't be detected because you can't mark on polyethylene bottles.
   d. Weighing to constant weight means weighing to a difference (between two consecutive weighings) of ≤0.5 mg with a minimum of six hours of desiccation between weighings.

Answers to
Reading Assignment 14 Review Exercises

1. (a) b c d

$$v_s = C_p\sqrt{\frac{T_s\Delta p}{M_s P_s}}$$

$$\frac{v_s^{corr}}{v_s} = \frac{C_p^{corr}}{C_p}$$

$$v_s^{corr} = \frac{C_p^{corr}}{C_p} v_s$$

$$v_s^{corr} = \frac{0.78}{0.82} 41.2 = 39.2$$

2. Yes. According to the figure on page 174 of this course manual, at a 6% O_2 flue gas level, the $\%CO_2$ should be about 14%.

3. a b c (d)

$$F_o = \frac{20.9 - \%O_2}{\%CO_2}$$

$$= \frac{20.9 - 5}{14} = 1.136$$

4. a (b) c d

5.

	Pretest calibration precision	Posttest deviation limit
Dry gas meter thermometer	±5.4°F (3°C)	10.8°F (6°C)
Dry gas meter correction factor	±0.02Y	< ±5%
Field barometer	±2.5 mm (0.1 in.)	±5 mm
Stack temperature sensor	±1.5% at three temperatures	±1.5% at ambient temperature

6. a b c (d)

7. a. False
   b. True
   c. False
   d. True

Reading Guidance—Assignment 15

Begin by reading Section 3.5.5—Postsampling Operations for Method 6. Read pages 1 of 16 through 16 of 16.

Subsection 5.1 (paragraphs 5.1.1 and 5.1.2) reviews some of the posttest calibration checks which have already been discussed. The important part of this reading assignment is Subsection 5.2, *Analysis* (Base Laboratory).

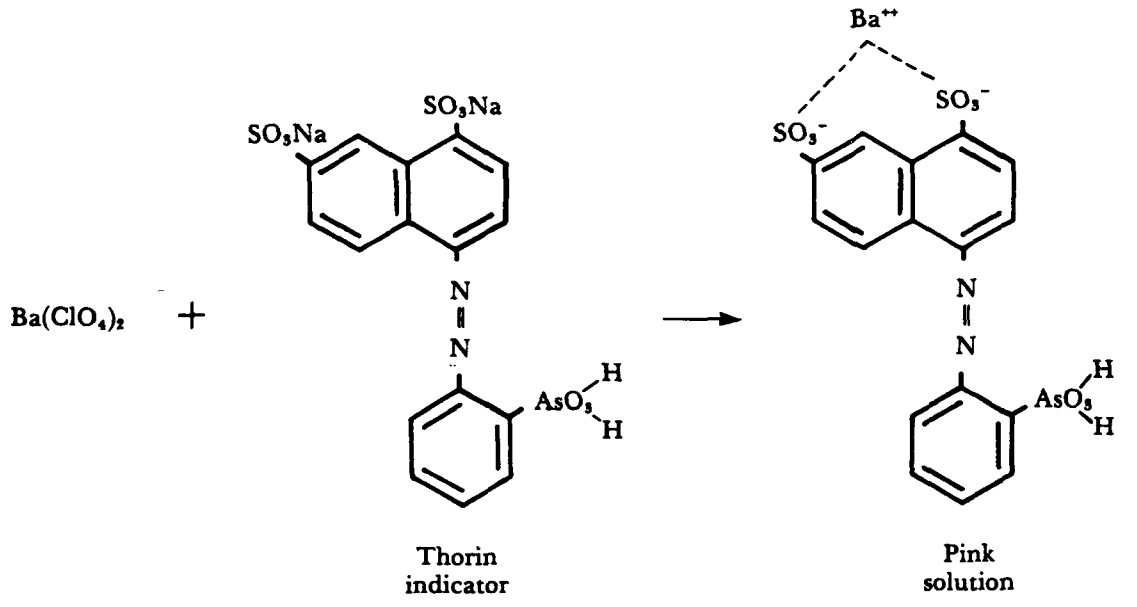
Subsection 5.2 provides detailed instructions for accurately performing the barium-thorin titration method for sulfate analysis. Procedures for the standardization of reagents, analysis of control samples, and analysis of the stack sample are given. The procedures are:

$Ba(ClO_4)_2$ Standardized (paragraph 5.2.4)	+	H_2SO_4 Standardized (paragraphs 5.2.2 and 5.2.3)	→	$BaSO_4\downarrow + 2\ HClO_4$
		$(NH_4)_2SO_4$ Control sample (paragraph 5.2.5)	→	$BaSO_4\downarrow + 2\ NH_4ClO_4$
		H_2SO_4 Stack sample (paragraph 5.2.6)	→	$BaSO_4\downarrow + 2\ HClO_4$

After just enough barium perchlorate solution is added to precipitate the sulfate in the sample, the next drop added will produce:

$$Ba(ClO_4)_2 \rightarrow Ba^{++} + 2\ ClO_4^-$$

If thorin indicator is in the sample solution, we then have:



In paragraph 5.2.1, a note is given in the discussion about water. Distilled deionized water meeting ASTM specifications is particularly important in this method.

It is highly recommended that manufacturer-guaranteed sulfuric acid be purchased for the barium perchlorate standardization. Diluting concentrated sulfuric acid and standardizing it with NaOH which has to be standardized with potassium acid phthalate is a procedure which can take a day or more. Purchased, standardized H_2SO_4 need merely be diluted up to some known volume in a volumetric flask—a procedure which takes only about 20 minutes. Also, the errors involved in the standardization of H_2SO_4 made up from concentrated reagent will in the end be much greater than the error in the value given for manufacturer-guaranteed H_2SO_4.

If you do use purchased H_2SO_4 solutions, you may skip reading paragraphs 5.2.2 and 5.2.3 of this reading assignment.

In paragraph 5.2.1, in the preparation of the $Ba(ClO_4)_2$ solution, it is noted that the solution should be protected from evaporation. This is an important point. Note, also, that $BaCl_2 \cdot 2\ H_2O$ may be used instead of the perchlorate, if desired.

Paragraph 5.2.4 gives the procedures for standardizing the barium perchlorate. The preparation and analysis of blanks is important in all analytical procedures. A blank analysis checks the purity of your reagents. Note item 3 of pararaph 5.2.4 in this regard and the limit of 0.5 ml of titrant.

The color change in the barium-thorin titration is not as sharp as the more familiar phenolphthalein-acid base titration. For those unfamiliar with the color changes of the thorin indicator, some practice is necessary. In the note of

paragraph 5.2.4, Volume III gives some suggestions how this can be done. Some other tricks that can be used in obtaining consistent results are listed here:

1. Put a white sheet of paper behind the burette and sample.
2. Illuminate the work area or back-light the white paper with fluorescent light. Don't use incandescent light.
3. Obtain an endpoint on a control sample or blank and titrate to the same color in succeeding samples by keeping the control sample close by.

The color intensity is not as important as is achieving the same color intensity for all titrations. **Consistency** is the key in this regard.

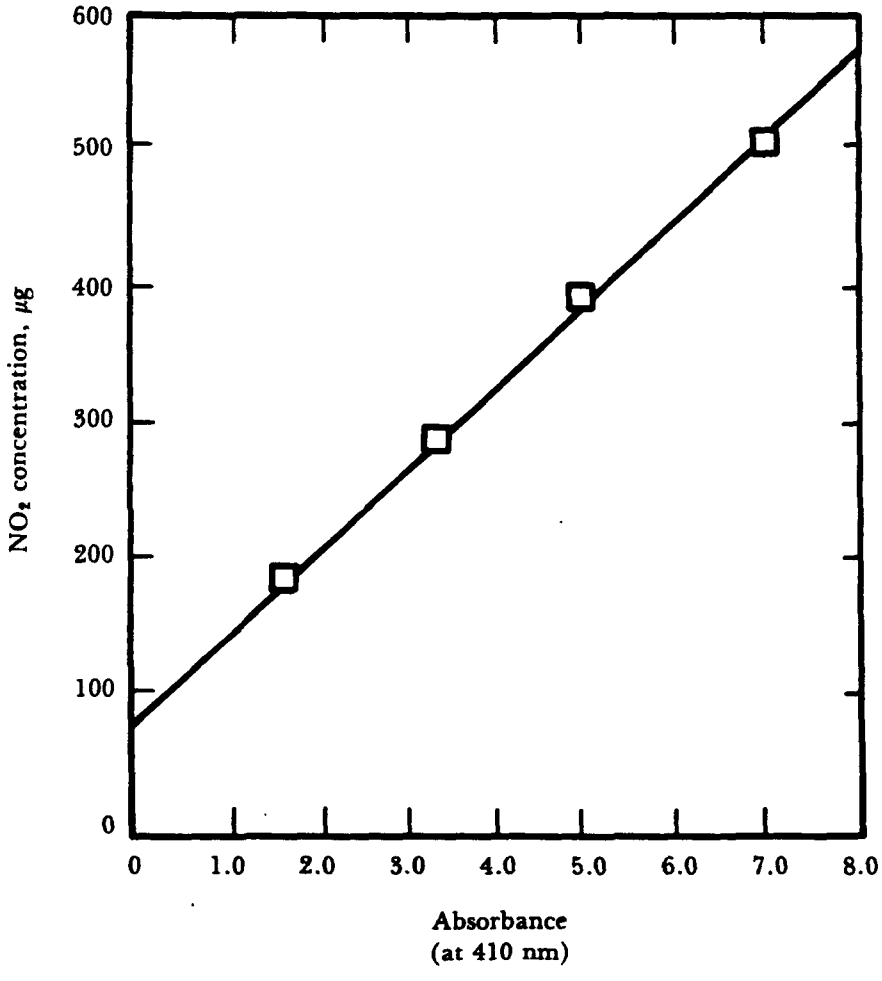
Control samples are very important as part of quality assurance activities of an analytical laboratory. Paragraph 5.2.5 gives the procedures for preparing and analyzing Method 6 control samples, and gives criteria for checking the technique of the analyst (items 10 and 11). The analysis of control samples should be an accepted activity in the laboratory and should not be by-passed for the sake of expediency.

We finally get to the analysis of the sample in paragraph 5.2.6. Once the analyst shows that he can do the analysis within the accepted limits, he can then go on and do his stack sample.

Continue your reading with Section 3.6.5—Postsampling Operations for Method 7. Read pages 1 of 10 through 10 of 10.

The analytical procedures for Method 7, which are given in Volume III, incorporate the use of control samples. The analysis of control samples is not required in the Federal Register, but is recommended in Volume III to help in validating data.

The control samples and working standards are made up and analyzed the same way. The difference between them comes about in the calculation procedures which are outlined in Figure 1 on page 6 of 10 (Section 3.6.5). The working standards are used to develop a calibration curve such as that shown below.



The least squares constant, K_c, of the calibration curve is obtained by using the formula given in Figure 5.1 on page 6 of 10 (Section 3.6.5). This value of K_c is then used to calculate an absorbance for each of the control samples at 100, 200, and 300 μg. This absorbance is then compared with the *actual* measured absorbance of the control samples. Volume III recommends that calculated and measured values should agree to within 15%.

In the more recent discussion on quality assurance procedures for Methods 6 and 7 (Shigehara, R.T. and Curtis, F. 1982, "Methods 6 and 7 Quality Assurance/Control Background Information", *Source Evaluation Society Newsletter* Vol. VII, No. 1, February 1982, pp. 15-25), it is recommended that for the working standards, K_c be multipled by each working standard absorbance to give

$$K_c \times OD_{meas} = \mu g_{calculated}$$

and that

$$\frac{\mu g_{calculated} - \mu g_{actual}}{\mu g_{actual}} < 7\%.$$

The analytical procedures for Reference Method 7 are quite straightforward. However, to obtain good precision and accuracy in the method, it must be performed by an **experienced** and **meticulous** analyst.

In order to make the analytical procedures of Volume III a little easier for you to follow, a review of the Method 7 chemical reaction sequence is provided here.

Reactions in sample flask at time of source test

$$\underset{\text{absorbing solution}}{NO + H_2O_2} \rightarrow NO_2 + H_2O$$

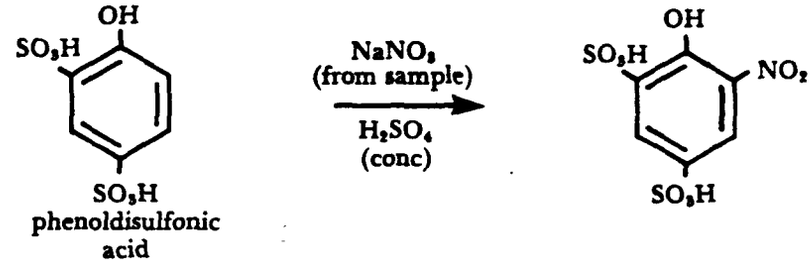
$$2\ NO + O_2 \rightarrow 2\ NO_2$$

$$\underset{\text{formed from NO or } NO_2 \text{ in stack gas}}{2\ NO_2 + H_2O_2} \rightarrow 2\ HNO_3$$

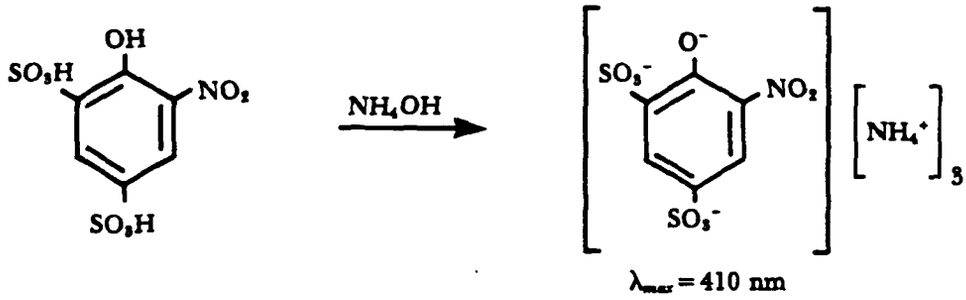
Salt formation on evaporating dish—lab analysis

$$HNO_3 + NaOH \rightarrow NaNO_3 + H_2O$$

Nitration step:



Color developing step:



$\lambda_{max} = 410$ nm

Note that in these procedures a blank solution is to be analyzed along with the control working standards and sample. The blank corresponds to the 0.0 pipetted sample alluded to in item 9 on page 3 of 10 (Section 3.6.5).

> You have completed your reading for Assignment 15. You may wish to read optional Section 3.7.5—Post-sampling Operations for Method 8. Much of this material has been covered in the Method 6 discussion.
>
> Do the review exercises which follow and check your answers after you complete them. The correct answers are given on the page following the review exercises.

Reading Assignment 15 Review Exercises

1. The barium perchlorate solution prepared for Method 6 analysis procedures is standardized with a sulfuric acid solution. How do you know the normality of the sulfuric acid solution?
   a. You can use a manufacturer-guaranteed solution and use the value stated.
   b. You can use a manufacturer-guaranteed solution and dilute it in a volumetric flask to the required value.
   c. You can use concentrated sulfuric acid diluted in a volumetric flask and then standardize the solution with an NaOH solution which has been standardized with a potassium acid phthalate solution.
   d. all of the above
   e. c only
2. What is the primary precaution that must be taken with the barium perchlorate solution.
   a. It must be kept out of the sun.
   b. It must be protected from evaporation.
   c. It must be used within two weeks from preparation.
   d. It must be refrigerated.

3. A blank solution of 25-ml deionized distilled water in 100 ml of isopropanol is titrated with 0.01 N (nominal) barium perchlorate solution in the barium perchlorate standardization procedure. If the thorin indicator turns the solution pink after 0.8 ml of 0.01 N barium perchlorate solution have been added, what should be done?
   a. Everything is fine and you can proceed with the standardization.
   b. The isopropanol is obviously contaminated with sulfate and must be replaced.
   c. Only 0.5 ml of titrant is allowed. The distilled water is not pure enough and must be replaced.
   d. The thorin indicator decomposed and must be replaced.

4. SST analysis of the third Method 6 sample from a recent test of Brimstone Power was accomplished by pipetting a 20-ml aliquot of the recovered H_2O_2 impinger fraction into a 250-ml Erlenmeyer flask, adding 80 ml of 80% IPA and three drops of thorin indicator, and titrating to a pink endpoint. Was this done correctly?
   a. No. The impinger catch was not diluted to a known volume of 100 ml in a volumetric flask.
   b. No. 100% isopropanol must be used because thorin is active only in an 80% alcohol solution.
   c. No. 20 ml of thorin indicator are required to give the proper color.
   d. Yes. The procedure was followed correctly.

5. Control samples for Method 6 analysis are prepared using:
   a. primary standard grade $BaCl_2 \cdot 2\ H_2O$.
   b. primary standard grade $(NH_4)_2SO_4$.
   c. manufacturer-guaranteed H_2SO_4.
   d. primary standard grade $Ba(ClO_4)_2$.

6. What is the recommended frequency for the analysis of control samples?
   a. two control samples before source sample analysis and two control samples after the last source sample is analyzed
   b. two control samples anytime during the day on which the source samples are analyzed
   c. one control sample before and one after the analysis of the source samples
   d. two control samples after the last collected source sample is analyzed each analysis day

7. Replicate titrant volumes for a Method 6 analysis must agree within:
   a. 1% or 1 ml, whichever is smaller.
   b. 0.2% or 1 ml, whichever is greater.
   c. 0.1% or 2 ml, whichever is smaller.
   d. 1% or 0.2 ml, whichever is greater.

8. In Reference Method 7, control samples and working standard solutions are:
   a. prepared in the same manner.
   b. analyzed in the same manner.
   c. used interchangeably.
   d. all of the above
   e. a and b, only

9. A value of 568.0 was found for the value of K_c using the Method 7 working standards. The Control Sample S2, corresponded to 200 μg NO_2. The measured absorbance of the control sample was equal to 0.321. Does this control sample meet the recommended accuracy limit?
   a. Yes. % difference = 8.81
   b. Yes. % difference = 18.8
   c. No. % difference = 8.81
   d. No. % difference = 18.8

10. Which of the following analytical sequences is correct for Reference Method 7?
   a. • NO_x reactions with peroxide in flask
      • spectrophotometric determination of phenoldisulphonic acid standard
      • sodium salt formation on evaporating dish
      • nitration of phenoldisulphonic acid
      • titration of complex
   b. • NO_x reactions with peroxide in flask
      • titration of nitrate with thorin
      • sodium salt formation on evaporating dish
      • nitration of phenoldisulphonic acid
      • formation of colored complex
      • spectrophotometric determination of colored complex
   c. • NO_x reactions with peroxide in flask
      • sodium salt formation on evaporating dish
      • nitration of phenoldisulphonic acid
      • formation of colored complex
      • spectrophotometric determination of colored complex
   d. • NO_x reactions with peroxide in flask
      • formation of colored complex
      • sodium salt formation on evaporating dish
      • nitration of phenoldisulphonic acid
      • spectrophotometric determination of colored complex

Answers to
Reading Assignment 15 Review Exercises

1. a b c (d) e
2. a (b) c d
3. a b (c) d
4. (a) (b) c d
5. a (b) c d
6. (a) b c d
7. a b c (d)
8. a b c d (e)
9. (a) b c d

$$OD = \frac{\mu g}{K_c} = \frac{200}{568} = 0.352$$

Absorbance comparison error:

$$\% = \frac{0.321 - 0.352}{0.352} \times 100$$

$$\% = -8.81$$

10. a b (c) d

You have now completed the material for **Quiz 2.**

Take **Quiz 2** under the direction of your test supervisor. (See page 5 of this guidebook for more detailed instructions.)

Calculations—Maintenance—Audits

Lesson H

Calculations

Lesson Goal

The goal of this lesson is for you to understand that correct calculation procedures are essential if one is to achieve valid test results.

Lesson Objectives

After completing this lesson, you should be able to:

1. properly round off numbers calculated in the reference methods and carry out calculations to the appropriate number of significant figures.
2. list at least four types of errors that frequently occur in source sampling calculations.
3. explain why it is important to use data expressed in the proper units when performing calculations specified in the reference methods.
4. discuss the relative importance of data errors with respect to final calculated values, for a number of example calculations.
5. discuss the use of computers or hand-held calculators in performing reference method calculations.

Materials

Assignment 16

- Section 3.1.6, Calculations in Section 3.1
  Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate
- Section 3.2.6, Calculations in Section 3.2
  Method 3—Determination of CO_2, O_2, Excess Air, and Dry Molecular Weight
- Section 3.3.6, Calculations in Section 3.3
  Method 4—Determination of Moisture in Stack Gases
- Section 3.4.6, Calculations in Section 3.4
  Method 5—Determination of Particulate Matter from Stationary Sources
- Section 3.5.6, Calculations in Section 3.5
  Method 6—Determination of Sulfur Dioxide from Stationary Sources
- Section 3.6.6, Calculations in Section 3.6
  Method 7—Determination of Nitrogen Oxide Emissions from Stationary Sources

Reading Guidance—Assignment 16

Begin by reading Section 3.1.6—Calculations for Method 2. Read pages 1 of 4 through 4 of 4.

This reading assignment consists of each of the short sections on calculations for the reference methods. The first subsection, 6.0, is much the same for all of the reference methods, so after reading it once, you can quickly skim through it on the others.

It is assumed that you are already familiar with the reference method equations. The goal of this lesson is not to provide background in their derivation or to give you practice in using them. The goal is to point out Volume III techniques that can reduce calculation errors.

Many source tests have been invalidated in the past because of calculation errors only. Considering the time, expense, and difficulty in conducting the reference methods, errors arising from calculations should be explicitly avoided.

With the advent of hand-held calculators and desk-top computers, programs can be and have been developed which can quickly perform the required reference method calculations. See for example: Ragland, J.W. et. al. 1976. *HP-65 Programmable Pocket Calculator Applied to Air Pollution Measurement Studies: Stationary Sources.* EPA 600/8-76-002, October 1976.

Errors still can arise with the use of programmable calculators or computers. It is good practice to punch in an example problem for which the answer is known to see if the program and calculator are working properly. A number of example calculations are given in the calculation sections of Volume III. Punching wrong numbers into a calculator will give wrong results. As with all other aspects of source sampling, care should be taken in this part of the process.

Note in Subsection 6.1 that a nomenclature list is provided, giving the units for each parameter in both metric and English terms. It is important that data be expressed in the correct units before being substituted into the method equations. Many of the equations contain constants which are dependent upon the system of units used. For example, the average stack gas velocity is calculated in units of feet per second if $(\Delta p)_{avg}$ is in inches of water, and $K_p = 85.49$.

Continue your reading with Section 3.2.6—Calculations for Method 3. Read pages 1 of 3 through 3 of 3.

Continue your reading with Section 3.3.6—Calculations for Method 4. Read pages 1 of 8 through 8 of 8.

Example calculations are given in Figures 6.1A and 6.1B on pages 4 of 8 and 5 of 8 of Section 3.3.6. Note the spaces denoting the number of significant figures that are to be used. This aid is provided for you in the calculation discussions of Methods 3, 6, 7, and 8 in Volume III. You may wish to develop similar forms for the other reference method calculations or for calculations not addressed in Volume III.

The following rules are used for determining the number of significant figures in a number:

1. Disregard all initial zeros.
2. Disregard all final zeros unless they follow a decimal point.
3. The remaining digits are significant.

Volume III recommends carrying out calculations to one decimal figure beyond the acquired data. Rounding off numbers is accomplished according to the following rules:

1. If the succeeding digit is greater than five, round off to the next highest number.
2. If the succeeding digit is less than five, drop the digit.
3. If the succeeding digit is equal to five and the preceding number is odd, round off to the next even number.
4. If the succeeding digit is equal to five and the preceding number is even, drop the five.

Examples: round off to three significant figures:

2.168→2.17 rule 1
2.153→2.15 rule 2
2.155→2.16 rule 3
2.165→2.16 rule 4

Continue your reading with Section 3.4.6—Calculations for Method 5. Read pages 1 of 10 through 10 of 10.

It is assumed that you are already familiar with the nomenclature of source sampling. If the symbols used in the calculations are new to you, just realize that they are similar to the words of a new language. Each symbol means something and is accompanied by a specific set of units (metric or English). As with all languages, the more that one uses it, the fewer mistakes he or she will make.

The isokinetic variation equation given on page 8 of 10 in Section 3.4.6 is very sensitive to roundoff error. Care should be taken in keeping the proper number of significant figures throughout the calculation (particularly in the value of A_n, the nozzle area, which is to be expressed in units of square feet). An example problem addressing this situation is given in the review exercises of this lesson.

Continue your reading with Section 3.5.6—Calculations for Method 5. Read pages 1 of 6 through 6 of 6.

Then go on to Section 3.6.6—Calculations for Method 7. Read pages 1 of 6 through 6 of 6.

A recent article has reviewed types of errors which can occur in Methods 6 and 7. (Shigehara, R. and Curtis, F. February 1982. "Methods 6 and 7 Quality Assurance/Control Background Information". *Source Evaluation Society Newsletter* Volume III, No. 1.) By evaluating several hundred audit sample reports, the authors found that approximately 10% of the reports contained some type of mistake in the calculations. The most common problems were: using the wrong normality for the barium standard in Method 6, using the wrong aliquot factors in Method 7, and making decimal-point errors. Fortunately, many of these errors can be corrected once they are detected.

Using certified samples and having organization management conduct independent reviews can help to assure that the calculations in the final report are done properly.

You have completed your reading for Assignment 16. Do the review exercises which follow and check your answers after you complete them. The correct answers are given on the page following the review exercises.

Reading Assignment 16 Review Exercises

1. How many significant figures are in each of the following?
   a. 304.0 _____
   b. 9000 _____
   c. 0.114 _____
   d. 0.007 _____
   e. 280.3 _____

2. Round off each of the following numbers to three significant figures.
   a. 714.2 _____
   b. 1725 _____
   c. 0.1432 _____
   d. 9.135 _____
   e. 9147.2 _____

3. State the units for each of the following symbols.
   a. ΔH, Δp, P_{std} _____ _____ _____
   b. V_m, v_s, V_{lc} _____ _____ _____
   c. A, A_n _____ _____ _____
   d. B_{ws}, Y, I _____ _____ _____

4. Mike recorded the sampling nozzle diameter as 0.263 inches during a source test. He later used a value of 0.0004 ft^2 for the nozzle cross-sectional area in order to calculate percent Isokinetic (%I). What is the problem here?
   a. There is no problem—roundoff is done correctly.
   b. The rounded-off A_n will give a %I that is 6% too high.
   c. The rounded-off A_n will give a %I that is 6% too low.
   d. The value of A_n should be expressed in $in.^2$.

5. Because of high SO_2 concentrations in the source, it was necessary for Super Stack Testers (SST) to cut their H_2O_2 samples for analysis. In the final report, the final dilution scheme was documented:

	Original volume (ml)	Aliquot size (ml)	Aliquot diluted to ml	Final sample size for analysis (ml)	Final dilution factor	Correct dilution factor
Run #1	100	10	250	20	125	_____
Run #2	100	10	100	10	100	_____
Run #3	250	20	100	25	50	_____

What should the correct dilution factors be?

Answers to
Reading Assignment 16 Review Exercises

1. a 4
   b. 1
   c. 3
   d. 3
   e. 4

2. a. 714
   b. 1720
   c. 0.143
   d. 9.14
   e. 9150

3.

a.	mm (in.) H_2O	mm (in.) H_2O	mm (in.) Hg
b.	dcm (dcf)	m/s (ft/sec)	ml
c.	m^2 (ft^2)	m^2 (ft^2)	
d.	dimensionless	dimensionless	%

4. a b (c) d

$$\%I = K_4 \frac{T_s V_m(std)}{\theta v_s P_s A_n (1 - B_{ws})}$$

Therefore

$$\frac{\%I_{(true)}}{\%I_{(rounded\ off)}} = \frac{A_{n(true)}}{A_{n(rounded\ off)}}$$

$$\%I_{(true)} = \frac{A_{n(true)}}{A_{n(rounded\ off)}} \%I_{(rounded\ off)}$$

Since

$$A_n = \pi r^2$$

$$= 3.14 \frac{0.263}{2} \cdot \frac{1}{12}^2$$

$$= 0.0003771\ ft^2$$

Then

$$\%I_{(true)} = \left(\frac{0.0003771}{0.0004}\right) \%I_{(rounded\ off)}$$

$$\%I_{(true)} = 0.94\ \%I_{(rounded\ off)}$$

or the true %I is 6% less than the %I calculated by rounding off A_n to 0.0004

5.

	Correct dilution factor
Run #1	125
Run #2	OK
Run #3	50

Lesson I

Maintenance Checks

Lesson Goal

The goal of this lesson is for you to understand the procedures recommended in Volume III for the routine maintenance of source sampling equipment.

Lesson Objectives

After completing this lesson, you should be able to:

1. explain why routine maintenance is required on source sampling equipment.
2. list at least two items which should be checked for on each of the following:
   - pitot tube
   - fiber vane pump
   - dry gas meter
   - sampling train glassware

Materials

Assignment 17

- Section 3.1.7, Maintenance in Section 3.1
  Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate
- Section 3.2.7, Maintenance in Section 3.2
  Method 3—Determination of CO_2, O_2, Excess Air, and Dry Molecular Weight
- Section 3.3.7, Maintenance in Section 3.3
  Method 4—Determination of Moisture in Stack Gases
- Section 3.4.7, Maintenance in Section 3.4
  Method 5—Determination of Particulate Matter from Stationary Sources
- Section 3.5.7, Maintenance in Section 3.5
  Method 6—Determination of Sulfur Dioxide from Stationary Sources
- Section 3.6.7, Maintenance in Section 3.6
  Method 7—Determination of Nitrogen Oxide Emissions from Stationary Sources.

Reading Guidance—Assignment 17

This is a relatively short reading assignment. The Volume III discussions on maintenance are much the same for Reference Methods 2 through 8, so they can be read rather quickly. Routine maintenance is an important part of any quality assurance program. A testing organization with a busy schedule may often neglect such maintenance and then have problems on site. The Volume III guidelines for maintenance are intended to provide a start for the development of your own program. Similar procedures can be extended to laboratory balances, spectrophotometers, continuous monitoring equipment, impactor systems, and so on.

Begin by reading Section 3.1.7—Maintenance for Method 2. Then read the Maintenance Sections

3.2.7—Method 3
3.3.7—Method 4
3.4.7—Method 5
3.5.7—Method 6
3.6.7—Method 7

You have completed your reading for Assignment 17. Do the review exercises which follow and check your answers after you complete them. The correct answers are given on the page following the review exercises.

Reading Assignment 17 Review Exercises

1. How often does Volume III recommend that routine maintenance checks be conducted for a sampling train?
   a. quarterly or after 1000 ft^3 of operation
   b. yearly or after 500 ft^3 of operation
   c. semi-annually or after 1000 ft^3 of operation
   d. every month

2. A fiber vane pump requires a periodic check of:
   a. oil level.
   b. oil appearance.
   c. oiler jar.
   d. all of the above

3. The dry gas meter of a sampling train should be checked for corrosion and excess oil:
   a. every month.
   b. every three months.
   c. every year.
   d. after every test.

4. Jeff was doing a Method 6 test when the ball in the rotameter stuck. In desperation, he stopped the run and cleaned out the rotameter with acetone. Was this a good thing to do?

   Yes _____

   No _____

   Why or why not? _____

5. Jeff had been in a hurry before going out to test Acme Power. He forgot to bring the condensing system for his Method 8 apparatus. Not wanting to go back or admit his forgetfulness, he hooked up his diaphragm pump directly to the probe to get his bag sample. What might be found in a routine check of the pump?
   a. oil in the pump from backup oil in the jar
   b. nothing—there should be no problem since diaphragm pumps are maintenance free
   c. corrosion of the diaphragm head because of condensation and acid deposition
   d. burned stator because of too high a motor speed

Answers to
Reading Assignment 17 Review Exercises

1. (a) b c d
2. a b c (d)
3. a (b) c d
4. No. Acetone could react with the plastic of the rotameter.
5. a b (c) d

Lesson J

Auditing Procedures

Lesson Goal

The goal of this lesson is for you to be able to implement auditing procedures in a source test quality assurance program.

Lesson Objectives

After completing this lesson, you should be able to:

1. describe what an audit is.
2. distinguish between a performance audit and a system audit.
3. list the functions of an auditor.
4. describe the auditing device that can be used in Reference Method 5.
5. tell what is in the audit samples commonly used for Reference Methods 3, 6, and 7.
6. list at least four check items that an auditor should observe on site for each of Reference Methods 3, 4, 5, 6, and 7.
7. describe how a source testing laboratory can use audit samples in its quality assurance program.
8. understand the use of percentile rankings in the EPA National Performance Audit Program.

Materials

Assignment 18

- Section 1.4.16, Quality Assuance Manual—Volume I. Audit Procedures. Pages 59 through 63 of this correspondence course manual
- Section 3.1.8, Auditing Procedure in Section 3.1
  Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate
- Section 3.2.8, Auditing Procedure in Section 3.2
  Method 3—Determination of CO_2, O_2, Excess Air, and Dry Molecular Weight
- Section 3.3.8, Auditing Procedure in Section 3.3
  Method 4—Determination of Moisture in Stack Gases
- Section 3.4.8, Auditing Procedure in Section 3.4
  Method 5—Determination of Particulate Matter from Stationary Sources
- Section 3.5.8, Auditing Procedure in Section 3.5
  Method 6—Determination of Sulfur Dioxide from Stationary Sources
- Section 3.6.8, Auditing Procedure in Section 3.6
  Method 7—Determination of Nitrogen Oxide Emissions from Stationary Sources

Reading Guidance—Assignment 18

This reading assignment is the last one in this correspondence course. The subject of auditing procedures is appropriate in completing this study of Volume III, since a proper audit will review all phases of the source test—from the pretest preparations to the on-site measurements and final calculations. Since it probably has been some time since you first started this course, let us begin this assignment by reviewing Lesson A—Reading Assignment 2.

> Begin by reviewing pages 59 through 63 of this correspondence course manual: Audit Procedures—Quality Assurance Manual Volume I Section 1.4.16.

Note especially the definitions given for performance audits and system audits. Much of this information is general. More specific procedures for source sampling methods are, of course, given in Volume III.

In the past, quality assurance and the use of audit samples focused on analytical laboratory practices. Obtaining a representative source sample for the analysis is as important as properly performing the analysis. In a source test, the source itself must be operating in a representative manner, the sampling site must be representative, and the test samples must be obtained in a representative manner. Performance or system audits need to be conducted for this whole setup. For this reason, in each of the reference method Audit Procedure sections which you are about to read, checklists are provided for the auditor or inspector. Each phase of the test, pretest, on-site measurements, postsampling, etc., have checkpoints which the auditor can use to evaluate the performance of the test. These points can provide a start for the development of your own auditing program.

> Continue your reading with Section 3.1.8—Auditing Procedure for Method 2. Read pages 1 of 5 through 5 of 5.

Note especially the four functions of the auditor summarized on page 2 of 5.

Many forms and checklists have been developed for the auditor or source test field inspector. Example forms are given throughout Volume III, one of which is

Figure 8.1 on page 4 of 5, Section 3.1.8. Other formats which could be adopted in your quality assurance program can be found in:

Industrial Guide for Air Pollution Control—Handbook. June 1978. EPA 625/6-78-004.

Source Sampling Administration Manual. Volume III—EPA Stationary Source Enforcement Series. November 1977.

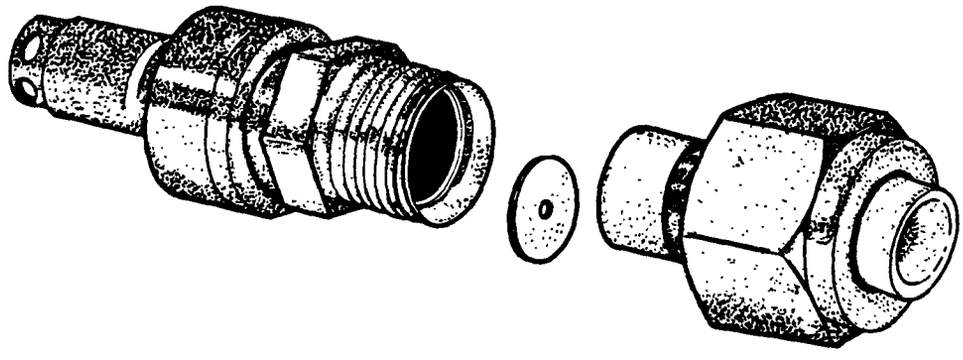
Continue your reading with Section 3.2.8—Auditing Procedure for Method 3. Read pages 1 of 5 through 5 of 5.

Certified gases can be used as audit materials for Reference Method 3. Note the discussion on page 1 of 5, Section 3.2.8, paragraph 8.1.2. Note the recommended difference value, D, of 1% or less, given for correspondence with the audit materials.

Continue your reading with Section 3.3.8—Auditing Procedure for Method 4. Read pages 1 of 4 through 4 of 4.

Then go on to Section 3.4.8—Auditing Procedure for Method 5. Read pages 1 of 7 through 7 of 7.

The audit procedures for Method 5 introduce the use of a calibrated critical orifice. The orifice plate is incorporated into a quick-connect coupling which is in turn inserted into the gas inlet of the meter box. The assembly is shown in the figure below.



Construction and calibration details of this audit device are given in: Mitchell, W.J. et al. 1981. New Orifice Opens Way For Fast Calibration. *Pollution Engineering*. June 1981. pp. 45-47.

The Environmental Protection Agency has conducted a National Performance Audit Program yearly since 1977. The program has involved sending audit samples for Methods 6 and 7 and the critical orifice device for Method 5 to source testing organizations across the country. The testing organizations use these audit devices

in their laboratories and then report their results to EPA. Part of the results of the program are given on page 3 of 7 of Section 3.4.8, paragraph 8.1.1. They show that 90% of the testing laboratories can come within 10% of the audit standard value. More detailed results of these surveys are given in the following:

Fuerst, R.G. et al. *A summary of the Interlaboratory Source Performance Surveys for EPA Reference Methods 6 and 7—1977.* EPA 600/4-79-045. August 1979.

Fuerst, R.G. and Midgett, M.R. *A summary of the Interlaboratory Source Performance Surveys for EPA Reference Methods 5, 6, and 7—1978.* EPA 600/4-80-029. May 1980.

Fuerst, R.G., Streib, E.W., and Midgett, M.R. *A Summary of the EPA National Source Performance Audit Program*—1979. EPA 600/4-81-029. April 1981.

The percentile ranges are given as a guide to show what level of correlation can be obtained using the audit devices. Note that audit samples and devices are intended to be used as an aid to assess the quality of source test data. Care should be taken in specifying a given number for %A such as "all audit samples analyzed must agree within 1% of the standard value, or else the test is rejected", since such a specification may be meaningless with respect to the overall test accuracy. However, if 90% of the laboratories which have analyzed audit samples agree to within, say, 5% of the sample's known value, a laboratory presenting a 15% discrepancy should be questioned about its capabilities.

We have already discussed calculation errors in Lesson H. The auditing of the source test report for calculation errors is one of the most important parts of any quality assurance program. Several techniques can be used to do this. A common one is for an agency or other organization to develop audit computer programs for all of the source test calculations. The raw source test data is then fed into the computer and the results compared to those given in the final report. With the wealth of information printed over the past ten years by the Environmental Protection Agency on source test procedures, and with the advent of inexpensive programmable calculators, there should be no excuse for calculation errors occurring in the reports of a professional source testing organization.

Continue your reading with Section 3.5.8—Auditing Procedure for Method 6. Read pages 1 of 7 through 7 of 7.

Ammonium sulfate solutions can be used as audit samples for Reference Method 6. The precision of Method 6 analytical procedures can be quite good, as is reflected in the percentile rankings on page 3 of 7, Section 3.5.8, paragraph 8.1.1.

A further discussion on the results of the National Performance Audit Program can be found in: Shigehara, R.T. and Curtis, F. 1982. Methods 6 and 7 Quality Assurance/Control Background Information *Source Evaluation Society Newsletter.* Vol. III, No. 1 pp. 15-25.

Continue your reading with Section 3.6.8—Auditing Procedure for Method 7. Read pages 1 of 8 through 8 of 8.

Problems with Method 7 are often the result of poor analytical technique. This is borne out in the percentile rankings given on page 3 of 8, Section 3.6.8, paragraph 8.1.1. Only 80% of the laboratories participating in the audit program were able to get within 15% of the known concentration of the KNO_3 audit samples. It is becoming evident that the problems with Method 7 lie not so much with the method itself, but in the capabilities of the analysts. It has been shown that experienced personnel using quality control procedures can achieve consistently greater precision than that which is reported in Volume III. See for example: Host, A.J. San Diego Gas and Electric CEM QA Program in Specialty Conference Proceedings—Continuous Emission Monitoring—Design, Operation and Experience. *Air Pollution Control Association.* 1981.

You have completed your reading for Assignment 18. Do the review exercises which follow and check your answers after you complete them. The correct answers are given on the page following the review exercises.

Reading Assignment 18 Review Exercises

1. Performance audits are normally:
   a. a qualitative appraisal of data quality.
   b. a quantitative appraisal of quality.
   c. independent checks conducted by the IRS.
   d. used as the basis for conducting a system audit.
2. List two types of performance audits.
   a. _____
   b. _____
3. Which of the following might be used in a system audit?
   a. calibrated critical orifice
   b. Figure 8-1, page 4 of 5, Section 3.1.8
   c. Figure 8-1, page 6 of 7, Section 3.4.8
   d. standard aqueous ammonium sulfate solution

4. List four functions of a source test auditor.

   a. _____

   b. _____

   c. _____

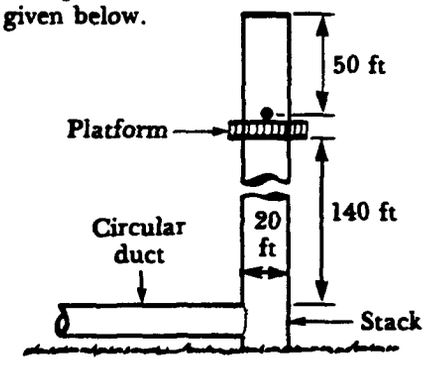
   d. _____

5. Mike analyzed an audit sample before analyzing his Method 6 samples. He was later informed by EPA that his %A was 3.2%. From this information you know:
   a. that Mike could perform the Method 6 titration as well as or better than 90% of the participating laboratories.
   b. that Mike could not perform the Method 6 titration as well as 90% of the participating laboratories.
   c. that Mike's sample had a 10% probability of being correct.
   d. that Mike's sample had a 10% probability of being incorrect.

6. Match the audit sample or device with the proper reference method.

Method 1 ____	a. $(NH_4)_2SO_4$ standard solution
Method 2 ____	b. none
Method 3 ____	c. calibrated orifice
Method 4 ____	d. 12% CO_2 6% O_2 calibration gas
Method 5 ____	e. KNO_3 standard solution
Method 6 ____	
Method 7 ____	
Method 8 ____	

7. Frank was assigned the task of observing a stack test performed by SST at Acme Power. The test involved the determination of particulate and SO_2 emissions. A number of things happened during the test. Frank used the checklists given in the auditing procedure sections for Methods 1 through 6 (Figures 8.1 in Sections 3.1.8, 3.2.8, 3.3.8, 3.4.8, and 3.5.8). For each event, give the potential problem and note whether or not the checklist would have been helpful to Frank in his audit.

Situation	What is the potential problem?	On which checklist would this appear? If on none, where is the situation addressed in Volume III?
a. Test ports were at the location given below.  Twelve traverse points were selected.		

Situation	What is the potential problem?	On which checklist would this appear? If on none, where is the situation addressed in Volume III?
b. The pitot tube C_p for the Type S tube attached to the probe used in the Method 5 sampling, was assumed to have a value of 0.84.		
c. No equipment calibration data were supplied to the observer.		
d. The gasket on the filter holder contained filter fibers after the first run, but the technician scraped all of the fibers off after the second run, using a pocket knife.		
e. During run #2 of the Method 5 test, Super Stack Testers took a break from the sampling routine. The following parameters concerning the sampling train were observed while they were on break. (1) pitot tube at a 30° yaw angle (2) outlet temperature from fourth impinger 78°F (3) water condensation ahead of condenser		
f. The sample jar for the third run of the Method 5 test has a label stating: "Acme Test—SST—Sept. 12, 1979".		
g. In determining the emission rate correction factor for Acme Power and Light Company, the Super Stack Testers opted to use their Fyrite-type combustion gas analyzer rather than their Orsat analyzer because of a time limitation.		
h. The analysis of the moisture content from Method 5 was determined by the following equation: $B_{ws} = \frac{V_{wc}}{V_{wc} + V_{m\,(std)}} + 0.025$		

Answers to
Reading Assignment 18 Review Exercises

1. a (b) c d

2. a. measurement system audit
   b. data processing audit

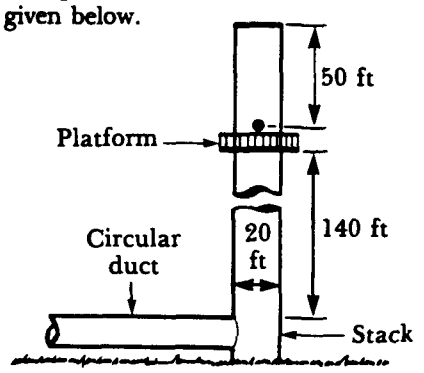
3. a (b)(c) d

4. a. informs test team of pretest audit results
   b. observes procedures and techniques of the field team during sample collection
   c. checks records of apparatus calibration
   d. records results of audit and reports them to test team management

5. (a) b c d

6. Method 1 b
   2 b
   3 d
   4 b
   5 c
   6 a
   7 e
   8 a

7.

Item	Potential problem?	Checklist
a. Test ports were at the location given below.  Twelve traverse points were selected.	Improper number of traverse points selected. Should sample at 18 points (for a circular duct—20).	Not addressed on audit checklists. Would need to refer back to Figures 1.3 and 1.5, Section 3.0.1, page 7 of 19 and page 12 of 19. Should modify audit checklist or develop own form if necessary.
b. The pitot tube C_p for the Type S tube attached to the probe used in the Method 5 sampling, was assumed to have a value of 0.84.	C_p might not have been equal to 0.84 and most probably would actually have been lower. This would lead to higher calculated flow rates and non-isokinetic sampling.	Figure 8.1, Section 3.1.8, page 4 of 5—Form M2-8.1 also Form M2-3.1 (MH), Pretest Sampling Checks or Form M5-4.5 (MH) On-site Measurements Checklist (Section 3.4, page 6 of 15)
c. No equipment calibration data were supplied to the observer.	Uncertainty as to adequacy of equipment for the test and values calculated for sampling rates, emission values, etc.	Method 2—Figure 8.1 Section 3.1.8, page 4 of 5 Form M2-8.1 Method 4—Figure 8.1 Section 3.3.8, page 3 of 4 Form M4-8.1 Method 5—Figure 8.1 Section 3.4.8, page 6 of 7 Form M5-8.1 Method 6—Figure 8.1 Section 3.5.8, page 6 of 7 Form M6-8.1 Method 7—Figure 8.1 Section 3.6.8, page 7 of 8 Form M7-8.1 Method 8—Figure 8.1 Section 3.7.8, page 6 of 7 Form M8-8.1 See also: appropriate forms in Method Highlights sections.

Item	Potential problem?	Checklist
d. The gasket on the filter holder contained filter fibers after the first run, but the technician scraped all of the fibers off after the second run, using a pocket knife.	Filter material on gaskets is to be scraped off and weighed with sample/filter. In the situation presented here, the results of the first run would be biased low. The results of the second run would be biased high.	Figure 8.1 Section 3.4.8, page 6 of 7 Form M5-8.1 (note item 7—On-site Measurements) also M5-4.5 (MH) On-site Measurements Checklist—Section 3.4, page 8 of 15.
e. During run #2 of the Method 5 test, Super Stack Testers took a break from the sampling routine. The following parameters concerning the sampling train were observed while they were on break. (1) pitot tube at a 30° yaw angle (2) Outlet temperature from fourth impinger 78°F (3) water condensation ahead of condenser	(1) An error would occur in velocity determination if in this position when sampling. No problem if data not taken during break. Probe, however, should not have been left in stack. (2) Outlet temperature too high. Specified to be 68°F. (3) Possible net loss in determination of % moisture when measuring water in impingers.	(1) Not directly addressed, but is improper procedure. (2) Not addressed in Form M5-8.1, but should be recorded on Form M5-4.2. (3) Not addressed in Form M5-8.1.
f. The sample jar for the third run of the Method 5 test has a label stating: "Acme Test—SST—Sept. 12, 1979".	Incomplete labeling; possible misidentification of sample in posttest operations.	Figure 8.1 Section 3.4.8, page 6 of 7 Form M5-8.1 (note item 7—On-site Measurements.) (Note also—Form M5-4.3.)
g. In determining the emission rate correction factor for Acme Power and Light company, the Super Stack Testers opted to use their Fyrite-type combustion gas analyzer rather than their Orsat analyzer because of a time limitation.	Improper procedure. Method requires Orsat measurements.	Not addressed in Form M5-8.1. Refer to Reference Methods 3 and 5 procedures.
h. The analysis of the moisture content from Method 5 was determined by the following equation: $B_{ws} = \frac{V_{wc}}{V_{wc} + V_{m\,(std)}} + 0.025$	Not the proper equation for the determination of moisture content. The 0.025 is the fraction associated with the vapor pressure of water at 68°F. Silica gel in last impinger would give dry gas stream.	Figure 8.1 Section 3.4.8, page 6 of 7 Form M5-8.1 Item 12—Postsampling. See also Section 3.4.6, page 7 of 10—Equation 6.3.

One point of this exercise has been to indicate to you that the auditor can make use of more than just the audit forms of Section 8 when observing a source test. The many forms and checklists provided in Volume III in the Method Highlights sections and Data Forms sections can be used when auditing a test. As noted earlier in this handbook, however, these forms should be used with discretion. Requiring a source tester to use a specific form may strain relations between the parties involved in the test. The application of Volume III audit procedures or other procedures should be agreed upon before any testing occurs.

You have now completed all of the reading assignments for this course.

Take the **final examination** under the direction of your test supervisor. (See page 5 of this guidebook for more detailed instructions.)

10/3/90

ERRATA SHEET

CC 414: Quality Assurance for Source Emission Measurement Methods

There are now Reference Methods 3A and 3B. For Methods 3A and 3B respectively, see 40 CFR 60, App. A, Meth. 3A and Federal Register notice Vol 55, p. 05211, Feb. 14, 1990.

For cyclonic flow, an average 20 degree angle of rotation is now acceptable. See 40 CFR 60, App. A, Meth. 1.

p. 196, Reading Assignment 16 Review Exercises. The correct answer to question 1.a., "How many significant figures are in 0.007?" is 1 not 3. This is supported by Rule 1 on p. 194 which states, "Disregard all initial zeros."

TECHNICAL REPORT DATA
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15. SUPPLEMENTARY NOTES

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16. ABSTRACT

This guidebook provides direction for Air Pollution Training Institute Course 414, "Quality Assurance for Source Emission Measurements." It contains reading assignments and review exercises covering the following topics:

Quality Assurance Policy and Programs
Procurement of Source Sampling Equipment
Calibration Methods
Presampling - Sampling - Postsampling Operations
Calculations
Maintenance and Audit Procedures

The Guidebook is designed for use in conjunction with "Quality Assurance Manual for Air Pollution Measurement Systems - Volume III, Stationary Source Specific Methods" (EPA-600/4-77-027b)

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