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Agency

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MD 17
Environmental Research Center
Research Triangle Park, NC 27711

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Air

EPA

APTI Course 470 Quality Assurance for Ambient Air Measurements

Participant Notebook

Notice

This is not an official policy and standards document. The opinions and selections are those of the author 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.

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Student Manual

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Lesson 1

Registration and Course Information

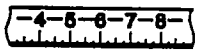
Notes

Lesson 2

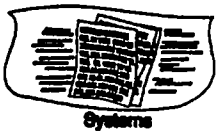
Basic Areas Of Quality Assurance Activities

Basic Areas of Quality Assurance Activities

Management



Measurement



Systems

$$\bar{X} = \frac{\sum X_i}{n}$$

Statistics

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Questions Answered in This Lesson

- How do you define quality assurance?
- What is the function of a quality assurance program?
- What are the four basic areas of quality assurance?
- What specific activities relate to each of the basic areas?

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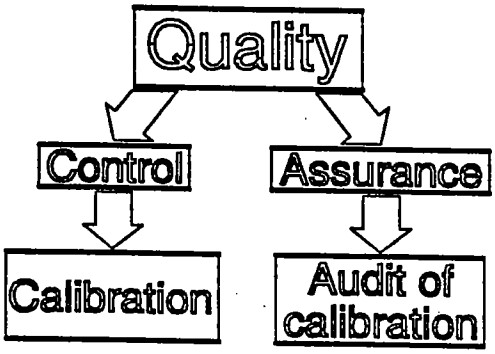

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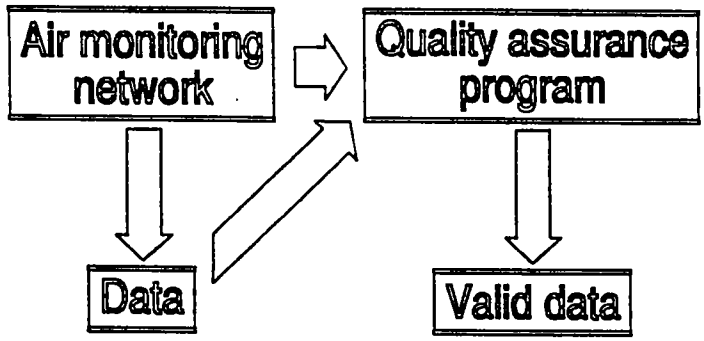

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Monitoring System

Variable	QC Activity
Method	Technical procedure
Materials Machines	Procurement
Maintenance	Preventive/Corrective
Men/Women	Training
Measurement	Calibration procedures Operating procedures

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Monitoring System (cont.)

Variable	QC Activity
Monitoring sites	Conditions
Mathematics	Computations
Management	Objectives Policies Procedures
Meteorology	Siting
Money	Quality costs

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The Quality Assurance Wheel

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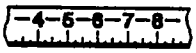
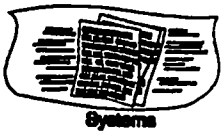
Four Basic Areas of QA

Management



Measurement



Systems

$$\bar{X} = \frac{\sum X_i}{n}$$

Statistics

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Management

- QA policy
- QA objectives
- Organization
- Training
- QA plans
- Quality planning
- Audit planning
- Quality reports
- Corrective action
- Quality costs

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Measurement

- Pretest preparation
- Measurement system reliability
- Calibration (standards traceability)
- Sample collection and analysis
- Audit procedures

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Système International d'Unités (International System of Units, or SI)

Length	Meter (m)
Mass	Kilogram (kg)
Time	Second (s)
Electric current	Ampere (A)
Temperature	Kelvin (K)
Amount of substance	Mole (mol)
Luminous intensity	Candela (cd)

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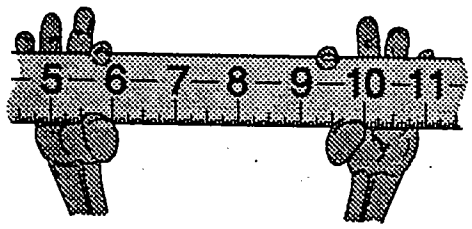
Metrology—the Science of Measurement



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Metrology References

- NIST Special Publication 260-100: Standard Reference Materials: Handbook for SRM Users
- NIST Special Publication 829: Use of NIST Standard Reference Materials for Decisions on Performance of Analytical Chemical Methods and Laboratories
- NIST Special Publications 672: Experimentation and Measurement

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Systems

- Quality planning
- Procurement quality control
- Document control
- Preventive maintenance
- Configuration control
- Data handling
- Data validation
- Performance/System audits
- Corrective action
- Quality costs

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Statistics

- Control charts
- Regression analysis
- Outlier tests

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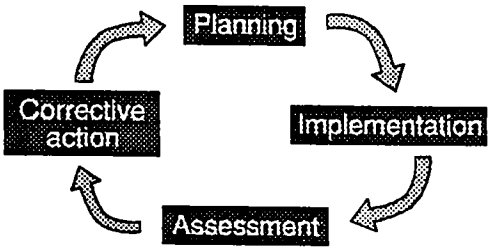
The QA Cycle



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Quality Assurance Management Staff (QAMS)

EPA's mandatory quality assurance program

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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 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 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

CHALLENGES OF IMPLEMENTING QUALITY ASSURANCE FOR AIR POLLUTION MONITORING SYSTEMS

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Director

ABSTRACT

Special considerations are necessary in implementing a quality assurance system for air pollution monitoring. Of particular concern are the following:

(1) Quality characteristics of environmental data.
(2) Network design and sampling.
(3) Measurement methods and standard reference materials.
(4) Statistical quality control
(5) Data analysis and validation.
(6) Preventive maintenance.

Accuracy, precision, completeness and representativeness are the quality characteristics of air monitoring data. The physical sampling of the air environment presents a number of unique and difficult problems. The technology of air pollution measurement has created special demands for measurement methods and standard reference materials. Because of the variability patterns of pollution data, and the nonuniform error variability of the measurement methods, particular types of statistical control and data analysis and data validation are required. The wide diversity in the scope and requirements of compliance and research monitoring makes it necessary to develop flexible quality assurance procedures. In spite of the many difficulties involved, much is being accomplished in implementing quality assurance for air pollution monitoring systems.

INTRODUCTION

With the increased interest and activity in the environment in recent years, a need exists to appy the principles and techniques of modern quality assurance to the various pollution monitoring systems. Pollution measurement methods involve field sampling and chemical laboratory analyses and, to these portions of the measurement process, most of the traditional laboratory quality control (Q.C.) techniques apply. Of concern, however, is the need to apply the general principles and techniques to the entire monitoring system.

The following elements of quality assurance (Q.A.) system are generally applicable to pollution monitoring systems:

Elements of a Quality Assurance System[2,8]

1. Quality Policy
2. Quality Objectives
3. Quality Organization and Responsibility
4. Quality Assurance Manual
5. Quality Assurance Plans
6. Training
7. Procurement Control
   - Ordering
   - Receiving
   - Feedback and Corrective Action
8. Calibration
   - Standards
   - Procedures
9. Internal Q.C. Checks
10. Operations
    - Sampling
    - Sample Handling
    - Analysis
11. Data
    - Transmission
    - Computation
    - Recording
    - Validation
12. Preventive Maintenance
13. Reliability Records and Analysis
14. Document Control
15. Configuration Control
16. Audits
    - On-Site System Audits
    - Performance Audits
17. Corrective Action
18. Statistical Analysis
19. Quality Reporting
20. Quality Investigation
21. Interlaboratory Training
22. Quality Costs

However, in a number of very important areas, special considerations must be made. These areas, which require special attention are:

1. Quality Characteristics of Environmental Data.
2. Network Design and Sampling.
3. Measurement Methods and Standard Reference Materials.
4. Statistical Quality Control.
5. Data Analysis and Validation
6. Preventive Maintenance.

The ultimate uses of air pollution monitoring information are decisions relative to human health and welfare. Air pollution monitoring data are used as measures of air quality to make the best decisions for human health and welfare.

The quality of air is measured by the cleanliness of the air—Are the pollutant concentrations below the levels established as standards? The quality of air pollution data is measured by the accuracy, precision, completeness, and representativeness of the data.

QUALITY CHARACTERISTICS OF ENVIRONMENTAL DATA

These quality characteristics of data may be defined as follows:

1. Accuracy—The closeness of a measured value to the true value.
2. Precision—The repeatability of the data (actually the repeatability of the measurement system).[4]

3. Completeness—The amount of the valid data obtained as a fraction of that intended or planned to be obtained.
4. Representativeness—The typicalness of the pollution samples with respect to time, location, and conditions from which the pollutant data are obtained.

These quality characteristics are not evident nor can they be determined from examination of the data itself. Measures of accuracy, precision, completeness, and representativeness must be obtained from other information. Provision for obtaining measures of these characteristics must be included in the Quality Plan for each monitoring effort because the relative importance of accuracy, precision, completeness, and representativeness depends upon the specific objectives of each monitoring program.

NETWORK DESIGN AND SAMPLING

The monitoring network design, which incorporates decisions with respect to time, location and conditions of sampling, along with the specification of pollution measurement methods and equipment, specify to a large extent the "process" of obtaining monitoring data. Quality assurance personnel should be involved with the network design for pollution monitoring because of the statistical aspects involved, and because of the need to establish the best possible network at the beginning of a monitoring effort. Changes in monitoring networks can destroy the previous history or baseline necessary for trend studies.

The process of media being sampled for air pollution measurement is not in statistical control, but is subject to many effects such as diurnal cycles, day-of-week differences, seasonal cycles, and local and area meteorological factors. The changing pattern of air pollution is a dynamic process, sometimes "out of control."[6] The objective of a quality assurance program for air monitoring is to assure that the *measurement system* remains "in control," no matter what the state or condition of the air.

Consideration for temporal and spatial effects in the location and scheduling of pollution sampling are critical concerns with respect to representativeness.

Planning of the network design and sampling schedules are very important since resampling in air monitoring is impossible. The air which was at the sampling point a moment ago is no longer available! Although duplicate sampling is desirable, in air monitoring, duplicate sampling is not possible for particulates, and is not very practical for gaseous pollutants. The most satisfactory way of duplicate sampling for quality assurance purposes is to use duplicate sampling equipment at the same site. Although such dual sampling requires an additional sampling instrument, this procedure is invaluable in estimating the precision of the total measurement process.

In most chemical analytical work duplicate analyses are desirable. However, for continuous, automated pollution analysis instruments, reanalysis is not possible. Reanalysis is possible for some of the manual methods where bubbler solutions or filter media have been used to collect the pollutants.

MEASUREMENT METHODS AND STANDARD REFERENCE MATERIALS

Most of the manual analytical measurement methods for gaseous pollutants involve bubbling the air through selective absorbing solutions for an extended period (usually 24 hours) and then analyzing the solution by wet chemical/absorbance techniques. These methods have the limitation of providing daily averages only. In the interest of obtaining more accurate measurements on a short-time basis, numerous automated instrumental methods have been developed in recent years. Problems with these instruments include the manufacturing and reliability problems associated with newly-designed equipment, and the technological problems of measuring minute concentrations (parts per million or parts per billion) in the presence of possible interference. Further, problems arise relating the stability and reliability of these instruments if operated remotely or unattended. The development of completely satisfactory measurement methods is a very important effort of quality assurance for air monitoring. Because of the instability of gaseous mixtures, primary standards (Standard Reference Materials of the National Bureau of Standards*) are difficult to prepare, and must be prepared and assessed from time to time as required by users. For some gases (for example, ozone**) no primary standard has yet been developed. Neither has a particulate standard for particle size or chemical content in a naturally-occurring matrix yet been developed.*** Because of the problems in developing and using primary standards for air pollution measurement, the achievement of comparability for accuracy is further magnified when comparability among different countries is considered. In most other physical measurement areas, comparability among nations is relatively easily achieved through traceability to common primary standards.

* The National Bureau of Standards is now the National Institute of Standards and Technology (NIST).

** For federally mandated monitoring, EPA specifies a UV photometric procedure for determining the concentration of ozone calibration gas.

*** An urban particulate standard (SRM 1648) is not available from NIST.

STATISTICAL QUALITY CONTROL

In traditional quality control systems much importance is placed on the establishment of average and range ($\bar{x}$,R) control charts to control quality. Averages are obtained from measurement of a sample from some assumed homogeneous rational subgroup of products. In this way, the average is used as a measure and means of control of the level of the quality characteristic and the range of the measurements is used as a measure and means of control of variability. Except in the laboratory, batches or rational subgroups seldom exist in pollution measurement, and even in these cases, replication is accomplished usually on a duplicate basis only, such as duplicate measures of the same sample, duplicate analyses by different analysts, or measurement from duplicate collocated sampling instruments. Further, except for repeated measurements of homogeneous control samples, the averages of the duplicates vary depending upon the concentration level. Therefore, the $\bar{x}$ chart is of little value in quality control for pollution measurements.

Further, in the cases of duplicate data, some identity can usually be associated with each of the pair of measurements, so that the range is not the best value of interest. Because of suspected bias between the two sources, *signed* differences should be used rather than the unsigned range. Further,

since the average levels may vary widely between pairs, and the error variation is usually proportional to levels, the value of concern is the *signed percentage* difference (or signed relative difference). This value is an appropriate parameter to plot on control charts as a means to control variability of the measurement process.

Control on the accuracy of the data must be maintained by frequent calibrations with materials traceable to primary standards. Some type of calibration is usually required on air pollution measurement systems daily or for each use, and occasionally calibration is necessary before, during, and after analysis of a given batch of samples. Control charts which may be maintained to assure that the calibration process remains in statistical control are those for the slope, intercept, and standard error of prediction for the calibration curves for multipoint calibrations, and zero and span drift checks to control the drift of continuous instruments.

DATA ANALYSIS AND VALIDATION

A number of special considerations exist in air pollution measurement systems with respect to data analysis and data validation. For most air pollution measurements, the error variations are proportional to the pollutant concentration level, thus complicating error analysis of the measurement system.

The aggregate frequency distributions of air pollution data are skewed, often lognormal or nearly, so, requiring logarithmic or other transformation when summarizing or analyzing data distributions. [1.3] Complications arise when taking logarithms of zero values! Also, special treatment of data below the minimum detectable levels may be required in the characterization or summarization of air pollution data.

Because of the many possible causes of variability in air pollution data, the data validation process as a separate activity is very important in air monitoring.[5] Since the quality of the data is not evident from the data itself, the routine checks of ancillary data for accuracy and precision must be made. Some further checks of the data with relation to other data or information may be made to validate the final product. Various types of checks which can and should be made include:

Manual Editing—checks for human error or equipment malfunction, such as:

1. impossibly high or low values
2. spikes, such as caused by electronic interference, and
3. repetitious values, such as caused by equipment malfunction.

Scientific Validation—checks involving scientific considerations, such as:

1. time continuity,
2. spatial continuity,
3. relationships among different pollutants, and
4. relationships with meteorological data.

PREVENTIVE MAINTENANCE

Preventive maintenance activities are not usually considered as part of quality assurance. However, for air pollution monitoring systems, the effectiveness of preventive maintenance is critical in determining the continuous operation of remote, unattended sampling equipment, particularly automatic sampling/analysis instruments. Unplanned malfunctioning of these instruments can prevent the obtaining of sample results of peak concentration periods, or prevent the accumulation of sufficient data to establish valid trend information.

Needless to say, all the above special and important features indeed make implementation of quality assurance of air pollution monitoring systems an interesting, but difficult and challenging effort.

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QUALITY ASSURANCE FOR POLLUTANT MONITORING

by

R. C. Rhodes

An on-going monitoring system will already have implemented a number of essential elements of a total quality assurance system. When reviewing an existing monitoring operation or when establishing a new monitoring effort, it is very desirable that a systematic review be made to consider or reconsider the quality assurance activities which should be required.

The various elements of a total quality assurance program, listed below, are discussed in the "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I, Principles," EPA 500/9-76-005, March 1976.

- Quality policy
- Quality objectives
- Quality organization and responsibility
- QA manual
- QA plans
- Training
- Procurement control
  - Ordering
  - Receiving
  - Feedback and corrective action
- Calibration
  - Standards
  - Procedures
- Internal QC checks
- Operations
  - Sampling
  - Sample handling
  - Analysis
- Data
  - Transmission
  - Computation
  - Recording
  - Validation
- Preventive Maintenance
- Reliability records and analysis
- Document control
- Configuration control
- Audits
  - On-site system
  - Performance
- Corrective action
- Statistical analysis
- Quality reporting
- Quality investigation
- Interlab testing
- Quality costs

The extent to which each of the above elements should be implemented by a given agency will depend upon (1) the objective of the monitoring, (2) the duration of the monitoring period, and (3) the type of sampling/analysis methods utilized. Each monitoring agency should review the quality assurance elements with respect to their particular needs, and should establish a prioritized long-range plan (schedule) for implementation. For ongoing monitoring efforts, the quality assurance program should be dynamic in nature, being continually improved and revised according to increased knowledge, changing conditions, and assigned priorities

The elements listed above fall into 4 general categories:

(1) *Management*—those activities which are of particular concern to, and must be initiated and sustained by management notwithstanding the fact that all activities of a monitoring system are management's responsibility.

(2) *Measurement*—those activities which are directly involved in the sampling and analysis of pollutant concentrations.

(3) *Systems*-those activities mainly involving the paperwork systems essential to operate and support the quality assurance system.

(4) *Statistics*—those computational and statistical analysis techniques and procedures which are necessary as part of the quality assurance system.

From the above, it is evident that a total quality assurance program is concerned with all activities which may affect the quality of the monitoring data, and is not limited in a very narrow sense to essential calibrations and a few routine duplicate analytical checks.

Management. It is obvious that management's responsibilities should include a stated written policy and objectives concerning quality. The need for monitoring data of high quality must be continually made evident by the management with a continual awareness of such need by all the people whose activities affect the quality of the data. One individual of the *organization* should be specifically designated and assigned the *responsibility* to oversee all quality assurance activities, even though the individual may have other assigned duties, and even though "Quality assurance is everybody's business." This individual should be designated as the "Quality Assurance Coordinator."

Management should establish *training* requirements for each individual whose activities affect quality. Detailed systematic written *plans* should be prepared summarizing the various quality control checks made for each pollutant measurement method or special project. A *manual* containing administrative-type procedures applicable to all measurement methods and projects and to general quality assurance activities should, in time, to be prepared to consolidate in one document all quality-related procedures. The manual should incorporate the above-mentioned plans by reference.

Management, obviously, is concerned with costs. And after operation of a monitoring system for, say, a year, a systematic review should be made of the *costs related to quality*, to assess the cost-effectiveness of these activities, and to make indicated changes in expenditures of effort to obtain the most high quality data for the least cost.

Additionally, management should establish some type of periodic (say quarterly) *report* summarizing quality assurance activities and providing some continual assessment or measure of data quality. This report should be prepared by the Quality Assurance Coordinator.

Measurement. Various EPA guideline documents have been prepared for each measurement method. These documents provide the identification of *calibration standards* and detailed *procedures* and *internal quality control checks* which should be made for the *sampling, sample handling,* and *analysis* for each of the methods.

It may be economically prohibitive to implement all of the recommended checks of these documents, at least initially. Specific minimum checks for ambient methods are included in EPA 600/4-77-027a, "Quality Assurance Handbook for Air Pollution Measurement Systems," Volume II, Ambient Air Specific Methods, May 1977. Specific minimum checks for source emission methods are included in EPA 600/4-77-027b, "Quality Assurance Handbook for Air Pollution Measurement Systems," Volume III, Source Emission Specific Methods, August 1977. Some judgment may need to be exercised as to which checks seem to be most critical and need to be implemented first. However, it is best to implement more checks at a lesser frequency than to concentrate heavily on just a few. The frequency of quality control checks should be flexible, being increased for those which by experience seem to give most problems, and being decreased for those which seem consistently to remain "in control." Similar reasoning applies with respect to the types and frequencies of independent *performance audits* described in the guideline documents.

One essential for obtaining high quality data is the procurement of measurement equipment and materials of adequate quality. Adequate specifications should be included in the procurement ordering documents, and the equipment and materials should be given adequate inspection when received. Generally, procured items should not be paid for until after they have been determined to meet the specifications. Obviously those methods and equipment designated or specified by the government as official for determining compliance to ambient air or source standards should be strictly and consistently complied with.

One part of the measurement method which may not receive adequate attention is that for flow measurement. For those methods which require flow measurement, the flow measurement is equally as important as the pollutant measurement.

A critical requirement of the measurement method (for pollutant and flow) is the use of secondary reference standards for calibration, traceable to a national or international primary standard.

Systems. Detailed, systematic and meticulous records need to be kept concerning all of the necessary measurements and computations integrally involved with the measurement process. Of equal importance is the recordkeeping concerning (1) the written procedures for calibration, operation, and computations, (2) *preventive maintenance* procedures and records, and (3) measurement equipment records. A *document control* system should be established to identify by number and date each written procedure or revisions thereof so that the exact procedure used at any specified time (past and present) can be determined. A *configuration control* system should be established to record the nature and dates of any changes in the hardware design, or major corrective maintenance of the sampling, sample handling, and analysis equipment. These records should be kept by manufacturer's serial number or an agency-assigned identification number. Such records should enable one to determine for any past and present time, the exact configuration of any specific piece of equipment. Also considered as part of a configuration control system is the site assignment history for each piece of identified sampling equipment.

Recordkeeping systems are essential to record changes to the procedures and equipment of the monitoring system. Experienced quality assurance and statistical personnel are suspicious of the possible effects of changes in the total measurement process. Their motto might well be "CAVE VICISSITUDINES" OR "CAVE VARIETAS."* Oftentimes, seemingly innocuous changes may

cause significant changes in the results. As a precaution against the introduction of such undesirable effects into the system, the basic principle of performing overlap checks or comparisons should be made to assure that such changes are appropriately valid.

Statistics. The use of statistical analyses is essential to an adequate quality assurance system. Some of the more basic statistical applications are presented in APTD 1132, "Quality Control Practices in Processing Air Pollution Samples." Other applications are included in the Appendices to EPA 600/9-76-005. If a given agency does not have a person with some training and experience in the basic statistical applications presented in these documents, either (1) an individual of the agency with mathematical capability should attend a course to receive such training, or (2) a statistician experienced in these applications should work with individuals of the agency on a temporary consulting basis to establish such techniques and provide such training. The applications of statistics to air monitoring extend from the simplest (control charts) to the very complex (modeling and computer simulation) and are limited only by the statistical and computation capability of available personnel and resources. The techniques of *data validation* and equipment *reliability analyses* are several specific applications of value in a local agency.

* CAVE VICISSITUDINES: Beware of changes
CAVE VARIETAS: Beware of differences

* This technique may be cost prohibitive for continuous instruments.

In addition to the above, several points deserve further emphasis with respect to the accuracy and precision of the measurement system. In addition to the use of good calibration standards and procedures, *interlaboratory* tests, such as the exchange of stable samples between peer laboratories, or the dissemination of blind samples from some recognized national or international laboratory is quite valuable in determining the accuracy of participating agencies. Such testing may reveal weaknesses in the system which would require special *quality investigations*. The use of statistics in planning such studies and in analyzing the data therefrom, is emphasized.

An excellent way to check the internal precision of an agency's system, is to establish at one (or a few) selected cities a dual or collocated sampling instrument for each measurement method.* This type of duplicate check is one form of the independent *performance audits* described in the EPA QA Guidelines document for manual integrated methods. The duplicate sampling instruments should be maintained as independently as possible from the regular instrument. For example, where possible, independent calibrations and flow measurements should be made for the collocated duplicate instrument. Similarly, for integrated manual methods the pollutant analyses should be performed as independently as possible in the laboratory. For example, the samples from the collocated instrument should be analyzed on a different batch (using a different calibration) from that in which the regular sample is analyzed. In the above-described manner, the best possible estimate for within-agency precision for the total measurement process can be made. Excessive differences in results between the paired instruments will indicate weaknesses in the system which should be isolated by investigation and corrected by appropriate *corrective* action.

As a part of the recordkeeping system, each agency should compile (or maintain) a "Significant Event History." Documentation of the location, nature, dates, and times of special events affecting pollutant concentrations should be kept in a systematic chronological file. Such events which might explain unusual results would be those such as dust storms, large fires, construction work, etc.

Quality Assurance System Review. On occasion, the Quality Assurance System of a given monitoring agency may be subject to an *on-site system audit* or review by an external organization, for the purpose of evaluating the capability of the agency to produce data of acceptable quality. Such an independent review is made of the agency's facilities, equipment, personnel, organization, procedures, etc. by persons knowledgeable in both quality assurance technology and the measurement technologies involved. The audit should include a review of the agency's actual operations, procedures, and recordkeeping for all of the elements of quality assurance system discussed herein. The audit team's evaluation should include specific identification of areas of weakness and specific recommendations for improvement.

* This technique may be cost prohibitive for continuous instruments.

Lesson 3

Establishing A Quality Assurance Program

Establishing a Quality Assurance Program

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Questions Answered in This Lesson

- What quality assurance elements are involved in establishing a quality assurance program and what factors should be considered in their implementation?
- What quality assurance elements are involved in recording changes in an air pollution monitoring system?

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Questions Answered in This Lesson (cont.)

- What is the purpose of document control?
- What is the purpose of a configuration control system?
- What factors should be considered in designing a preventive maintenance system?

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Managerial Quality Assurance Elements

- Establishing a quality assurance program
- Recording changes in the air quality monitoring system

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Establishing a Quality Assurance Program

- Policy and objectives
- Organization
- Quality assurance plans
- Training
- Audit procedures
- Corrective action
- Reports to management

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Quality Assurance Policy and Objectives

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

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Measurement Quality Objectives

Data meeting user requirements

- Completeness
- Precision
- Accuracy
- Representativeness
- Comparability

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Measurement Quality Objectives (cont.)

- Data are complete if a prescribed percentage of total measurements is present
- Precision—extent of agreement with the mean
- Accuracy—nearness to true value

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Measurement Quality Objectives (cont.)

- Data must be representative of the condition being measured
- Data from several agencies should be in the same units and should be corrected to the same conditions (standard temperature and pressure) to allow comparison among groups

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Data Quality Objectives (DQOs)

- Take into account measurement quality objectives
- Expressed as probability of making a wrong decision

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Organization

Quality assurance is normally a separate function in the organization

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Basic Functions of QA Organization

QA Policy Formulation

- Agency policy
- Contracts
- Procurement
- Staff training and development

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QA Guidance and Assistance

- Laboratory operations
- Monitoring network operations
- Data reduction
- Special field studies
- Instrument maintenance and calibration

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QA Guidance and Assistance (cont.)

- Preparation of legal actions
- Source emission testing
- Development of control regulations
- Preparation of technical reports

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General QA Plans, Requirements, and Information

- QA policy statement
- Measurement requirements
- Data quality objectives
- QA organization chart
- QA job descriptions

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Training

- Essential for all personnel in any function affecting data quality
  - Sample collection
  - Analysis
  - Data reduction
  - Quality assurance

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Training (cont.)

- On-the-job training (OJT)
- Short-term course training (normally 2 weeks or less)
- Long-term course training

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Performance Audit Procedures

- Independent checks
- Evaluate data quality of measurement system
- Quantitative appraisal of quality

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System Audit Procedures

- On-site inspection and review of quality assurance system
- Qualitative appraisal of quality assurance system

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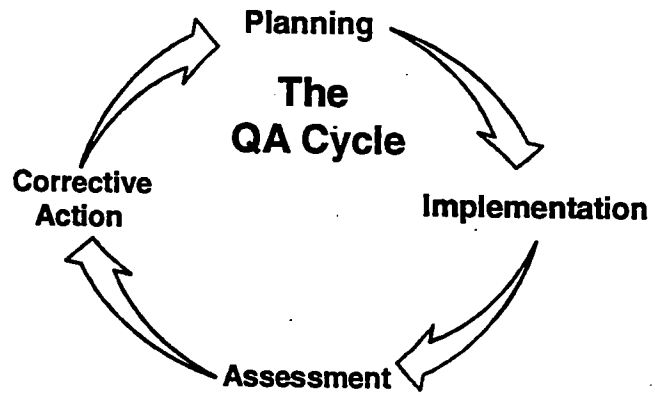

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Quality Reports to Management

- Quality data usually periodically reported:
  - Percentage duplication or replication of determinations
  - Instrument or equipment downtime
  - Percentage voided samples vs total samples
  - Quality cost in terms of prevention, appraisal, and correction costs

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Quality Reports to Management (cont.)

- Quality data usually reported:
  - System audit (on-site inspection) results
  - Performance audit results
  - Interlaboratory and intralaboratory test results (precision and accuracy)
  - Status of solutions to major quality assurance problems

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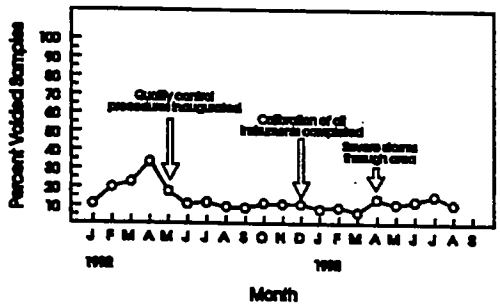
Graphic Report to Management



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Recording Changes in the Monitoring System

- Document control—for written procedures
- Configuration control—for design and location of the monitoring system
- Preventive maintenance—for routine service after operation has begun

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Document Control System Purpose

To provide the latest written procedures to all concerned personnel

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Document Control System

- Should include an easy way to make changes:
  - Removable pages
  - Easily identifiable pages
  - Indexed by:
    - Section and revision numbers
    - Date
    - Page number and total pages

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Document Control System (cont.)

Should include a distribution record system

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Configuration Control System

Purpose is to record changes in equipment and the physical arrangement of equipment in the monitoring system

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Two Types of Configuration Control Systems

- Historical record-keeping system
- First article configuration inspection (FACI) system

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Preventive Maintenance

- An orderly program of positive actions for preventing failure of a monitoring system
  - Cleaning
  - Lubricating
  - Reconditioning
  - Adjusting
  - Testing

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Purpose of Preventive Maintenance

Increased measurement system reliability

⇩

- Increased data completeness

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Development of a Preventive Maintenance Program

- Review equipment—identify pieces most likely to fail
- Define spare parts inventory
- Define frequency for servicing
- Prepare a checklist

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Daily Checklist for NO_2 Analyzer

City _____		At last calibration				NO	NO_2	
Site location _____		Scale range						
		Zero knob setting						
Site number _____		Span knob setting						
Serial number _____		Date last calibrated						
Date		M	T	W	Th	Fri	Sat	Sun
Unadjusted NO zero reading								
Unadjusted NO_2 zero reading								
NO zero setting (new)								
NO span setting								
NO_2 zero setting (new)								
NO_2 span setting								
✓	Valve in NO-NO_2/H_2O position							
✓	NO-NO_2 range in 0-5 position							
✓	Inspect inlet line, probe, and filter holder							

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Lesson 4

Basic Concepts Of Statistical Control Charts

Basic Concepts of Statistical Control Charts

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Questions Answered in This Lesson

- What does a control chart based on a period of acceptable performance look like?
- What is the difference between assignable (nonrandom) and unassignable (random) causes of variation?

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Questions Answered in This Lesson (cont.)

- What steps are involved in developing a control-chart system?
- What are the characteristics of a normal (Gaussian) frequency distribution?
- What considerations are important in using control charts?

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Control Chart

- Shows how a process should behave
- Shows how a process is behaving
- Signals when action should be taken to make the process behave as it should

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"Constant Cause" System

A system that measures something whose variability remains constant

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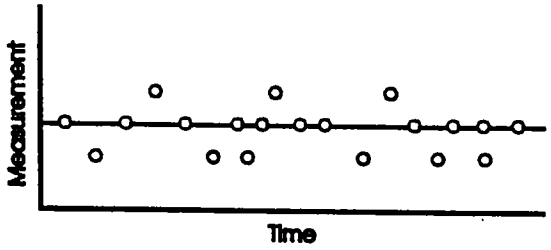
Measurements will vary over time because of fluctuating parameters



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Types of Variations

Random
- Unassignable
- Statistical control

Nonrandom
- Assignable
- Out-of-control

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Objectives of a Control Chart

- Detect assignable causes
- Trigger investigation leading to corrective action

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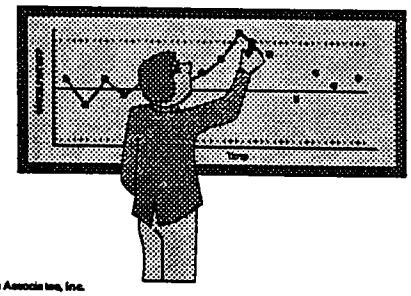
Development and Use of a Control Chart



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How to Develop and Use a Control Chart

- Determine what data to chart
- Accumulate data
- Prepare histogram
- Determine form of frequency distribution
- After eliminating outliers, calculate the mean and the standard deviation
- Establish limits

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How to Develop and Use a Control Chart (cont.)

- Construct chart
- Plot points
- Highlight out-of-control conditions
- Take corrective action
- Revise control limits
- Maintain historical file

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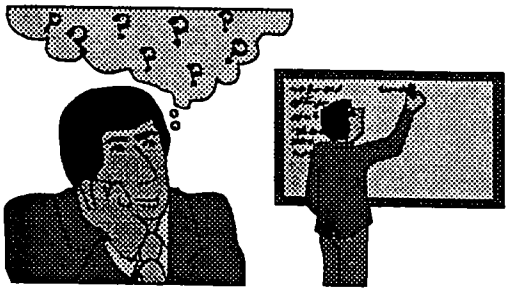
Determine What Data to Chart



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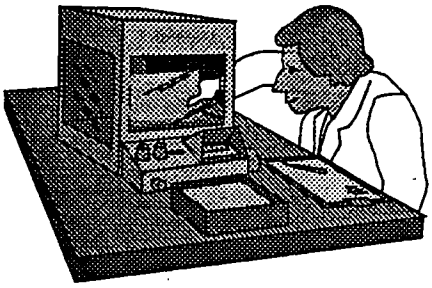
Accumulate Data



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Prepare Histogram

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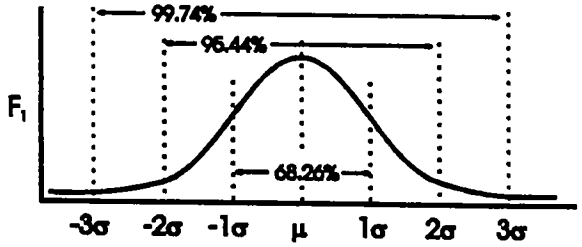
Determine Form of Frequency Distribution



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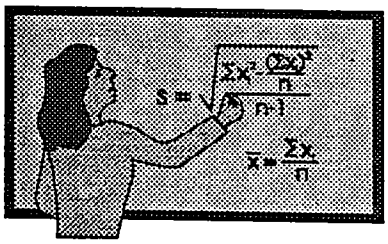
After Eliminating Outliers, Calculate the Mean and the Standard Deviation



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Establish Limits

	Control	Warning
American	• ± 3 s • 99.74%	• ± 2 s • 95.44%
British	• ±3.09 s • 99.8%	• ± 1.96 s • 95.0%

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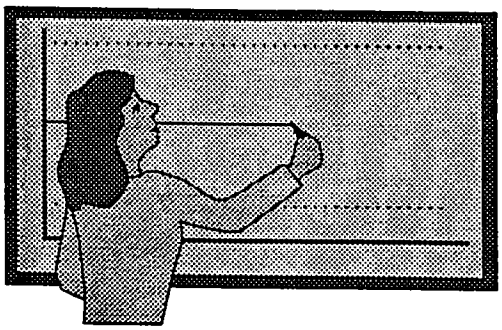
Construct Chart



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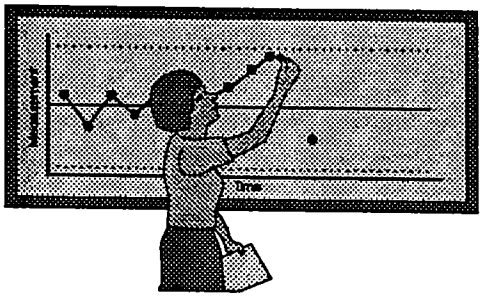
Plot Points



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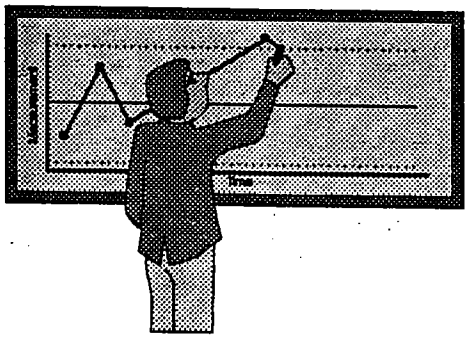
Highlight Out-of-Control Conditions



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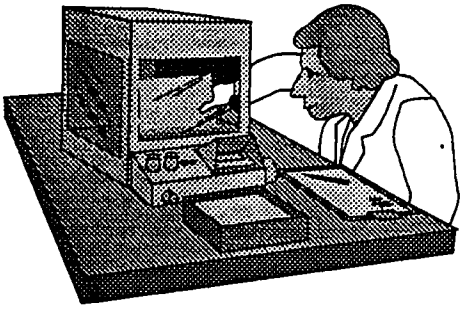
Take Corrective Action



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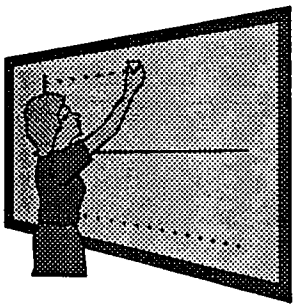
Assess/Revise Control Limits



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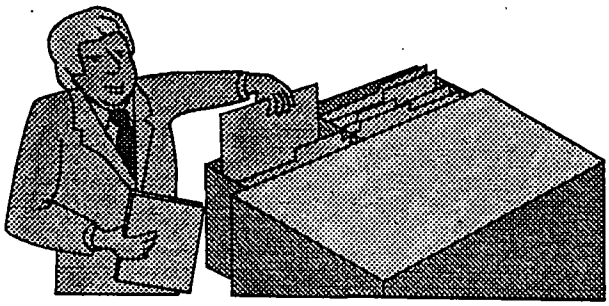
Maintain Historical File



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Lesson 5

$\bar{x}$-R Statistical Control Charts

$\bar{x}$-R Statistical Control Charts

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Questions Answered in This Lesson

- What is the Shewhart concept of local control (i.e., use of rational subgroups) as a basis for developing control charts?
- How are situations involving rational subgroups different from situations in which no rational subgroup exists?
- What is the difference between control charts based on only a period of acceptable performance and control charts based on rational subgroups?

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Questions Answered in This Lesson (cont.)

- How do you compute control limits for $\bar{x}$-R control charts?
- What are the three rules for detecting out-of-control data points?
- What five types of out-of-control patterns can be visually detected using a control chart?
- What three assumptions relate to the detection and correction of assignable causes of measurement process variability?

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Local Statistical Control: Shewhart

Control limits based on:

- Short-term rational subgroups
- Smaller or homogeneous variation

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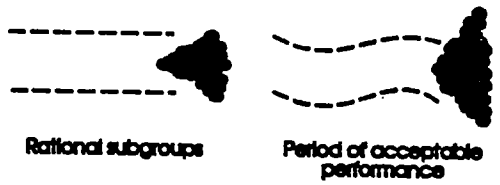
Control charts can be based on:



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Constructing a $\bar{x}$-R Control Chart

- Identify rational subgroups
- Calculate each subgroup's arithmetic mean ($\bar{x}$) and range (R)
- Calculate overall arithmetic mean ($\bar{\bar{x}}$) and average range ($\bar{R}$)

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Lesson 5

$\overline{x}$-R Statistical Control Charts

$\bar{x}$-R Statistical Control Charts

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Questions Answered in This Lesson

- What is the Shewhart concept of local control (i.e., use of rational subgroups) as a basis for developing control charts?
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Questions Answered in This Lesson (cont.)

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Local Statistical Control: Shewhart

Control limits based on:

- Short-term rational subgroups
- Smaller or homogeneous variation

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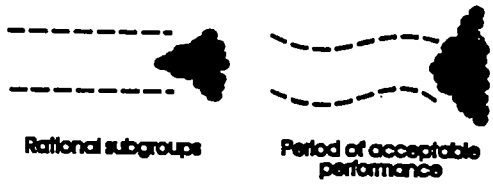
Control charts can be based on:



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Constructing a $\bar{x}$-R Control Chart

- Identify rational subgroups
- Calculate each subgroup's arithmetic mean ($\bar{x}$) and range (R)
- Calculate overall arithmetic mean ($\bar{\bar{x}}$) and average range ($\bar{R}$)

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Factors for Computing Control Chart Lines Using Range

Number of Observations in Subgroup, n	Factor A_2	Factor D_3	Factor D_4	Factor D_5	Factor D_6
2	1.88	0	3.27	0	2.51

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$\bar{x}$ Chart Control Limits (Upper Control Limit)

$$UCL_{\bar{x}} = \bar{\bar{x}} + (A_2)(\bar{R})$$

Where:

$\bar{\bar{x}} = 29.92$

$A_2 = 1.88$ (for subgroups containing two data values)

$\bar{R} = 4$

$UCL_{\bar{x}} = 29.92 + (1.88)(4)$

$UCL_{\bar{x}} = 37.44$

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$\bar{x}$ Chart Control Limits (Lower Control Limit)

$$LCL_{\bar{x}} = \bar{\bar{x}} - (A_2)(\bar{R})$$

$$LCL_{\bar{x}} = 29.92 - (1.88)(4)$$

$$LCL_{\bar{x}} = 22.40$$

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$\bar{x}$ Chart Control Limits (Upper Warning Limit)

$$UWL_{\bar{x}} = \bar{\bar{x}} + (2/3)(A_2)(\bar{R})$$
$$UWL_{\bar{x}} = 29.92 + (2/3)(1.88)(4)$$
$$UWL_{\bar{x}} = 34.93$$

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$\bar{x}$ Chart Control Limits (Lower Warning Limit)

$$LWL_{\bar{x}} = \bar{\bar{x}} - (2/3)(A_2)(\bar{R})$$
$$LWL_{\bar{x}} = 29.92 - (2/3)(1.88)(4)$$
$$LWL_{\bar{x}} = 24.91$$

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R Chart Control Limits (Upper Control Limit)

$$UCL_R = (D_4)(\bar{R})$$

Where:

$D_4 = 3.27$ (for subgroups containing two data values)

$\bar{R} = 4$

$UCL_R = (3.27)(4)$

$UCL_R = 13.08$

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R Chart Control Limits (Lower Control Limit)

$LCL_R = (D_3)(\bar{R})$

Where:

$D_3 = 0$ (for subgroups containing two data values)

$\bar{R} = 4$

$LCL_R = (0)(4)$

$LCL_R = 0$

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R Chart Control Limits (Upper Warning Limit)

$UWL_R = (D_5)(\bar{R})$

Where:

$D_5 = 2.51$ (for subgroups containing two data values)

$\bar{R} = 4$

$UWL_R = (2.51)(4)$

$UWL_R = 10.04$

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R Chart Control Limits (Lower Warning Limit)

$LWL_R = (D_4)(\bar{R})$

Where:

$D_4 = 0$ (for subgroups containing two data values)

$\bar{R} = 4$

$LWL_R = (0)(4)$

$LWL_R = 0$

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Construct $\bar{x}$-R Control Chart

- Draw control and warning limits
- Plot individual x's and R's
- Use prepared $\bar{x}$-R control chart for evaluating future $\bar{x}$'s and R's

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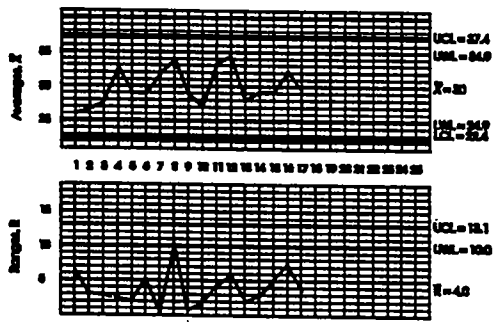

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Out-of-Control Criteria

- Points beyond limits
- Runs
- Patterns

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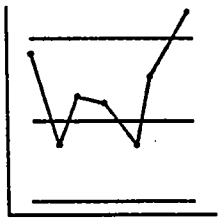
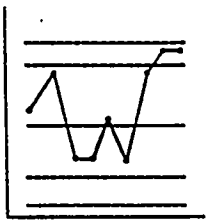
Points Beyond Limits



One point outside control limits



Two points outside warning limits

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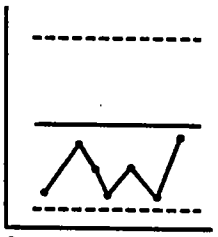
Runs

Seven points—all up or all down



Seven points—all above or all below central line

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Nonrandom Data Patterns

- Recurring cycles
- Change in level
- Lack of variability
- Trends
- Most points near outside limits

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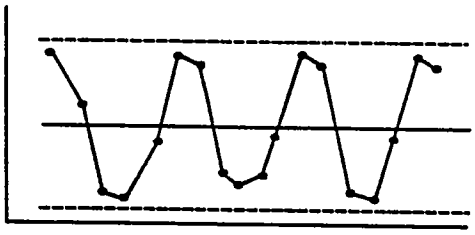
Recurring Cycles



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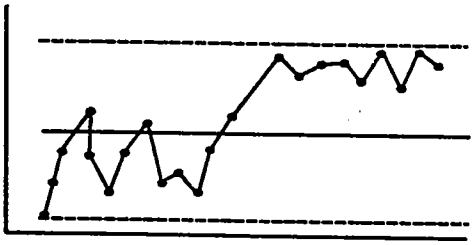
Change in Level



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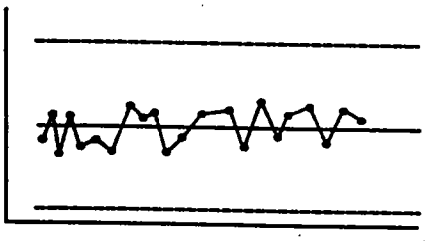
Lack of Variability



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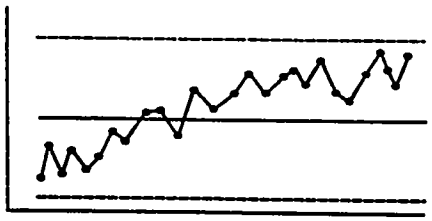
Trends



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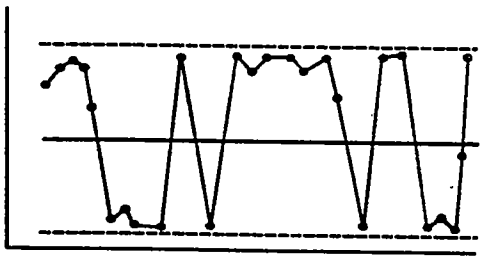
Most Points Near Outside Limits



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Assumptions Concerning Assignable Causes

- Possible to identify and correct
- Technically feasible to correct
- Economically practical to correct

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I. Homework Assignment

A standard material is checked at periodic intervals during routine analyses to ensure that the analytical measurement process remains in control. Following are the results, in the chronological order in which they were obtained:

1. 19.0	14. 18.5
2. 18.3	15. 19.1
3. 18.0	16. 21.8
4. 17.2	17. 20.1
5. 17.4	18. 20.6
6. 18.3	19. 18.4
7. 19.6	20. 21.0
8. 20.7	21. 25.1
9. 18.2	22. 21.1
10. 18.8	23. 20.9
11. 20.4	24. 20.8
12. 20.1	25. 23.3
13. 19.6	26. 20.2

A. Prepare and plot a control chart with appropriate limits, assuming a single analysis is performed each day.

B. Prepare and plot $\bar{x}$ and R control charts with appropriate limits, assuming two analyses are performed each day; i.e., results 1 and 2 were obtained on day 1, results 3 and 4 were obtained on day 2, etc. (Hint: each day is a subgroup.)

C. Do the charts indicate any out-of-control conditions? If so, describe them.

Project Name _____ No Subgroups _____ Measurement Performed _____ Measurement Units _____

Date																										
Measurement	Code	1																								
		2																								
		3																								
	Result	1																								
		2																								
		3																								
Sum																										
Average, $\bar{x}$																										
Range, R																										

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25
Individual Values, x																									
Ranges, R																									
Comments (Correct. Action, etc.)																									

Project Name _____ Using Subgroups _____ Measurement Performed _____ Measurement Units _____

Date																										
Measurement	Code	1																								
		2																								
		3																								
	Result	1																								
		2																								
		3																								
Sum																										
Average, $\bar{x}$																										
Range, R																										

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25
Averages, $\bar{x}$																									
Ranges, R																									
Comments (Correct. Action, etc.)																									

Lesson 6

The Measurement Process, With Emphasis On Calibration

The Measurement Process, with Emphasis on Calibration

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Questions Answered in This Lesson

- What are the three components of an air pollutant measurement?
- What are the quality control considerations for these components?
- What is calibration?

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Questions Answered in This Lesson (cont.)

- What are the six general elements of a calibration program?
- What is traceability?
- What services are provided by EPA's Standards Laboratory?

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Air Pollutant Measurement Process

- Separate pollutant from air
- Determine pollutant quantity and air volume
- Calculate pollution concentration by dividing pollutant quantity by air volume

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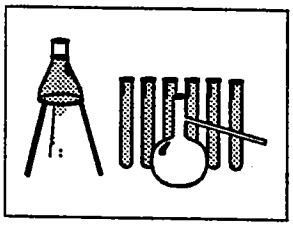
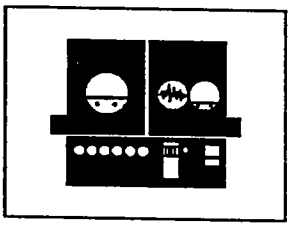
Separation of Pollutant



Manual



Automated

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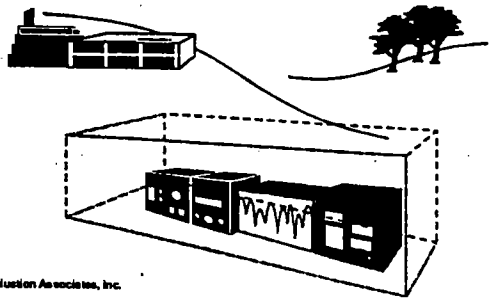
Determination of Amount of Pollutant and Volume of Air Sampled



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Calibration

The process of establishing the relationship between the output of a measurement process and a known input

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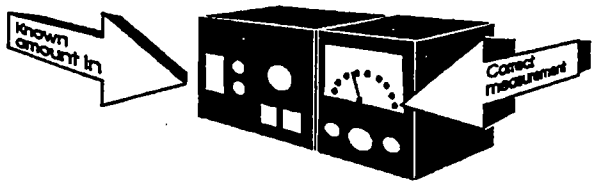

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Elements of a Calibration Program

- Statements of allowable time between calibrations
- Statements of minimum quality of calibration standards
- Provisions for standards traceability
- Provisions for written procedures
- Statements of proper environmental conditions
- Provisions for proper record-keeping

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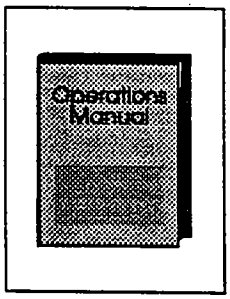
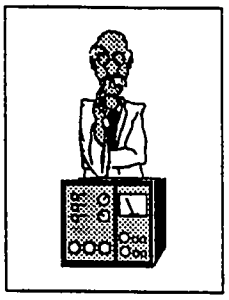
Vendor Recommendations





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Contact Users for Opinions

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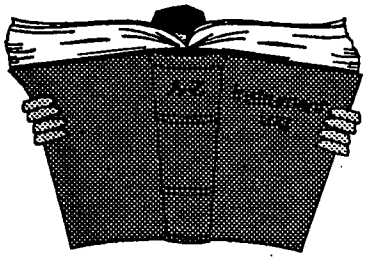
In-House Records/Former Experience



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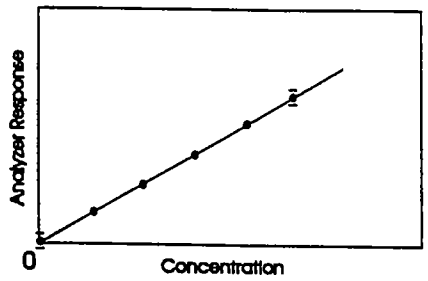
Zero and Span Drift Limits



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Statements of Minimum Quality of Calibration Standards

Instruments used as standards should have 4 to 10 times the accuracy of the instruments they are being used to calibrate

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Provisions for Standards Traceability

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Tracing of Standards

- NIST-SRMs
- CRMs

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EPA's Standards Laboratory

Certification of client-owned calibration and auditing materials

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Certification Services Available (Government Personnel and Their Contractors Only)

- Cylinder gases
- Permeation tube rates
- Flow measuring devices
- Calibration/Audit devices
- Special analyses on request

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Write To:

Atmospheric Research and Exposure Assessment Laboratory

Quality Assurance and Technical Support Division

US EPA, MD-78A, Research Triangle Park, NC 27711

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Provisions for Written Procedures

Written procedures are needed to:

- Assure that calibrations are always performed in the same manner
- Make the calibration process less operator-dependent
- Evaluate the calibration process

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Statements of Proper Environmental Conditions

Common environmental conditions needing control to assure accurate calibrations:

- Dust and vapor
- Vibration—especially for weighing procedures
- Temperature
- Humidity
- Electrical line voltage
- Lighting—especially important in titrimetric procedures

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Provisions for Proper Record-Keeping

- Assure adequate documentation of calibrations
- Use bound calibration logbooks
- Trace calibrations
- Report items for each instrument calibration

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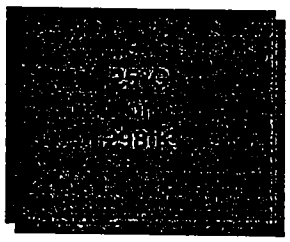
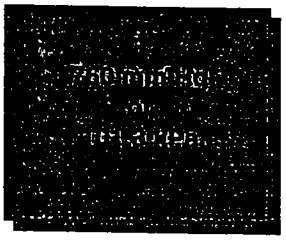
6-30-83

Calculation of Ambient Pollutant Concentration



Standard Temperature



Standard Pressure

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Lesson 6A

Group Problem

Lesson 6A

Group Problem

Lesson Goal

To integrate student quality assurance (QA) knowledge, increase student involvement in the course, and enhance quality assurance information interchange among course participants.

Lesson Objectives

At the conclusion of this lesson, each student will be able to understand the relationship among various course topics.

Group Problem Planning Sheet

Group _____

1. Write what you consider to be the QA policy for the group problem.

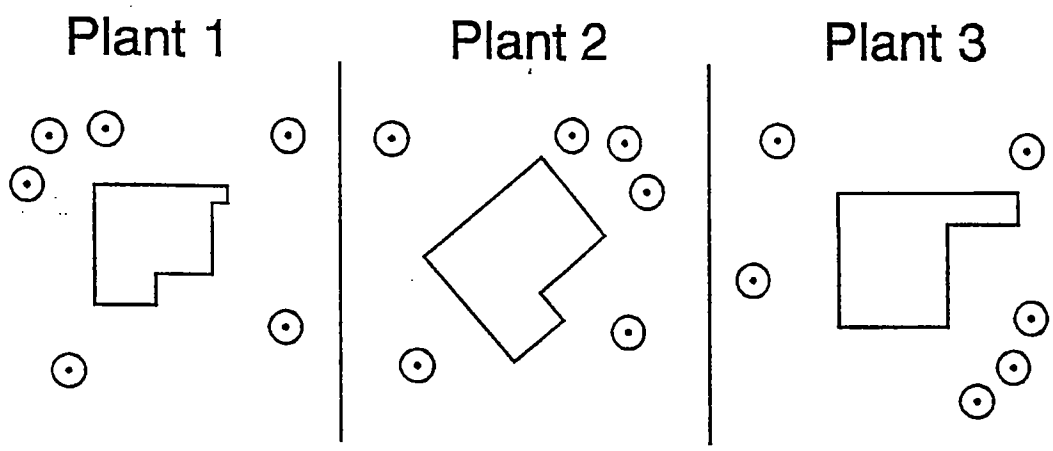
2. List the measurement quality objectives for the group problem.

Group Problem

A highly toxic, gaseous pollutant, cyclolehmdone (CL), has recently been reported in the ambient air. Each group is to develop a monitoring and quality assurance plan to determine the ambient level of CL.

The following data are provided:

- This is a statewide problem. All efforts are coordinated through the state central office.
- Three local offices, located throughout the state, will be engaged in the field work. Each local office has a laboratory where CL analyses will be performed. Assume each local office and the state office have adequate staffing and funding.
- Just by coincidence, three plants suspected of CL emissions are located in the state—one plant is located in each of the jurisdictional areas of the local offices. Each plant uses CL in the manufacture of its products.
- Both a manual method and a continuous monitoring (instrumental) method exist. Each local office has a gas chromatograph, as does the state office, for analyzing manual samples. Each office also has one continuous monitoring instrument available for use in the study. Gas chromatographs must remain in their labs. Continuous monitoring instruments of the local offices must remain in the field. The purchase of additional continuous monitors is not possible.
- The length of the sampling program is two months.
- For manual sampling, 24-hour integrated sampling will be done every day.
- Sampling sites have been properly selected around each plant, using historical meteorological data. The siting team has decided that six stations are needed:



- Manual sampling equipment and supplies must be procured.
- There is a NIST-SRM (permeation tube) at the state office; cylinders of "known" concentrations of CL are available from FBN, Inc. Purchase of additional permeation tubes is not possible.

Manual Method—Attachment I
Continuous Method—Attachment II

ATTACHMENT I

Manual Method For Cyclolehmdone In Air

Analyte	Cyclolehmdone	Method no: Range:	470-1 0.2 - 1500 nanograms per injection
Matrix:	Air		
Procedure:	Adsorption on charcoal, desorption with carbon disulfide, analysis with gas chromatograph		
Date issued:	9/3/92	Precision:	Unknown
Date revised:	10/15/92	Classification:	D (operational)

1. Principle of the method

   1.1 A known volume of air is drawn through a charcoal tube to trap the cyclolehmdone present.

   1.2 The charcoal in the tube is transferred to a small vial containing carbon disulfide, where the cyclolehmdone is desorbed.

   1.3 An aliquot of the desorbed sample is injected into a gas chromatograph.

   1.4 The area of the resulting peak is determined and compared with areas obtained from the injection of standards.

2. Range and sensitivity

   2.1 The minimum detectable amount of cyclolehmdone was found to be 0.2 nanograms per injection at a 1 x 1 attenuation on a gas chromatograph.

   2.2 At the recommended sampling flow rate of 50 mL/min, the total volume to be sampled should not exceed 75.0 L. This value is the volume of air containing 200 ppm of cyclolehmdone that can be sampled before a significant amount of cyclolehmdone is found on the back-up section. [The charcoal tube consists of two sections of activated charcoal separated by a section of urethane foam (see Section 6.2).] If a particular atmosphere is suspected of containing a high concentration of contaminants and/or a high humidity, the sampling volume should be reduced by 50 percent.

3. Interferences

3.1 When the amount of water in the air is so great that condensation occurs in the tube, organic vapors will not be trapped. Preliminary experiments indicate that high humidity severely decreases the capacity of the charcoal to adsorb organic vapors.

3.2 When two or more substances are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample, because these compounds might interfere with the analysis for cyclolehmdone.

3.3 Any compound that has the same retention time as cyclolehmdone at the operation conditions described in this method is an interference. Hence, retention time data on a single column, or even on a number of columns, cannot be considered as proof of chemical identity. For this reason, it is important that a sample of the bulk material be submitted at the same time so that identity (identities) can be established by other means.

3.4 If the possibility of interference exists, separation conditions (e.g., column packing and temperature) must be changed to circumvent the problem.

4. Precision and accuracy

The precision and accuracy of the total sampling and analytical method have not been determined.

5. Advantages and disadvantages of the method

5.1 The sampling device is small, portable, and does not involve the use of liquids. Interferences are minimal, and most of those that do occur can be eliminated by altering chromatographic conditions. The tubes are analyzed by a rapid, instrumental method. The method can also be used for the simultaneous analysis of two or more components suspected to be present in the same sample by simply changing gas chromatographic conditions from isothermal to a temperature-programmed mode of operation.

5.2 One disadvantage of the method is that the amount of sample that can be taken is limited by the saturation capacity of the tube. When the sample value obtained for the back-up section of the charcoal trap exceeds 20 percent of that found on the front section, there's a possibility of sample loss. During sample storage, volatile compounds such as cyclolehmdone will migrate throughout the tube until equilibrium is reached. At this time, 33 percent of these compounds will be found in the back-up section. This might lead to some confusion as to whether sample loss has occurred. This migration effect can be considerably decreased by shipping and storing the tubes at -20°C.

5.3 The precision of the overall method is limited by the reproducibility of the pressure drop across the tubes. This drop will affect the flow rate and cause the measurement of the volume of air sampled to be imprecise, because the pump is usually calibrated for one tube only.

6. Apparatus

6.1 An approved and calibrated personal sampling pump for personal and area samples whose sampling flow rate can be determined accurately at 50 mL/min.

6.2 Charcoal tube—glass tube with both ends flame sealed, 7 cm long with a 6-mm O.D. and a 4-mm I.D., containing two sections of 20/40 mesh activated coconut charcoal separated by a 2-mm portion of urethane foam. The activated charcoal is prepared from coconut shells and is fired at 600°C before packing to remove material possibly adsorbed on the charcoal. The primary adsorbing section contains 100 mg of charcoal and the back-up section contains 50 mg. A 3-mm portion of urethane foam is placed between the outlet end of the tube and the back-up section. A plug of silicated glass wool is placed in front of the adsorbing section. The pressure drop across the tube must be less than one inch of mercury at a flow rate of 1 L/min.

6.3 Gas chromatograph equipped with a flame ionization detector.

6.4 Stainless steel column (20 ft x 1/8 in) packed with 10 percent SE-30 on 80/100 mesh Chromosorb W (acid-washed, silanized with dimethyldichlorosilane). Other columns capable of performing the required separations can be used.

6.5 A mechanical or electronic integrator or a recorder and some method for determining peak area.

6.6 A batch of 2-mL vials that can be sealed with caps containing Teflon®-lined silicone rubber septa.

6.7 Microliter syringes: 10 µL and other sizes convenient for making standards.

6.8 Gas-tight syringes: 1 mL, with an open/close valve.

6.9 Pipets: 0.5-mL delivery pipets or a 1.0-mL pipet graduated in 0.1-mL increments.

6.10 Volumetric flasks—10 mL or other sizes convenient for making standard solutions. It is preferable to have plastic stoppers for the volumetric flasks.

7. Reagents

7.1 Spectroquality carbon disulfide.

7.2 Cyclolehmdone, lecture bottle, 99.9-percent minimum purity.

7.3 Toluene, chromatographic quality.

7.4 Bureau of Mines Grade-A helium.

7.5 Prepurified hydrogen.

7.6 Filtered, compressed air.

8. Procedure

8.1 All glassware used for the laboratory analysis should be detergent-washed and thoroughly rinsed with distilled water.

8.2 Each personal pump must be calibrated with a representative charcoal tube in line. This will minimize errors associated with uncertainties in the sample volume collected.

8.3 Collection and shipping of samples

8.3.1 Immediately before sampling, the ends of the tube are broken to provide an opening at least one-half the internal diameter of the tube (2 mm).

8.3.2 The smaller section of charcoal is used as a backup and is positioned nearest the sampling pump.

8.3.3 The charcoal tube is placed in a vertical position during sampling to prevent "channeling" of the charcoal.

8.3.4 Air being sampled is not to be passed through any tubing before entering the charcoal tube.

8.3.5 Bulk air samples (i.e., 10-20 L samples of the ambient air) are taken along with personal samples.

8.3.6 The flow rate, time, and/or volume of sampled air must be measured as accurately as possible. The sample is taken at a flow rate of 50 mL/min. The maximum volume to be sampled should not exceed 75.0 L (see Section 2.2).

8.3.7 The temperature and pressure of the ambient air being sampled is measured and recorded.

8.3.8 The charcoal tubes are capped with the supplied plastic caps immediately after sampling. Under no circumstances are rubber caps to be used.

8.3.9 One tube is handled in the same manner as the sample tube (break, seal, and transport), except that no air is sampled through this tube. This tube is labeled as a blank.

8.3.10 Capped tubes are packed tightly before they are shipped, to minimize tube breakage during transport to the laboratory. If the samples will spend a day or more in transit, cooling (e.g., with dry ice) is necessary to minimize migration of cyclolehmdone to the back-up section.

8.3.11 Samples received at the laboratory are logged in and immediately stored in a freezer (around -20°C) until time for analysis. Samples can be stored in this manner for two months with no appreciable loss of cyclolehmdone. Even around -20°C, cyclolehmdone will equilibrate between the two sections of charcoal (i.e., cyclolehmdone will migrate to the back-up section). This phenomenon is observable after two weeks and might be confused with sample loss after one to two months.

8.4 Analysis of samples

8.4.1 In preparation for analysis, each charcoal tube is scored with a file in front of the first section of charcoal and broken open. The glass wool is removed and discarded. The charcoal in the first (larger) section is transferred to a small vial containing 1 mL of carbon disulfide. (Note: the addition to the carbon disulfide is important.) The vial is topped with a septum cap (see Section 6.6). The separating section of foam is removed and discarded; the second section is transferred to another small vial containing 1 mL of carbon disulfide. These two sections are analyzed separately. Tests indicate that desorption is complete in 30 minutes if the sample is agitated occasionally during this period. In any case, samples should be analyzed within 60 minutes after addition to carbon disulfide.

8.4.2 The typical operating conditions for the gas chromatograph are:

(1) 40 mL/min (80 psig) helium carrier gas flow rate
(2) 65 mL/min (20 psig) hydrogen gas flow rate to detector
(3) 500 mL/min (50 psig) air flow rate to detector
(4) 230°C injector temperature
(5) 230°C manifold temperature (detector)
(6) 60°C isothermal column temperature (oven)

8.4.3 The first step in the analysis is the injection of the sample into the gas chromatograph. To eliminate difficulties arising from blowback or distillation within the syringe needle, the solvent flush injection technique is used. The 10-µL syringe is first flushed with solvent several times to wet the barrel and plunger. Two microliters of solvent are drawn into the syringe to increase the accuracy and reproducibility of the injected sample volume. The needle is removed from the solvent and the plunger is pulled back about 0.4 µL to separate the solvent flush from the sample with a pocket of air to be used as a marker. The needle is then immersed in the sample and a 5-µL aliquot is withdrawn to the 7.4 µL mark (2 µL of solvent + 0.4 µL of air + 5 µL of sample = 7.4 µL). After the needle is

removed from the sample and before injection, the plunger is pulled back a short distance to minimize evaporation of the sample from the tip of the needle. Duplicate injections of each sample and standard are made. No more than a 3-percent difference in peak area is to be expected.

8.4.4 The area of the sample peak is measured by an electronic integrator or some other suitable form of area measurement. Preliminary results are obtained from a standard curve, prepared as discussed in Section 9.

8.5 Determination of desorption efficiency

8.5.1 The desorption efficiency of a particular compound can vary from one laboratory to another and also from one batch of charcoal to another. Thus, it is necessary to determine at least once the percentage of cyclolehmdone removed in the desorption process. Desorption efficiency should be determined on the same batch of charcoal tubes used in sampling. Results indicate that desorption efficiency varies with loading (total cyclolehmdone on the tube), particularly at lower values (e.g., 2.5 µg).

8.5.2 Charcoal tubes from the same batch as that used to obtain samples are used in this determination. A measured volume of cyclolehmdone gas is injected into a bag containing a measured volume of air. The bag is made of Tedlar® (or a material that will retain the cyclolehmdone and not absorb it) and should have a gas sampling valve and a septum injection port. The concentration of cyclolehmdone in the bag can be calculated from room temperature and pressure. A measured volume is then sampled through a charcoal tube with a calibrated sampling pump. At least five tubes are prepared in this manner. These tubes are desorbed and analyzed in the same manner as the samples (see Section 8.4). Samples taken with a gas-tight syringe from the bag are also injected into the gas chromatograph. The concentration of cyclolehmdone in the bag is compared to the cyclolehmdone concentration obtained from the tubes.

The desorption efficiency equals the amount of cyclolehmdone desorbed from the charcoal divided by the quantity of cyclolehmdone contained in the volume of synthetic atmosphere sampled, or:

$$\text{Desorption efficiency} = \frac{\text{quantity of cyclolehmdone desorbed from the charcoal}}{\left(\begin{array}{c}\text{concentration of cyclolehmdone}\\ \text{in the atmosphere}\end{array}\right)\left(\begin{array}{c}\text{volume of atmosphere}\\ \text{sampled}\end{array}\right)}$$

9. Calibration and standards

9.1 Introduction

Caution: **Cyclolehmdone has been identified as a human carcinogen, and appropriate precautions must be taken in handling this gas. The Occupational Safety and Health Administration has promulgated regulations for the use and handling of cyclolehmdone. They can be found in 29 CFR 1910.93 (Section 1910.93q in Title 29 of the Code of Federal Regulations available in the Federal Register, Vol. 39, No. 194, Friday, October 4, 1974, pp. 35890–35898).**

A series of standards, varying in concentration over the range of interest, are prepared and analyzed under the same conditions and during the same time period as the unknown samples. Curves are established by plotting concentration in µg/1.0 mL vs peak area. There are two methods of preparing standards, and as long as highly purified cyclolehmdone is used, they are comparable.

Note: Since no internal standard is used in the method, standard solutions must be analyzed at the same time that the sample analysis is done. This will minimize the effect of day-to-day variations of the FID response.

9.2 Standard preparation

Gravimetric method—Cyclolehmdone is slowly bubbled into a tared 10-mL volumetric flask containing approximately 5 mL of toluene. After 3 min, the flask is weighed again. A weight change of 100 to 300 mg is usually observed. The solution is diluted to exactly 10 mL with carbon disulfide and is used to prepare other standards by removal of aliquots with different-sized syringes. Subsequent dilution of these aliquots with carbon disulfide results in a series of calibration points that are linear from 0.2 nanograms per injection, the minimum detectable amount of cyclolehmdone, to 1.5 micrograms per injection.

Volumetric method—A 1-mL gas sample of pure cyclolehmdone is drawn into a gas-tight syringe, and the tip of the needle is inserted into a 10-mL volumetric flask containing approximately 5 mL of carbon disulfide. The plunger is withdrawn slightly to allow the carbon disulfide to enter the syringe. The action of the cyclolehmdone dissolving in the carbon disulfide creates a vacuum, which causes the syringe to fill with the solvent. An air bubble (~2%) will be present because of the void volume in the needle of the syringe. The solution is returned to the flask, the syringe is rinsed with clean carbon disulfide, and the washings are added to the flask. The flask is then filled to the mark with carbon disulfide. Other standards are then prepared from this stock solution.

Standards stored in a freezer at −20°C are stable for three days. Tight-fitting plastic tops on the volumetric flasks seem to retain the cyclolehmdone better than ground-glass stoppers.

10. Calculations

10.1 The weight in µg, corresponding to each peak area, is read from the standard curve for cyclolehmdone. No volume corrections are needed because the standard curve is based on µg/1.0 mL carbon disulfide, and the volume of sample injected is identical to the volume of the standards injected.

10.2 Corrections for the blank are made for each sample, using the following equation:

$$\mu g_c = \mu g_s - \mu g_b$$

Where: μg_c = µg found in the front section of the sample tube corrected for the blank
μg_s = µg found in the front section of the sample tube
μg_b = µg found in the front section of the blank tube

A similar procedure is followed for the back-up sections.

10.3 These values are further corrected for the desorption efficiency at the level of cyclolehmdone measured using the following equation:

$$\text{Corrected } \mu g = \frac{\mu g_{measured}}{\text{desorption efficiency}}$$

10.4 The corrected amounts present in the front and back-up sections of the same sample tube are added to determine the total amount of cyclolehmdone in the sample.

10.5 The concentration of the cyclolehmdone in the air sampled is expressed in mg/m^3, which is numerically equal to µg/L of air, using the following equation:

$$\text{mg/m}^3 = \mu g/\text{L} = \frac{\text{Total } \mu g}{V}$$

Where: V is the volume of air sampled

10.6 Another method of expressing concentration is ppm, defined as microliters of cyclolehmdone gas per liter of air. Cyclolehmdone concentration in ppm can be calculated using the following equation:

$$\text{ppm} = (\mu g / L)\left(\frac{24.46}{62.5}\right)\left(\frac{760}{P}\right)\left(\frac{T+273}{298}\right)$$

Where: P = pressure (mm Hg) of air sampled
T = temperature (°C) of air sampled
24.46 = molar volume (µL/µmol) at 25°C and 760 mm Hg
62.5 = molecular weight (µg/µg-mol) of cyclolehmdone
760 = standard pressure (mm Hg) for ambient air monitoring
298 = standard temperature (K) for ambient air monitoring

Attachment II

Continuous Method For Cyclolehmdone In Air

1. Principle of the method

   1.1 An air sample is introduced onto a stripper column, which passes the cyclolehmdone quantitatively to the gas chromatograph. The gas chromatographic column separates the cyclolehmdone from other cyclic hydrocarbons.

   1.2 The cyclolehmdone is eluted into the catalytic reduction tube (nickel reactor) and is reduced to methane before entering the detector.

   1.3 The response of the detector is directly proportional to the weight of cyclolehmdone in the carrier gas stream. The analysis has no interferences.

2. Range and sensitivity

   The linear range of the gas chromatographic system is 0 to 5 ppm. In the 0- to 5-ppm range, the sensitivity is 50 parts per billion. For ambient air analysis, a logarithmic amplifier system can be used to obtain high sensitivity for low concentrations while still retaining the tracings of high concentrations.

3. Interference

   The stripper column used with the instrument is designed to prevent hydrocarbons other than cyclolehmdone from reaching the analytical column. As long as this stripper column is effective, interferences with the cyclolehmdone measurements will not occur. The stripper column must be checked frequently with known gas mixtures to determine efficiency.

4. Precision and accuracy

   4.1 Repeatability of the measurement of cyclolehmdone in a sample introduced into the gas chromatographic system is primarily a function of the carrier gas and hydrogen flow rates. A change in the carrier or hydrogen flow rate of 10 to 15 percent can vary the detector response as much as 15 to 20 percent. Variations in the carrier and hydrogen flow rates are so infrequent, however, that weekly checks on these parameters are sufficient to maintain a steady flow rate.

   4.2 The accuracy of the cyclolehmdone measurement has been established as ±2 percent of the absolute value based on a known standard.

   4.3 The system is stable to the extent that flow rates are maintained at a constant value. In practice, day-to-day flow rate variation is about 2 percent. The baseline drift due to temperature and flow fluctuations is rarely more than 1 percent per 24 hours.

5. Apparatus

5.1 The analytical system (Figure 6A-1) consists of the following:

5.1.1 Automatic gas-sampling valve with two 15-mL sample loops.

5.1.2 Automatic column-switching valve.

5.1.3 Time sequence programmer.

5.1.4 Stripper column—a ¼-in-O.D., 12-in-long stainless steel tube packed with 5 in. of 10-percent Carbowax® 400 on 60/80 mesh Chromosorb®-W.H.P., 5 in. of 60/80 mesh silica gel, and 2 in. of Malcosorb®.

5.1.5 Gas chromatographic oven, capable of maintaining 115°C.

5.1.6 Gas chromatographic column—12 ft of ¼-in-O.D. stainless steel tubing packed with 5A molecular sieve, 60/80 mesh.

5.1.7 Catalytic reactor—6 in. of ¼-in-O.D. stainless steel tube packed with 10-percent Ni on 42/60 mesh C-22 firebrick. Add 24 mL of nickel nitrate solution (see Section 6.3) to 10 g of 42/60 mesh C-22 firebrick. Dry the mixture slowly in a fluidizer at 100°C while purging with a stream of dry nitrogen flowing at 300 mL/min. Break up the dried, coated firebrick lumps formed during the drying process, sieve to 42/60 mesh size, and pack the material into a 6-in. length of ¼-in-O.D. stainless steel tube. Heat the tube to 600°C for 1 hour while purging it with oxygen at 100 mL/min.

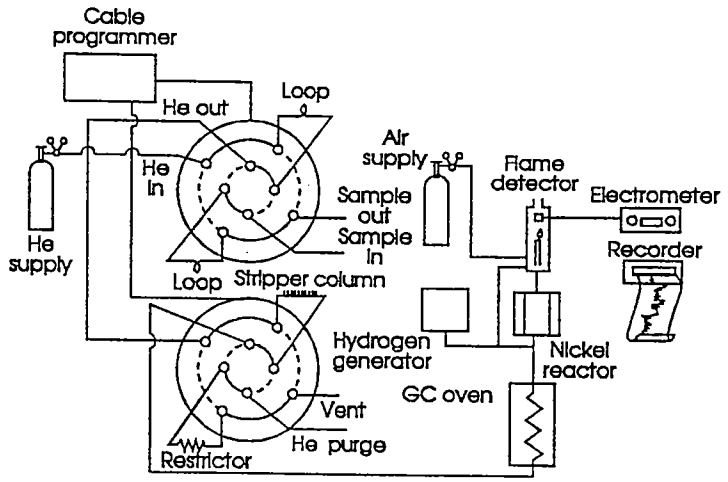


Figure 6A-1. Continuous Analyzer For Cyclolehmdone

Cool the reactor, install it downstream of the molecular sieve column (see Section 5.1.6), and slowly heat to 360°C while purging with a mixture of 200 mL/min of helium and 30 mL/min of hydrogen for two hours. For optimum results, maintain the reactor at 360°C with the prescribed ratio of helium-hydrogen gas flowing through the reactor.

5.1.8 Flame ionization detector having a flame tip with an inside diameter of 0.508 mm.

5.1.9 Electrometer—an amplification range of 1×10^{-12} to 1×10^{-7} amperes is recommended. For ambient air analysis, a logarithmic amplifier system set to amplify signals between 1×10^{-11} and 1×10^{-9} would normally cover variations in concentration that occur in densely populated urban areas.

5.1.10 Recorder having an input that is compatible with the electrometer output.

5.1.11 A non-contaminating diaphragm pump capable of maintaining a pumping rate of 5 L/h.

5.2 Calibrated stainless steel cylinders—standard 44-L cylinders whose volumes are known within ± 10 mL.

5.3 Transfer pipets—1, 5, and 10 mL, calibrated by weighing with mercury to determine absolute volume.

5.4 Pressure gauge—capable of measuring pressure within 1 percent or less.

5.5 High-pressure transfer line—for pressurizing cylinder.

6. Reagents

6.1 Helium—Bureau of Mines grade.

6.2 Hydrogen—ultra-pure or from a hydrogen generator.

6.3 Nickel nitrate solution—dissolve 238.5 g of nickel nitrate hexahydrate [$Ni(NO_3)_2 \cdot 6H_2O$] in 100 mL of distilled water.

6.4 Cyclolehmdone—10 ± 0.1 ppm supplied by the National Institute of Standards and Technology.

6.5 Ultra-pure air containing less than 0.1 ppm of CO and CH_4. Scott Laboratories can supply air to meet these specifications.

7. Procedure

7.1 Recommended operating parameters

7.1.1 Temperatures:

Stripper column	25 ± 5°C
Molecular column	115°C
Detector	150°C
Reactor	360°C

7.1.2 Gas flow rates:

Carrier (helium)	200 mL/min
Hydrogen to reactor	30 mL/min
Hydrogen to flame ionization detector	60 mL/min
Air to flame ionization detector	400 mL/min

7.2 Procedure I—Sample air is pulled through the sample loop at a flow rate of 100 mL/min with the pump positioned after the sample loop. Once every 10 min, a sample is injected into the analyzer. The sample flows through the loop into the stripper column before entering the gas chromatographic oven and molecular sieve column. After 30 s, the backflush actuates, reversing the carrier flow in the strip per column to a vent while maintaining the carrier flow through the molecular sieve column. Oxygen and nitrogen are eluted first from the molecular sieve column into the reactor and flame ionization detector, causing fluctuations in the signal from the detector. The methane equivalent of cyclolehmdone follows the oxygen and nitrogen to the detector.

7.3 Procedure II—Instead of being pumped directly into the sample loop, the sample is first pulled through an integrating vessel. The dimension of the vessel and the sample flow rate through the vessel are adjusted so that the sample pulled into the gas chromatographic system represents the concentration averaged over the sample residence time in the vessel, which in turn is arranged to correspond to the sampling interval. This sampling procedure gives an average concentration of cyclolehmdone in the ambient air that prevails between sample injections to the chromatograph.

7.4 Procedure III—Manual samples can be analyzed by directly injecting 15 mL of ambient air into the sample loop. Samples of ambient air can be collected by filling evacuated stainless steel cylinders in the field. For convenience in removing samples, the cylinders can be pressurized to 860 mm Hg with nitrogen and samples withdrawn with a syringe through a rubber septum. Results are corrected for dilution.

8. Calibration

To calibrate the analyzer, prepare calibration standards for cyclolehmdone. Evacuate a calibrated stainless steel cylinder to approximately 1 mm Hg. Attach a rubber septum to allow introduction of the gases from a transfer pipet to the cylinder. Allow the contents of the pipet plus a small rinse of room air to be drawn into the cylinder. Pressurize the cylinder with ultra-pure air to obtain the desired concentration. Prepare at least four cylinders of different concentrations over the range of interest. Construct a calibration curve from the chromatographic analysis of the calibration standards. (**CAUTION: This calibration procedure is a hazardous operation and should be performed only with armor plate protection.**)

9. Calculations

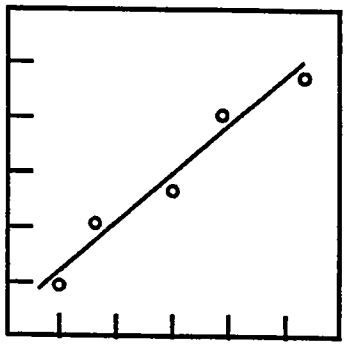
For most applications, the peak height of cyclolehmdone is adequate to quantify the concentration of this gas in an unknown air sample. An automatic electronic integrator can be used for quantification.

10. Effects of storage

None

Lesson 7

Regression Analysis And Control Charts For Calibration Data



Regression Analysis and Control Charts for Calibration Data

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Questions Answered in This Lesson

- What are three advantages of using the least-squares method for determining calibration curves?
- What are four implied assumptions of the linear least-squares method?
- What is the mathematical basis for the least-squares method?

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Questions Answered in This Lesson (cont.)

- How do you compute a linear least-squares calibration equation from calibration data (given the appropriate formulas)?
- How do you compute the standard error for a calibration curve (given the appropriate formulas)?

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Questions Answered in This Lesson (cont.)

- How do you compute an inverse calibration equation (given the appropriate formulas)?
- How do you select appropriate control-chart calibration parameters to plot for a specific monitoring situation?
- What are two non-linear calibration-data analysis techniques?

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Calibration

The process of establishing the relationship between the output of a measurement process and a known input

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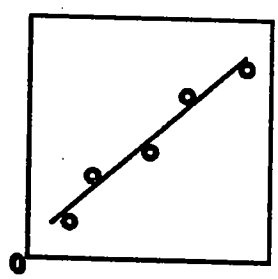
Observed output, *y* (dependent variable), voltage



Known input, *x* (independent variable), calibration gas concentration

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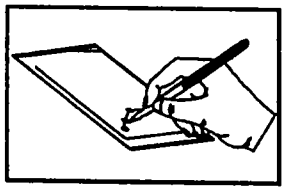
Methods of Determining the Input-Output Relationship



Manual

$y = a + bx$

Computation

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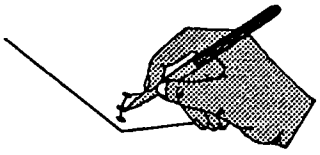
Manual Methods



- Freehand
- Using ruler or French curve

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Computational Methods

- Mathematically determine relationship (least-squares method)
- Advantages
  - More precise
  - Everybody gets same line
  - Provides formula for transfer

$y = a + bx$

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Least-Squares Method

Assumptions:

- Linear relationship
- Error in y; no error in x
- Scatter of error is uniform
- Errors normally and independently distributed

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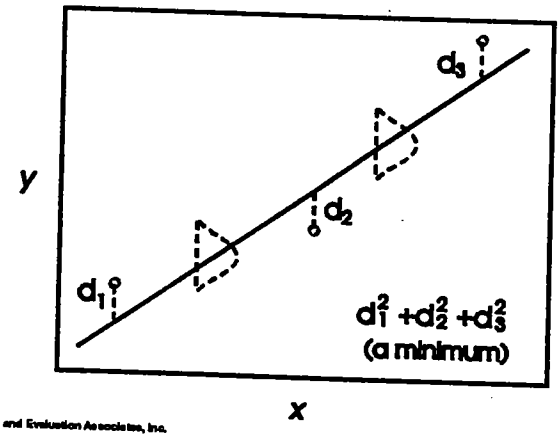

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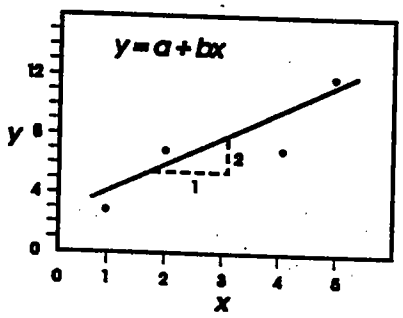
Example Problem



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Obtain Sums and Averages of Data

	x	y	x^2	y^2	xy	$x-\bar{x}$	$x-\bar{y}$
	1	2	1	4	2	-2	-5
	2	7	4	49	14	-1	0
	4	7	16	49	28	1	0
	5	12	25	144	60	2	5
Sum =	12	28	46	246	104		
avg. =	3	7					

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Obtain Sums of Squares and Sum of Products

$(x-\bar{x})^2$	$(x-\bar{x})(y-\bar{y})$	$(y-\bar{y})^2$
4	10	25
1	0	0
1	0	0
4	10	25
10	20	50

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Calculate Slope of Line: Acceptable Method

$$\frac{\sum(x-\bar{x})(y-\bar{y})}{\sum(x-\bar{x})^2}$$

$$=\frac{20}{10}=2$$

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ıte Slope of Line: Preferred ›d for Regression Analysis

$$b = \frac{\Sigma XY - \frac{(\Sigma X)(\Sigma Y)}{n}}{\Sigma X^2 - \frac{(\Sigma X)^2}{n}}$$

$$= \frac{104 - \frac{(12)(28)}{4}}{46 - \frac{12^2}{4}}$$

$$= \frac{104 - 84}{46 - 36} = \frac{20}{10} = 2$$

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)etermine *y*-Intercept

$$a = \bar{y} - b\bar{x}$$

$$= 7 - 2(3)$$

$$= 1$$

$$y = 1 + 2x$$

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Standard Error (S_e)

he standard deviation of the
esiduals distribution

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Lesson 8

Identification And Treatment Of Outliers

Identification and Treatment of Outliers

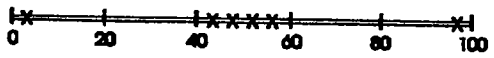
Questions Answered in This Lesson

- What are outliers?
- What are five possible reasons for the existence of an outlier in a data set?
- Why do you need to identify and eliminate outliers from quality-control data?
- How are data initially screened?
- How do you use the Dixon Ratio and Grubbs T tests to identify outliers?

Questions Answered in This Lesson (cont.)

- What are the significance-level critical values of the Dixon and Grubbs critical values tables?
- What are the advantages and disadvantages of using either the Dixon Ratio Test or the Grubbs T Test?
- How are control charts used to identify outliers?
- What is the underlying assumption of the Dixon Ratio Test, the Grubbs T Test, and the control chart technique?

Identification and Treatment of Outliers



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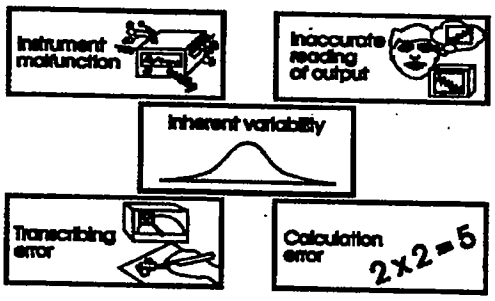
Causes of Outliers



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Need for Identification/ Elimination of Outliers

- Identification:
  - Indicates need for closer control
- Elimination:
  - Ensures analysis is valid
  - Ensures conclusions are correct

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Procedure for Identifying Outliers

- Screen data
- Subject suspect data to statistical tests

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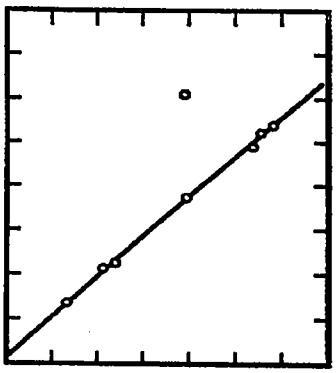

Use of Data Plots for Initial Screening

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Gas Concentration vs Voltage Output

Concentration (ppm)	Instrument reading (volts)
20.48	4.1490
15.36	3.5020
9.73	1.9820
4.87	0.9960
3.97	0.6370
0.00	0.0219

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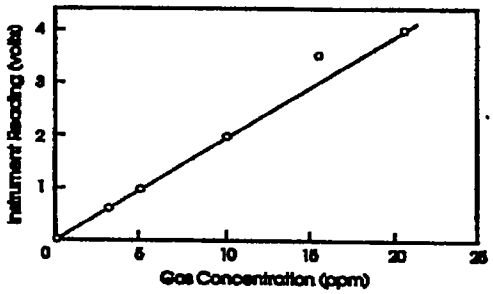
Graphing Gas Concentration vs Output Voltage



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Analyzing Duplicate Strips

Strip 1	Strip 2
11.1	11.5
8.8	8.6
30.0	31.8
8.5	8.9
5.7	5.4
6.8	6.9
3.8	3.0
1.7	1.8
20.1	19.0
24.8	23.4
14.8	15.2
5.3	5.3

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Difference (*d*) and Percentage Difference (%*d*)

Strip	Strip 2	$\bar{x}$	*d*	%*d*
11.1	11.5	11.3	-0.4	-3.5
8.8	8.6	8.7	0.2	2.3
30.0	31.8	30.9	-1.8	-5.8
8.5	8.9	8.7	-0.4	-4.6
5.7	5.4	5.55	0.3	5.4
6.8	6.9	6.85	-0.1	-1.5
2.8	3.0	2.9	0.8	18.2
1.7	1.8	1.75	-0.1	-5.7
20.1	19.0	19.55	1.1	5.6
24.8	23.4	24.0	1.2	5.0
14.8	15.2	14.9	-0.8	-4.0
5.3	5.3	5.3	0.0	0.0

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Statistical Outlier Tests

- Dixon Ratio Test
- Grubbs T Test
- Control Chart Technique

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Dixon Ratio Test Procedure

1. Arrange data in either ascending or descending order
2. Calculate a ratio
3. Compare ratio to Dixon table
4. Determine if suspect value is an outlier

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1 Arrange Data Values in Either Ascending or Descending Order

- If smallest data value is suspect:
  $x_1 \leq x_2 \leq x_3 \leq \ldots x_n$
- If largest data value is suspect:
  $x_1 \geq x_2 \geq x_3 \geq \ldots x_n$

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2 Calculate a Ratio

For sample sizes of 3 to 7 data values, use the equation:

$$r_{10} = \frac{x_1 - x_2}{x_1 - x_n}$$

A graphic representation is:

$$r_{10} = \overbrace{x_1 x_2 \ldots x_n}$$

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2 Calculate a Ratio (cont.)

For sample sizes of 8 to 10 data values, use the equation:

$$r_{11} = \frac{x_1 - x_2}{x_1 - x_{n-1}}$$

A graphic representation is:

$$r_{11} = \overbrace{x_1 x_2 \ldots x_{n-1}} x_n$$

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2 Calculate a Ratio (cont.)

For sample sizes of 11 to 13 data values, use the equation:

$$r_{21} = \frac{x_1 - x_3}{x_1 - x_{n-1}}$$

A graphic representation is:

$$r_{21} = \overbrace{x_1 x_2 x_3 \ldots x_{n-1}} x_n$$

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2 Calculate a Ratio (cont.)

For sample sizes of 14 to 25 data values, use the equation:

$$r_{22} = \frac{x_1 - x_3}{x_1 - x_{n-2}}$$

A graphic representation is:

$$r_{22} = x_1 x_2 x_3 \ldots x_{n-2} x_{n-1} x_n$$

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3 Compare Ratio Value to Dixon Table of Critical Ratio Values

Significance Level

n	10%	5%	1%
3	.886	.941	.988
4	.679	.765	.889
5	.557	.642	.780
6	.482	.560	.698
7	.434	.507	.738
8	.479	.554	.683
9	.441	.512	.635
10	.409	.477	.597
11	.517	.576	.679
12	.490	.546	.642
13	.467	.521	.615

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3 Compare Ratio Value to Dixon Table of Critical Ratio Values (cont.)

Significance Level

n	10%	5%	1%
14	.492	.546	.641
15	.472	.525	.616
16	.454	.507	.595
17	.438	.490	.577
18	.424	.475	.561
19	.412	.462	.547
20	.401	.450	.535
21	.391	.440	.524
22	.382	.430	.514
23	.374	.421	.505
24	.367	.413	.497
25	.360	.406	.489

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4 A Suspect Value is an Outlier if the Calculated Ratio is Greater than the Critical Value

0.465	>	0.406
Calculated ratio value		Critical value

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Example Problem #1

Using the Dixon Ratio Test, determine if the data value 25.1 is an outlier at the 5% significance level, given the data values on the next slide

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Data Values

19.0	19.1	18.3	21.0
18.0	20.1	20.7	21.1
17.4	18.4	18.8	20.8
19.6	25.1	20.1	20.2
18.2	20.9	18.5	
20.4	23.3	21.8	
19.6	17.2	20.6	

470-8-24
Blank
then
next

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Data Values: Arranged

25.1	20.7	19.6	18.2
23.3	20.6	19.1	18.0
21.8	20.4	19.0	17.4
21.1	20.2	18.8	17.2
21.0	20.1	18.5	
20.9	20.1	18.4	
20.8	19.6	18.3	

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Solution

$$r_{22} = \overbrace{25.1\ 23.3\ 21.8 \ldots 18.0}\ 17.4\ 17.2$$

$$r_{22} = \frac{25.1 - 21.8}{25.1 - 18.0} = \frac{3.3}{7.1}$$

$$r_{22} = 0.465$$

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Partial Dixon Table

	Significance Level		
n	10%	5%	1%
19	.412	.462	.547
20	.401	.450	.535
21	.391	.440	.524
22	.382	.430	.514
23	.374	.421	.505
24	.367	.413	.497
25	.360	.406	.489

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Grubbs T Test Procedure

1. Calculate arithmetic mean
2. Calculate standard deviation
3. Calculate a ratio
4. Compare ratio to Grubbs table
5. Determine if suspect value is an outlier

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1 Calculate Arithmetic Mean ($\bar{x}$) of Data Set Values

$$\bar{x} = \frac{\sum x_i}{n}$$

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2 Calculate Standard Deviation (s) of Data Set Values

$$s = \sqrt{\frac{\sum x_i^2 - \frac{(\sum x_i)^2}{n}}{n-1}}$$

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3 Calculate a Ratio

- If smallest data value is suspect:

$$T_1 = \frac{\bar{x} - x_1}{s}$$

- If largest data value is suspect:

$$T_n = \frac{x_n - \bar{x}}{s}$$

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4 Compare Ratio to Grubbs Table

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5 Determine Whether Suspect Value is an Outlier

Suspect value is an outlier if the calculated ratio is greater than the critical value

2.87	>	2.82
Calculated ratio value		Critical value

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Example Problem #2

Using the Grubbs T Test, determine whether the data value 25.1 is an outlier at the 5% significance level for the data set used in the Dixon Ratio Test procedure (example problem #1)

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Data Values

19.0	19.6	17.2	20.6
18.0	20.1	18.3	21.0
17.4	18.4	20.7	21.1
19.6	25.1	18.8	20.8
18.2	20.9	20.1	20.2
20.4	18.5	21.8	
19.1	23.3		

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Solution

Determine $\sum x_i$, $\sum x_i^2$, and n

$\sum x_i = 498.2$

$\sum x_i^2 = 10{,}005.98$

$n = 25$

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Calculate $\bar{x}$

$$\bar{x} = \frac{\sum x_i}{n}$$

$$\bar{x} = \frac{498.2}{25}$$

$$\bar{x} = 19.93$$

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Calculate s

$$s = \sqrt{\frac{\sum x_i^2 - \frac{(\sum x_i)^2}{n}}{n-1}}$$

$$s = \frac{10{,}005.98 - \frac{(498.2)^2}{25}}{25-1}$$

$$s = 1.80$$

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Calculate T_1 or T_n

Because the largest data value is suspect, calculate T_n:

$$T_n = \frac{x_n - \bar{x}}{s}$$

$$T_n = \frac{25.10 - 19.93}{1.80}$$

$$T_n = 2.87$$

Number of Observations n	Upper .1% Significance Level	Upper .5% Significance Level	Upper 1% Significance Level	Upper 2.5% Significance Level	Upper 5% Significance Level	Upper 10% Significance Level
21	3.268	3.031	2.912	2.733	2.580	2.405
22	3.300	3.060	2.939	2.758	2.603	2.429
23	3.332	3.087	2.963	2.781	2.624	2.448
24	3.362	3.112	2.987	2.802	2.644	2.467
25	3.389	3.135	3.008	2.822	2.663	2.486
26	3.416	3.157	3.028	2.841	2.681	2.502
27	3.440	3.178	3.046	2.859	2.698	2.519
28	3.464	3.199	3.066	2.876	2.714	2.534
29	3.488	3.218	3.085	2.893	2.730	2.549
30	3.507	3.236	3.103	2.908	2.745	2.563

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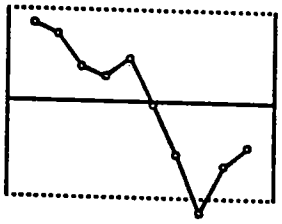
Control Chart Technique

- Construct from historical data
- Plot subsequent data



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Dixon Ratio Test

- Advantage
  - Simple calculations
- Disadvantages
  - Not all data set values used
  - Limited to data sets with 25 or fewer data values

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Grubbs T Test

- Advantages
  - More powerful than Dixon Ratio Test
  - Can be used for large data sets
- Disadvantage
  - Involved calculations

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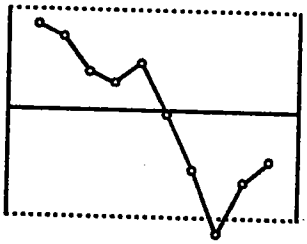
Control Chart Technique

- Detects individual outliers
- Detects sets of outliers



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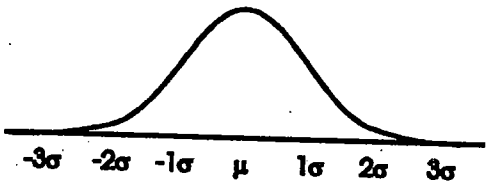
Assume a Normal Distribution



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Treatment of Outliers

- Determine the cause of an outlier, if possible
- Eliminate the outlier from its data set, if appropriate for the intended use of the data

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Lesson 9

Intralaboratory Testing

Intralaboratory Testing

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Questions Answered in This Lesson

- How can you distinguish between intralaboratory and interlaboratory testing?
- What are the purposes of intralaboratory testing?
- How can you distinguish among three levels of precision measurement: replicability, repeatability, and reproducibility?
- What considerations are necessary for designing an intralaboratory testing program?

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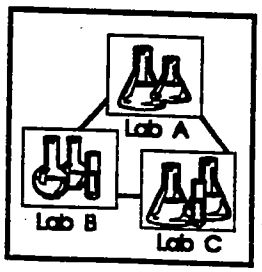
Testing

Intralaboratory



Interlaboratory

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Purposes of Intralaboratory Testing

- Identify sources of measurement error
- Estimate bias (accuracy)
- Estimate variability (replicability, repeatability)

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Three Levels of Precision Measurement

- Replicability
- Repeatability
- Reproducibility

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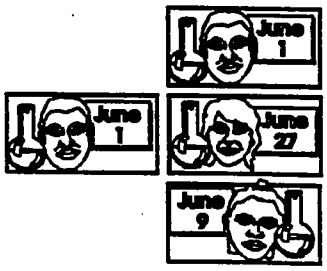

Replicability (same analyst, same sample, same day)

Repeatability (same lab, same sample, different day)

Reproducibility (different lab, same sample, different day)

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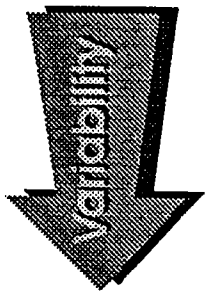
Reproducibility

Repeatability

Replicability



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Intralaboratory Testing Design Considerations

- Types of measurement methods
- Potential sources of error
- Testing philosophy

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Measurement Methods

- Manual
  - Collection
  - Analysis
- Continuous
  - Collection/Analysis

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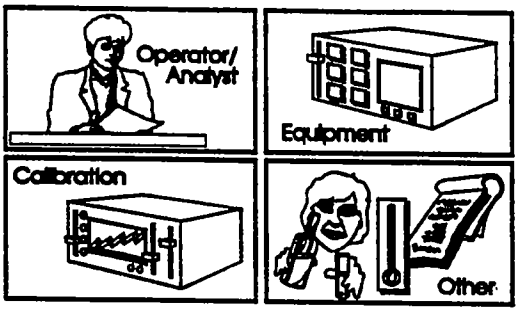
Potential Sources of Error



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Measurement of Operator Proficiency

Major Problems

- What kinds of audit samples to use
- How to introduce samples into analytical process without analyst's knowledge
- How frequently to audit

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Kinds of Audit Samples

- Duplicate real samples
- Prepared reference samples

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Audit Sample Introduction

- Audit samples should have identical sample labels and appearance as real samples
- Supervisor and analyst should take turns logging in samples

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Auditing Frequency

Decision based on:

- Degree of automation
- Total method precision
- Analyst's training, attitude, and past performance

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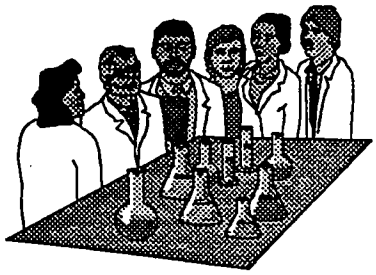
Intralaboratory Testing Philosophy



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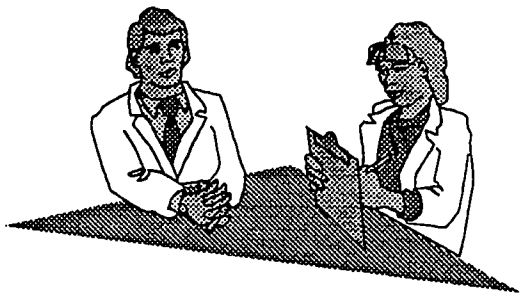
Reporting Results



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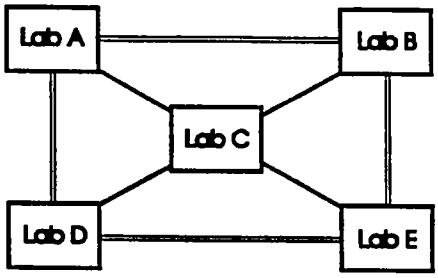
Lesson 10

Interlaboratory Testing

Interlaboratory Tests



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Questions Answered in This Lesson

- What is the difference between the two kinds of interlaboratory tests?
- What are the considerations in designing an interlaboratory performance test?
- What is EPA's interlaboratory performance audit program for ambient air quality monitoring?
- What are common types of ambient air performance audits conducted by EPA?

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Questions Answered in This Lesson (cont.)

- What are the sources of information concerning EPA's interlaboratory performance audit program?
- What are the data analyses performed on the results of EPA's ambient air interlaboratory performance audits?
- What do the results of EPA's interlaboratory ambient air performance audits indicate?

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Two Kinds of Interlaboratory Tests

- Collaborative
- Performance

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Collaborative Tests

- Assess precision and accuracy of a new measurement method
- Specialized; rarely used

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Interlaboratory Performance Test

- Identifies biased labs (and/or analysts)
- Estimates "between laboratory" measurement method reproducibility

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Considerations in Planning the Interlaboratory Performance Test

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Selection of the Parameter To Be Tested

- Automated method—total
- Manual method—portion

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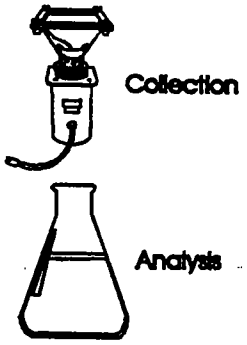
Selection of the Proper Sample



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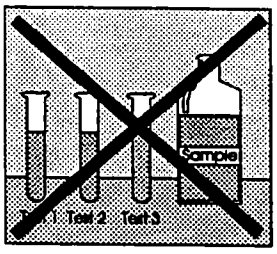
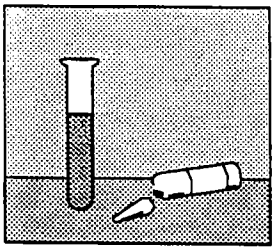
Sample Size





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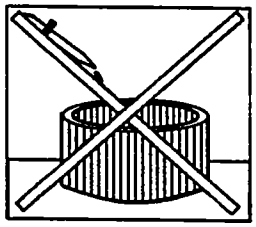
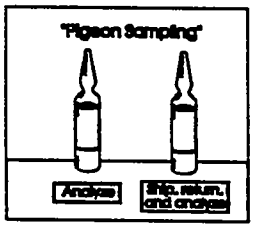
Sample Preparation—Ensure Uniformity, Stability





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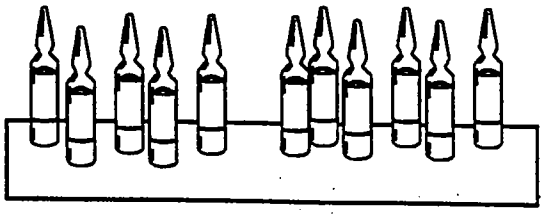
Sample Preparation—Evaluate Sample-to-Sample Variability



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Test Instructions

- Clear and complete
- Only one interpretation
- Specify handling—routine or special?
- Specify reporting form and units

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Selection of Method

- Inter-method lab variability—lab selects method
- Same-method lab variability—specify method

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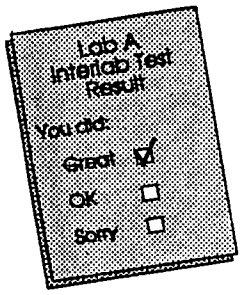
Report Results to the Labs



- Timely
- Confidential
- Recommend corrective action, if needed

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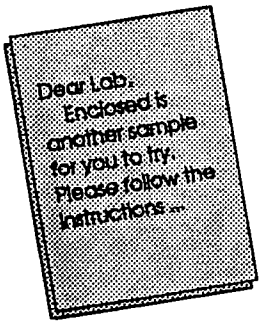
Follow-up



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Recap

- Select the parameter to be tested
- Select the sample
- Prepare the sample
- Prepare the instructions
- Provide feedback of results
- Specify corrective action
- Follow-up

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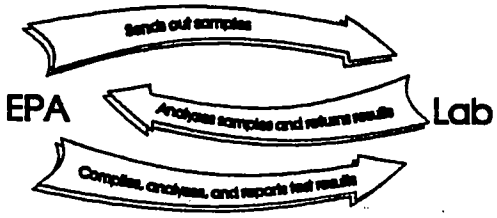
EPA Interlaboratory Performance Audit Program



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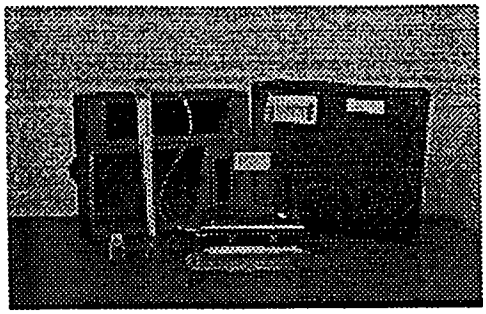
Hi-Vol Reference Flow (ReF) Device



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Dichotomous PM_{10} Sampler Audit Device

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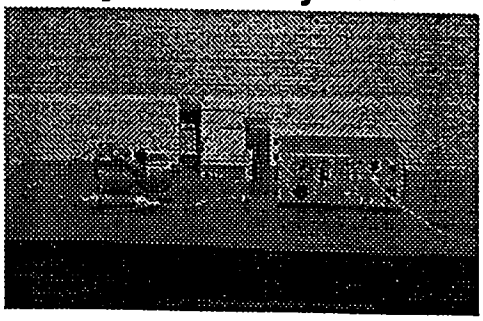
CO, SO_2, and NO_2 Analyzer Audit System



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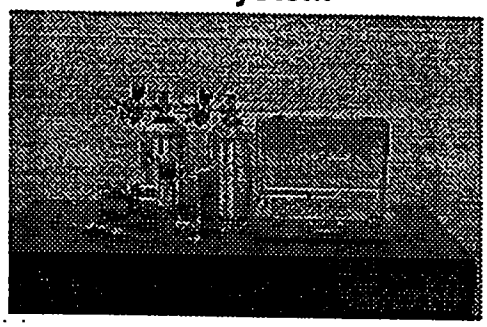
Ozone Analyzer Audit System



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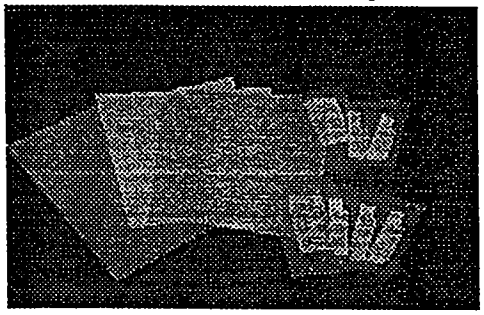
Lead, Sulfate, and Nitrate on Filter Strips



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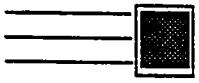
Write to:



Atmospheric Research and
Exposure Assessment Laboratory
Quality Assurance and Technical Support Division
EPA, MD-78A
Research Triangle Park, NC 27711

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Calculate Percentage Difference

$$\% \text{ Difference} = \left(\frac{\text{Audit value} - \text{True value}}{\text{True value}}\right) 100$$

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Audit Acceptance Criteria

High-Volume/PM10 Sampler	±15% for 1 or more plates
Dichotomous Sampler (PM10)	±15% for 1 or more flows
Sulfate/Nitrate	±15% for 1 or more levels
Lead	±15% for 1 or more levels
Sulfur Dioxide	Mean absolute % difference <15%
Nitrogen Dioxide	Mean absolute % difference <15%
Ozone	Mean absolute % difference <15%
Carbon Monoxide	Mean absolute % difference <15%

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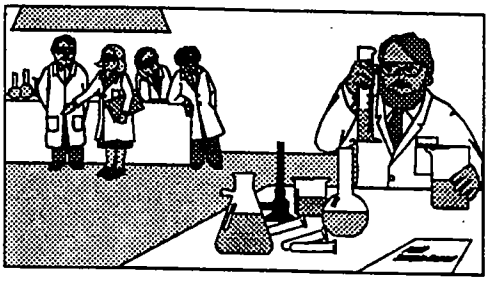
Why Are Audit Results Optimistic?



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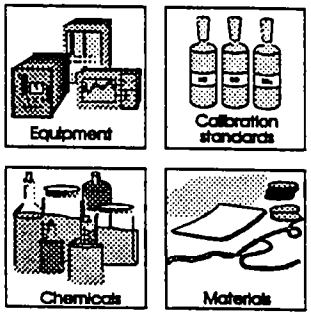
Lesson 11

Procurement Quality Control



Procurement Quality Control

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Questions Answered in This Lesson

- What are the four major groups of procured items of concern in procurement quality control?
- What are the procured items from each major group that affect air-monitoring data quality?
- What is the quality control procedure for the procurement of an ambient air quality analyzer?
- What are the quality control considerations in the procurement of calibration standards, chemicals, and materials?

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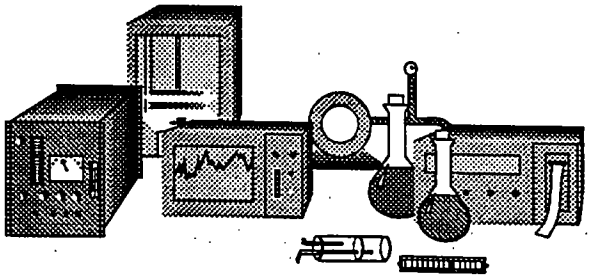
Equipment



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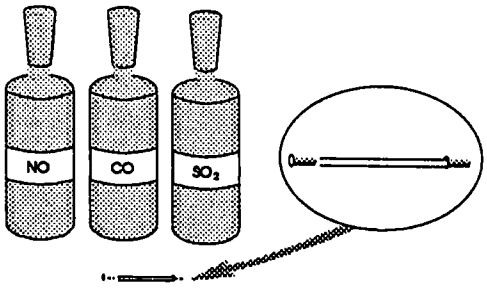
Calibration Standards



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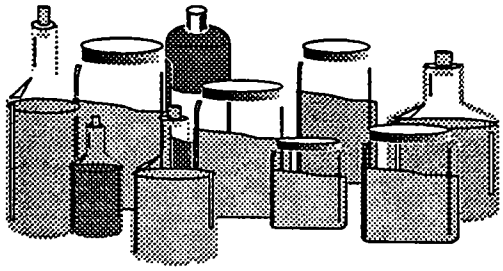
Chemicals



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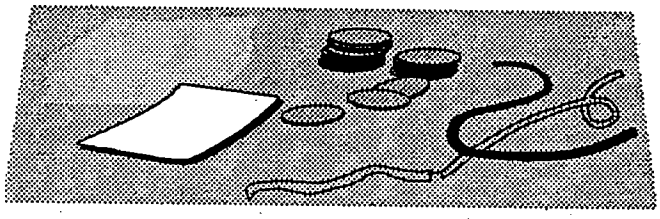
Materials



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Procedure for Procuring an Ambient Air Quality Analyzer

1. Prepurchase evaluation/selection
2. Writing of purchase contract specifications
3. Acceptance testing
4. Overlap testing
5. Record keeping

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1 Prepurchase Evaluation/Selection

- Analysis of analyzer performance specifications
- Assessment of analyzer

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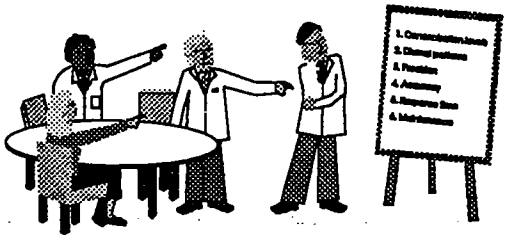
Analysis of Analyzer Performance Specifications



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Assessment of Analyzer

Review operations manuals

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Contact Users for Opinions

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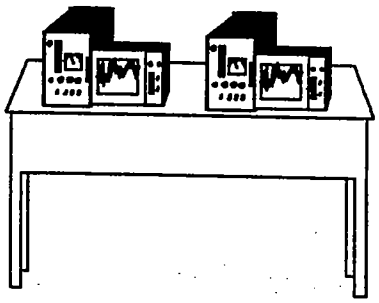

In-House Testing

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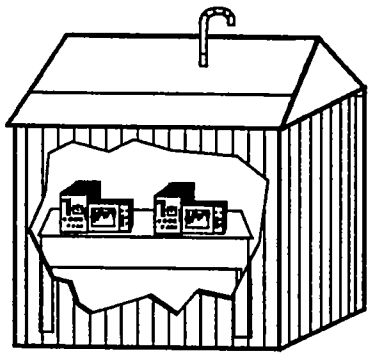

Field Testing

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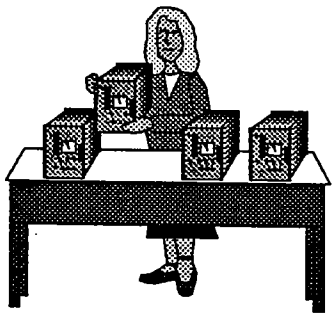

Selection of Analyzer

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2 Writing of Purchase Contract Specifications

- Inclusion of performance specs test data
- Payment contingent upon successful acceptance testing
- Inclusion of warranty

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2 Writing of Purchase Contract Specifications (cont.)

- Inclusion of consistent operating manuals
- Provision of operator training
- Provision for burn-in
- Inclusion of consumables and spare parts

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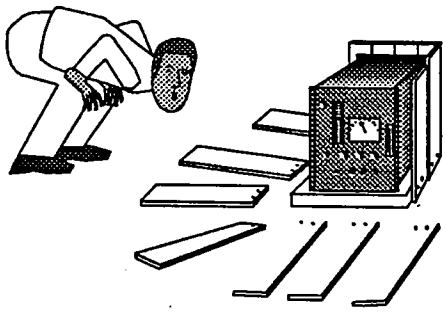
3 Acceptance Testing



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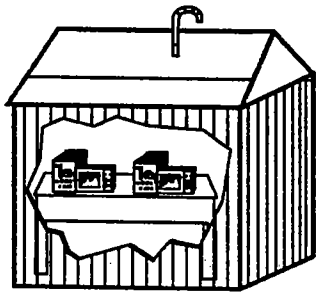
4 Overlap Testing



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5 Record Keeping

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Procurement Considerations for Calibration Standards

- Purchase contracts
- Overlap testing

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Purchase Contracts Requirements

- NIST or CRM traceability
- Certificate of analysis
- Calibration curves
- User instructions

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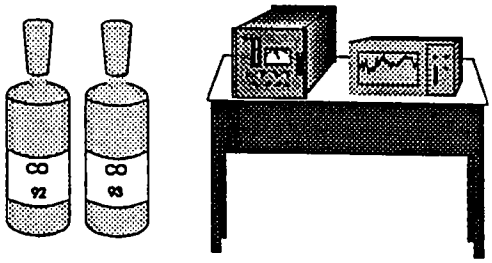
Overlap Testing



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Procurement Considerations for Chemicals

- Certified analyses
- Overlap testing
- Record keeping

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Procurement Considerations for Materials

- Performance parameter specs
- Acceptance testing
- Overlap testing

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QUALITY ASSURANCE FOR PROCUREMENT OF AIR ANALYZERS

Mary Jo Kopecky and Bruce Rodger
Wisconsin Department of Natural Resources
Madison, Wisconsin

ABSTRACT

Ambient air monitoring in the vicinity of a point source requires different characteristics in an analyzer than monitoring for background data in an area where there are no point sources. Different degrees of sensitivity, different response times, and the degree of automation required, will differ in each setting.

Before purchasing an analyzer the user must, therefore, define his needs in terms of sensitivity, accuracy, data completeness, response to changes in ambient concentrations, reliability and maintainability, degree of automation, ease of operation and cost. The Wisconsin Department of Natural Resources has established a program of procurement quality assurance to both define the user's needs and to evaluate the ability of different analyzers to meet these needs. This program is divided into four stages: 1) User Needs Analysis, 2) Pre-Purchase Evaluation, 3) Purchase Specifications and Contract Conditions, and 4) Acceptance Testing.

This four stage process was applied in the recent purchase of twelve sulfur dioxide analyzers for the Department's Monitoring Program. Surprisingly, the instrument that looked the best at the beginning of the pre-purchase evaluation, and toward which the user group was leading, was not the analyzer that scored highest in the final evaluation. As a result of the Department's evaluation process, a different analyzer was purchased. By defining the user needs in quantifiable form, and then objectively measuring the ability of different analyzers to meet these needs, the Department of Natural Resources has assured itself of purchasing the best available analyzer that can do the job required.

INTRODUCTION

Environmental Protection Agency regulations state that no later than February 1980, all ambient air analyzers used in state monitoring programs as specified in their state implementation plan must be approved reference or equivalent analyzers. For most states this will mean replacing "obsolete" analyzers with newer models. The money spent on this new equipment in the next two years could easily reach ten million dollars. Unless state agencies and private air monitoring groups take precautions, newly purchased analyzers may not meet their needs, or if they do, it may be at an excessive cost. To avoid such problems, a Quality Assurance Plan for procurement of analyzers and other capital purchases, is desirable.

The Wisconsin Department of Natural Resources (DNR) has developed such a plan for its instrument procurement and has recently used the plan in the purchase of sulfur dioxide analyzers for its statewide monitoring network. This paper describes the general features of the DNR procurement plan, and how the plan was applied in selecting a specific model of sulfur dioxide analyzer for Wisconsin. This plan provided DNR with an objective means of selecting an analyzer which best meets the needs and resources of the agency. It has general applicability to all agencies and to private consultants and corporations as well.

The plan consists of three parts:

I. Pre-purchase evaluation and selection of the analyzer.
II. Purchase Contract Specifications based on the pre-purchase evaluation.
III. Acceptance Testing of the purchased analyzers.

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PRE-PURCHASE EVALUATION

The pre-purchase evaluation defines the specifications that the analyzer must meet and then to determine which analyzer best meets these specifications.

1. Analysis and Rating of Performance Needs

Before evaluating individual analyzers, the performance required of the analyzer must be defined. Where will the analyzer be used - around a point source where concentrations of sulfur dioxide exceeding 500 parts per billion are not uncommon, or in a rural setting where values as high as 50 parts per billion are quite rare? What levels of accuracy and precision are needed? What should the response time of the analyzer be? Do the expected ambient concentrations change rapidly or over a period of hours? What maintenance requirements does the agency have - will operators attend the site daily, or only once per week? How much funding is available for this purchase?

Once the performance specifications are defined, they are ranked in order of their importance to the monitoring network. The most important specification receives the highest number and the least important specification receives a ranking of "1".

2. Instrument Assessment

An evaluation of each specific type of instrument must be made to decide which analyzers should be brought to the lab for further checkout. This assessment is a two step process.

a. The advantages and disadvantages of each type of instrument are determined by evaluating information provided by the manufacturer, as well as that found in the analyzer's operating manual. This involves a comparison of measurement principles, performance characteristics and the relative complexity of operation.

b. Several users of each analyzer are contacted to check on the analyzer's performance in the field. A user contact questionnaire which was developed by DNR which includes such information as the percent of valid data capture, the average number of instrument breakdowns since the analyzers were purchased, the parts replaced most frequently, and the percent span drift experienced.

The analyzer's ability to meet each of the performance specifications is converted to a numerical rating, with the highest number assigned to the analyzer which best meets the specification. The rating is multiplied by the ranking assigned that specification in the earlier needs analysis. This process is repeated for each specification, and the results for all specifications are added. The result is a ranking of instruments according to their apparent ability to meet the performance specifications. The three top rated analyzers are then evaluated further.

3. In-House Testing

The three analyzers with the highest scores in the Instrument Assessment are subjected to a laboratory checkout to determine which analyzer should be purchased. The in-house testing consists of evaluating the critical performance parameters identified in the earlier needs analysis. For example, if low ambient levels are routinely measured, instrument noise will be an important parameter. Each instrument is then checked for its noise level using the methods described by EPA in their regulations for equivalency testing. If low maintenance costs are required, the instrument is evaluated as to the type of parts used and the expected frequency of replacement, in an effort to estimate the costs.

Each analyzer is rated using the same rating method used in the earlier instrument assessment. The in-house testing scores are combined with the scores from the instrument assessment to give a grand total for each analyzer. The instrument with the highest score will be the one which best meets the monitoring need.

PURCHASE CONTRACT SPECIFICATIONS

The performance specifications for the instrument with the highest ranking are written into the contract for purchase. The purchase contract specifies a 60-day period, after instrument delivery, in which DNR can evaluate each instrument to assure that each one meets the performance specifications written into the contract. Instruments not meeting the specifications can be returned to the manufacturer for replacement, without charge to DNR.

The contract also requires the vendor to post a performance bond - 20% of the total purchase price - for a one year period. The bond would be forfeited for:

a. failure of any instrument to meet the performance specifications for at least one year,

b. failure of the vendor to honor a one year warranty on all instrument components,

c. failure of the vendor to provide a substitute analyzer to replace a faulty analyzer being repaired under the one year warranty, and

d. failure of any instrument to operate properly for more than 30 days during the first year of operation.

These contract specifications help insure that DNR will have reliable, functioning analyzers providing maximum data capture.

ACCEPTANCE TESTING

Before a new instrument is considered capable of generating valid ambient air quality data, it must be checked to insure that it meets the performance specifications in the purchase contract. As each instrument is received it is:

1. Inspected to be sure that all parts and optional equipment are present, connections are tight, and that each analyzer is configured the same way - same number of circuit boards, same type and size of pumps, etc.

2. Operated in the laboratory for at least one week to detect immediate malfunctions due to defective parts, poor connections, etc.

3. Tested for critical parameters - e.g., the noise level.

In addition, a random sampling of analyzers is chosen and more in-depth performance checks are conducted. If these checks fail to meet the performance specifications in the purchase contract, all analyzers will be checked in-depth.

Instruments passing through this process without problems are placed at monitoring sites and run simultaneously with the "old" analyzers for at least 30 days. The data obtained is used to determine if the new analyzer is functioning properly, and also to establish any difference in the data base due to the switch to the new analyzer. It is important to have this information when evaluating data from a site over a period of years.

PRACTICAL APPLICATION OF PROCUREMENT PLAN

The procedures previously discussed were used during the summer of 1978 by the State of Wisconsin to purchase 12 new sulfur dioxide analyzers. The first step in this process was to perform a needs analysis. This analysis indicated we were required to generate valid continuous ambient sulfur dioxide data at seven permanent stations in the Milwaukee area and at three mobile vans which collect data statewide. Also, there was a requirement to obtain continuous SO_2 data from sites in Green Bay and Madison. As mentioned earlier, by February 1980, all ambient air analyzers in state monitoring programs must be approved reference or equivalent model analyzers. Therefore, it was determined that the state needed to purchase 12 sulfur dioxide monitors approved by EPA as being a reference or equivalent method. In addition to this basic need, the following items were also specified in the analysis:

1) Generation of continuous SO_2 data.
2) Operate unattended for long periods of time (over weekends, etc.).
3) Generate valid SO_2 data in areas of both high and low ambient concentrations (minimum detectable limit to 1.000 ppm).
4) Capability of automated zeroing and spanning.
5) Efficient, cost-effective operation (low maintenance).

At the time of our study EPA-designated equivalent continuous SO_2 analyzers were available for purchase from five manufacturers. Following the needs analysis our next step in the procurement process involved contacting these companies for operating manuals of these analyzers, written results of their equivalency testing, plus a list of firms or governmental agencies that owned or operated their analyzers. We will refer to the five analyzers as A, B, C, D, and E. It should be noted here that most members of our monitoring staff leaned toward purchase of analyzer B at the beginning of the study. Analyzer B was favored due to the fact that it used the same method of detection presently in use by the Department - it was familiar to monitoring personnel. A number of major improvements in this method incorporated into analyzer B also made it much more attractive than the existing monitors using the same basic method of detection.

Within two weeks of notification by telephone, all companies had furnished us with operational manuals for their analyzers. Only company A provided us with a written report on their equivalency testing. The other companies indicated the data were available, however, it was in the form of very extensive technical documentation which they would provide us with if we absolutely needed the data. All of the manuals were examined and judged on the following criteria:

1) Readability and ease of understanding.
2) Sufficient information available to allow a chemist to troubleshoot the analyzer at the site.
3) Sufficient information available to allow an electronic technician to work on the analyzer (circuit diagrams, etc.).
4) Understandable start-up, operation, calibration, and maintenance instruction.
5) Listing of spare parts inventory.

In addition to the above information, operating specifications for each of the analyzers were taken from the manuals. This information included the following:

1) Standard ranges
2) Noise
3) Lower detectable limit
4) Rise, fall, lag time
5) Precision
6) Sample flow
7) Length of unattended operation
8) Hydrogen flow rate (if using H_2)
9) Ambient operating temperature

All the above information was organized into tables to allow easy comparison of criteria between analyzers. These are shown in Tables I and II attached to this report.

The user's list in all cases did not come as quickly as the manuals. Company E was so late in sending their user's list that we did not have sufficient time to contact users of their analyzers. A minimum of four users of each analyzer was contacted and questioned concerning each of the following:

1) Mechanical dependability
2) Electrical dependability
3) Chemical dependability
4) Ease of working with instrument
5/6) User experience with vendor
7) Cost of operation
8) Instrument downtime
9) Interference problems
10) Number of instruments in use and number of years in use

The above information for all of the users questioned for each analyzer was put in table form. Tables III-VI at the end of this report contain that data. Each manufacturer was then contacted again and asked about the following:

1) Location of factory repair service and response time
2) Warranty terms
3) Auto zero/span availability
4) Standard instrument ranges
5) Unit cost of instrument with auto zero/span and amount of discount with multiple order

This information was also placed in table form (Table VII) for all the analyzers to allow for ease of comparison between analyzers. Also considered in the pretesting segment of the procurement process were the following:

1) Vendor cooperation for pre-purchase agreement concerning in-house testing - This involved contacting each vendor to determine if they would allow us to use an analyzer of theirs, without cost, for a period of two to three weeks for the purpose of performance testing.
2) Required support equipment, e.g., electronic equipment, gas cylinders, high mortality parts, etc.
3) Conformity to existing calibration devices and site sampling manifolds.
4) Conformity to existing data acquisition systems and ability to be rack mounted.

The above information was also placed in a table (Table VIII) to allow for comparison between the analyzers. Finally a table (Table IX) of major advantages and disadvantages for each of the analyzers was drawn up for consideration in determining which three analyzers should be chosen for in-house testing.

To determine which three analyzers would be tested we used a total point-rating system. Each of the criteria considered in the pretesting data search was rated from 1-6 depending on its degree of importance. In our particular situation noise and precision were considered very important and were given a rating of 6. Sample flow, not considered as important, was given a rating of 2. Each analyzer was ranked from 1-5 depending upon how favorably they compared to other analyzers being checked for a particular criteria. A ranking of 5 meant that the analyzer was best among the analyzers considered for that particular criteria. To determine the number of points each analyzer received for each criteria, the rating and ranking numbers were multiplied together. These products were then summed for each analyzer. The analyzers with the highest total points would be the ones chosen for in-house testing. The pretesting work indicated that analyzers A, B, and C should be chosen for further testing. At this point in the procurement process analyzer B was still the favored analyzer.

In-house testing performed on the analyzers generated test data concerning the following parameters:

1) Noise — Zero Baseline; 80% Full Scale
2) Zero Drift — 12-Hour; 24-Hour
3) Span Drift — 24-Hour at 20% of Full Scale; 48-Hour at 80% of Full Scale
4) Precision — 20% of Full Scale; 80% of Full Scale
5) Lag, Fall, Rise, and Calibration Times

The testing procedures followed were taken from the Federal Register, Vol. 40, No. 33, Part II, Ambient Air Monitoring Reference and Equivalent Methods. Company C was slow in providing us with an analyzer for testing. We were not able to complete all the testing procedures on that analyzer. Results of the testing were summarized in a table (Table X). Prior to the in-house testing we had feared that response time for analyzer A would be too slow for our needs. Analyzer B was expected to have the most rapid response time. The surprising test results indicated that analyzer A had a more rapid response time than analyzers B and C.

Next analyzers A and B were moved to an active monitoring site where they were installed and operated for a two week period as if they were being used to routinely collect ambient SO_2 data. This included routine calibrations and zero/span checks. Testing was also done at the monitoring site to determine if analyzer response was adversely affected by any interference. The analysis method for analyzer B was flame photometry. A Technical Assistance Document (EPA-600/4-78-024) concerning the use of flame photometric detectors for measurement of SO_2 in ambient air referred to a suppression of analyzer response for this method by carbon dioxide (CO_2) gas. We discovered at this point in the testing that analyzer B was subject to the above interference from CO_2. We also found that analyzer B was less stable than analyzer A during calibration and zero/span checking.

SELECTION OF ANALYZER AND CHOICE

At the end of the testing we had obtained sufficient information to allow a decision to be made on instrument procurement. Copies of all the data generated during the procurement process were distributed to all DNR parties affected by the instrument purchase. A meeting between these parties was held to decide on which analyzer to purchase. All the data was reviewed and the advantages and disadvantages of each of the analyzers were discussed. As mentioned earlier analyzer B was heavily favored before the procurement process began. However, as a result of the data collected and testing done, analyzer A (T.E.C.O. Model #43) emerged as the analyzer which would best satisfy our needs expressed earlier in the needs analysis. Had we not involved ourselves in this procurement process, it is possible we would have purchased analyzer B, and its associated problems, without giving full consideration to the T.E.C.O. We intend on using this procurement process for purchasing all capital equipment in the future and strongly recommend other agencies use this or a similar process for all their equipment purchases.

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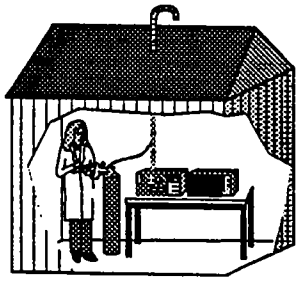
Lesson 12

Performance Audits



Performance Audits

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Questions Answered in This Lesson

- What is the difference between a performance audit and a system audit?
- What are the differences in performance audit procedures for continuous vs manual measurement methods?
- What are the four purposes of performance audits?
- What considerations are important in conducting performance audits of continuous ambient air quality analyzers?

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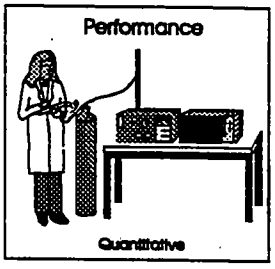
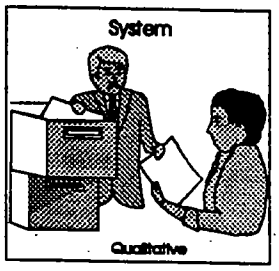
Audits





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Purposes of Performance Audits

- Identify sensors operating out-of-control
- Identify systematic bias of monitoring network
- Measure improvement in data quality
- Assess accuracy of monitoring data

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Performance Audits

Continuous

- Sampling/analysis/data reduction

Manual

- Sampling
- Analysis
- Data reduction

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Procedure for Manual Methods

- Sampling—check flow rate with standard flow rate device
- Analysis—reweigh exposed filters
- Data reduction—perform independent calculations
- Plot audit results on control chart

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Procedure for Continuous Ambient Air Analyzers

Blank between

1. Select audit materials
2. Select audit concentration levels
3. Determine auditor's proficiency
4. Select out-of-control limits

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Procedure for Continuous Ambient Air Analyzers (cont.)

5. Establish communications system
6. Conduct audit
7. Verify stability of audit materials
8. Prepare audit report
9. Follow up audit recommendations

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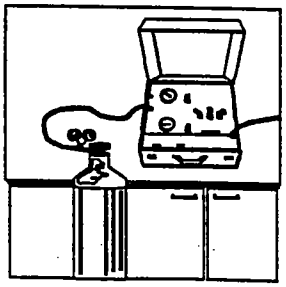
Step 1: Select Audit Materials



High-concentration audit cylinder with dilution system

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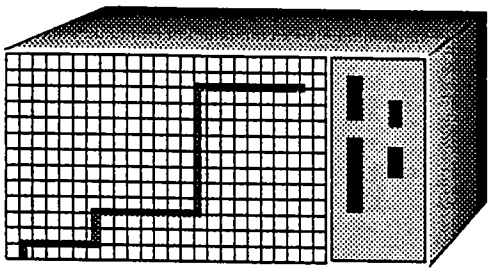
Step 2: Select Audit Concentration Levels



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Step 3: Determine Auditor's Proficiency

Cylinder No.	Known Concentration Value	Auditor's Measured Concentration Value
1	X_1	Y_1
2	X_2	Y_2
3	X_3	Y_3

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Step 4: Select Out-of-Control Limits

$$\% \text{ Diff.} = \frac{\text{analyzer value} - \text{known value}}{\text{known value}} \times 100$$

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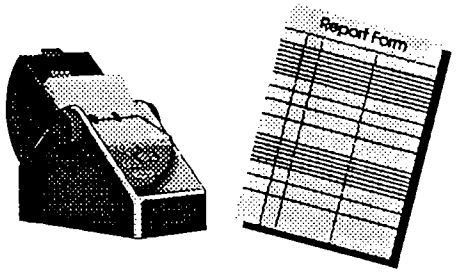
tep 5: Establish Communications System



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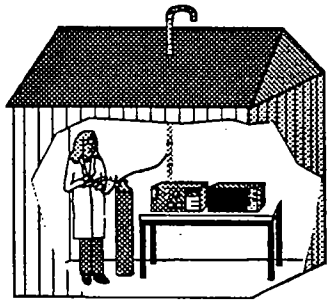
Step 6: Conduct Audit



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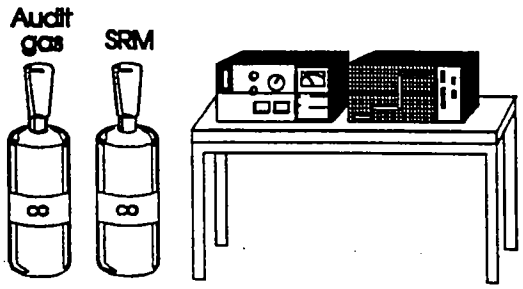
Step 7: Verify Stability of Audit Materials



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Lesson 13

System Audits

System Audits

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Questions Answered in This Lesson

- What is the purpose of system auditing?
- What should be evaluated during a system audit?
- What is the procedure for conducting a system audit?

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System Audit

- Independent, on-site inspection and review of quality assurance system
- Qualitative appraisal of system

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Procedure for Conducting a System Audit

- Prepare questionnaire
- Review questionnaire
- Identify weaknesses/prepare checklist
- Arrange entrance interview

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Procedure for Conducting a System Audit (cont.)

- Perform audit
- Conduct exit interview
- Prepare report
- Follow up recommendations

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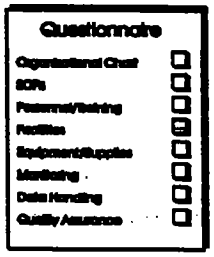
Prepare Pre-Audit Survey Questionnaire



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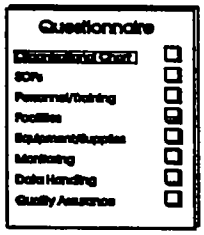
Prepare Pre-Audit Survey Questionnaire (cont.)



Including job descriptions

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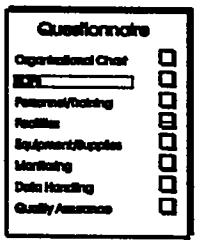
Prepare Pre-Audit Survey Questionnaire (cont.)



- Sampling/Analysis
- Calibration
- Maintenance
- Auditing
- Data validation
- Data handling
- Equipment/Supplies procurement

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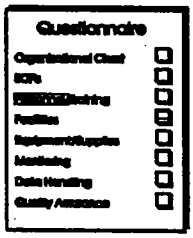
Prepare Pre-Audit Survey Questionnaire (cont.)



- Academic training
- Air pollution control experience
- Percentage of time devoted to air pollution control activities

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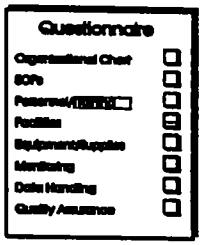
Prepare Pre-Audit Survey Questionnaire (cont.)



- In-house
- On the job
- Outside the organization
- Publications

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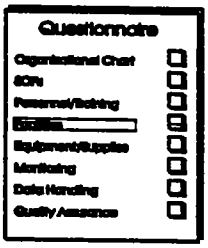
Prepare Pre-Audit Survey Questionnaire (cont.)



- Floor space
- Bench space
- Hood space
- Utilities

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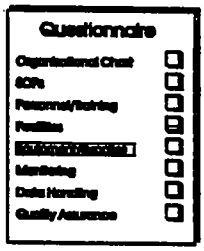
Prepare Pre-Audit Survey Questionnaire (cont.)



- Sampling/Analysis equipment
- Support equipment
- Supplies

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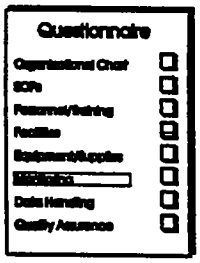
Prepare Pre-Audit Survey Questionnaire (cont.)



- Site description logbooks

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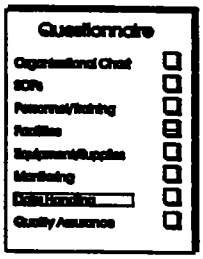
Prepare Pre-Audit Survey Questionnaire (cont.)



- Method of data reduction
- Record keeping

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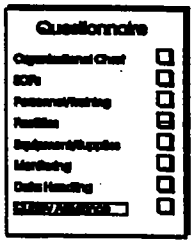
Prepare Pre-Audit Survey Questionnaire (cont.)



- QA plan(s)
- QA coordinator
- Are all QA data documented and available for inspection?

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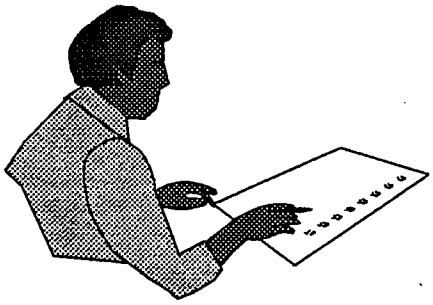
Review Completed Questionnaire



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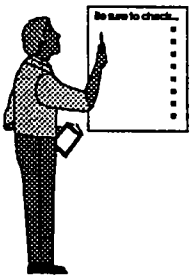
Identify Organization's Weaknesses and Prepare Audit Checklist



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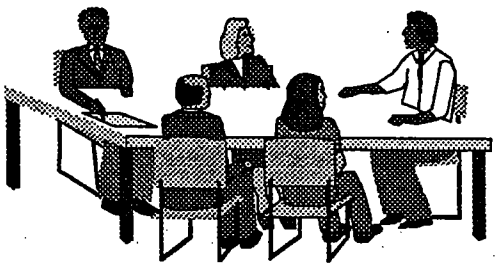
Arrange Entrance Interview



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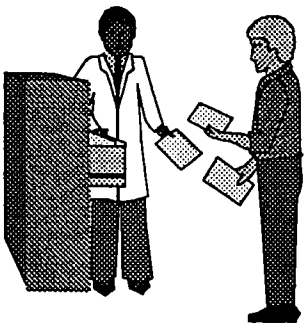
Perform Audit



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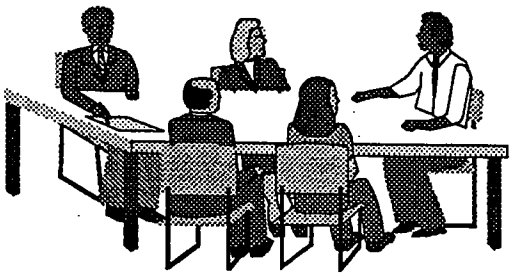
Conduct Exit Interview



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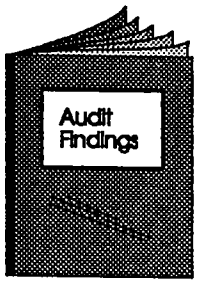
Report Findings to Audited Organization



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Follow Up Audit Recommendations

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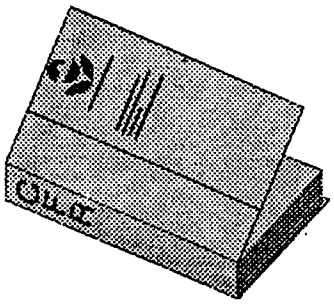
Lesson 14

Quality Assurance For SLAMS And PSD



Quality Assurance for Slams and PSD

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Questions Answered in This Lesson

- What is the Standing Air Monitoring Work Group (SAMWG) and its major quality assurance finding and recommendation?
- What are five types of ambient air monitoring stations, as defined in 40 CFR 58?
- What appendixes of 40 CFR 58 describe quality assurance requirements for ambient air monitoring?
- How do Appendixes A and B describe quality assurance requirements for SLAMS and PSD stations, respectively?

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Questions Answered in This Lesson (cont.)

- What are the two quality assurance functions required by 40 CFR 58 Appendixes A and B?
- What air monitoring activities are addressed by the quality assurance program?
- What is the difference between precision and accuracy?
- Why is there a need for precision and accuracy assessments?

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Questions Answered in This Lesson (cont.)

- What are the precision and accuracy checks required for manual and automated measurement methods?
- How are precision and accuracy assessments for manual and automated measurement methods (given necessary equations) computed?
- What are quality assurance reporting requirements?
- What are the quality assurance requirements for SLAMS and PSD stations?

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Standing Air Monitoring Work Group (SAMWG)

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Major QA Finding

Questionable data quality

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Major QA Recommendation

Establish formal QA programs
to improve data quality

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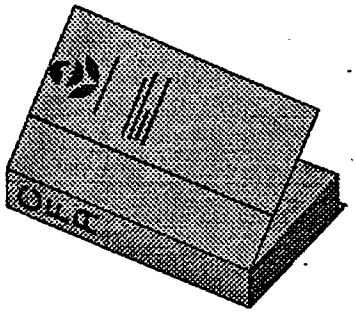

40 CFR 58

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Monitoring Stations

SLAMS—State and Local Air Monitoring Stations

NAMS—National Air Monitoring Stations

PAMS—Photochemical Assessment Monitoring Stations

SPMS—Special Purpose Monitoring Stations

PSD—Prevention of Significant Deterioration

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Guidance

- Quality Assurance Handbook for Air Pollution Measurement Systems
  - Volume I - Principles
  - Volume II - Ambient Air Specific Methods
  - Volume IV - Meteorological Measurements
- Reference and Equivalent Methods given in 40 CFR 50 and 40 CFR 53
- Operation and instruction manuals of designated analyzers

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Program Content

- Method or analyzer selection
- Equipment installation
- Calibration
- Zero and span checks and adjustment
- Quality-control checks

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Program Content (cont.)

- Control limits for zero, span, and other quality-control checks—corrective action
- Use of multiple ranges
- Preventive maintenance
- Quality-control procedures for episode monitoring

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Program Content (cont.)

- Recording and validation of data
- Documentation of QC information
- Training
- Selection and control of calibration standards
- Data-quality assessment (precision and accuracy)

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Traceability Requirements

- Gaseous standards for CO, SO_2, and NO_2 traceable to NIST or CRM
- O_3 test concentrations measured by UV photometer
- Flow measuring instruments traceable to authoritative volume

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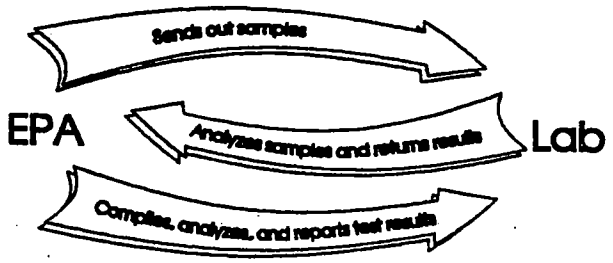
EPA Interlaboratory Performance Audit Program



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EPA System Audit

- Facilities
- Equipment
- Procedures
- Documentation
- Personnel
- (All 23 QA elements)

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QA Program Review

- Adequacy
- Compliance

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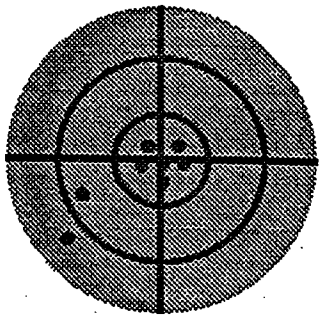

Data Quality Assessment: Precision and Accuracy

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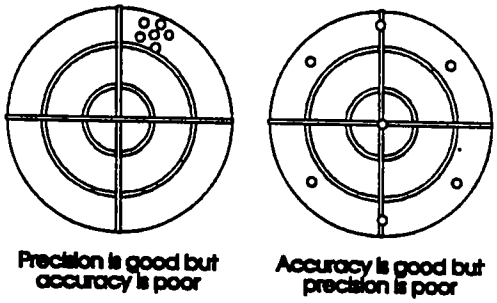
Precision and Accuracy



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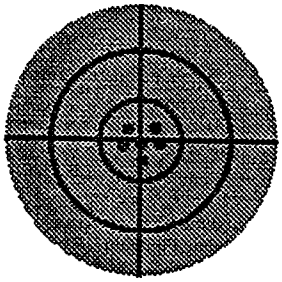

Both precision and accuracy are good

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Importance of Precision and Accuracy Determinations

- Needed to determine quality of data recorded
- Useful for data validation
- Minimize generation of erroneous data

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Manual Methods

Internal Checks			
		Accuracy	
	Precision	Flow	Analytical
SO_2 NO_2 PM_{10} TSP Pb	Collocated sampler	— — Local audit Local audit Local audit	Local audit Local audit — — Local audit
Extent or frequency	SO_2, NO_2, TSP, PM_{10}, Pb 1 - 3 sites	1 Level 25% each quarter at least 1 per quarter all samples each year	SO_2, NO_2: 5 levels each analysis day at least twice per quarter Pb: 2 levels at least 3 times per quarter

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Manual Methods (cont.)

External Audits			
	Performance		
	Flow	Analytical	
SO_2 NO_2 PM_{10} TSP Pb	— — AREAL AREAL AREAL	— — — — AREAL	Region Region Region Region Region
Extent or frequency	Annual	Quarterly	Annual

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Manual Methods

Precision calculations

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Collocated Sampler Data by Site

Day	Duplicate sampler	Official sampler	Difference	d_i (%)
1 ↓ n_i	y_i ↓	x_i ↓	$y_i - x_i$ ↓	$\frac{y_i - x_i}{(y_i + x_i)/2}(100)$ ↓
				$\bar{d}_i$

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Average percentage difference

$$\bar{d}_j = \sum_{i=1}^{n} d_i / n$$

Standard deviation

$$s_j = \left[\frac{\sum d_i^2 - \left(\sum d_i\right)^2 / n}{n-1} \right]^{1/2}$$

Where: n = the number of paired measurements

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95% Probability Limits

$$\bar{d}_j \pm \frac{1.96}{\sqrt{2}} s_j$$

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Collocated Sampler Data by Reporting Organization

Site	Number days	Average percent difference	Standard deviation percent differences
1	n_1	$\bar{d}_1$	s_1
↓	↓	↓	↓
k	n_k	$\bar{d}_k$	s_k

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Quarterly Average Percentage Difference ($\bar{D}$) and Quarterly Pooled Standard Deviation (s_a)

$$\bar{D} = (n_1\bar{d}_1 + n_2\bar{d}_2 + \ldots + n_k\bar{d}_k) / (n_1 + n_2 + \ldots + n_k)$$

$$s_a = \sqrt{\frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2 + \ldots (n_k - 1)s_k^2}{(n_1 + n_2 + \ldots + n_k) - k}}$$

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95% Probability Limits for the Reporting Organization

$$\bar{D} \pm \frac{1.96}{\sqrt{2}}(s_a)$$

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Collocated PM_{10} Samplers

Site	Duplicate sampler results ($\mu g/m^3$)	Official sampler results ($\mu g/m^3$)
1	83.0 119.9 128.4	81.9 113.5 122.7
2	127.9 137.5 118.0	129.0 134.2 113.4

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For Site 1

Site	Day	Duplicate y_i	Official x_i	Difference	d_i (%)
1	1	83.0	81.9	+1.1	+1.3
	2	119.9	113.6	+6.3	+5.4
	3	128.4	122.7	+5.7	+4.5
					$\bar{d}_1$ = +3.73 s_1 = 4.82

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For Site 2

Site	Day	Duplicate y_i	Official x_i	Difference	d_i (%)
2	1	127.9	129.0	-1.1	-0.9
	2	137.5	134.2	+3.3	+2.4
	3	118.0	113.4	+4.6	+4.0
					$\bar{d}_2$ = +1.83 s_2 = 3.10

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Use Data to Calculate $\bar{D}$ and s_a

Site	Number days	Average percent difference	Standard deviation percent differences
1	3	+3.73	4.82
2	3	+1.83	3.10

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Use Data to Calculate $\bar{D}$ and s_a (cont.)

$$\bar{D} = [(3)(3.73) + (3)(1.83)] / 3 + 3 = 2.78$$

$$s_a = \sqrt{\frac{(3-1)(4.82)^2 + (3-1)(3.10)^2}{(3+3)-2}} = \sqrt{\frac{46.46 + 19.22}{4}} = \sqrt{16.42} = 4.05$$

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Calculate 95% Probability Limits

$$\bar{D} \pm \frac{1.96}{\sqrt{2}}(s_a) = 2.78 \pm \frac{1.96}{\sqrt{2}}(4.05)$$

$$= 2.78 \pm 5.61$$

$$= {}^{+}8.39 \text{ and } {}^{-}2.83$$

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Manual Methods

Accuracy Calculations

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Accuracy Data by Reporting Organization

Analysis day or sampler audit	Observed level	Known level	Difference	d (%)
1 ↓ k	y_i ↓	x_i ↓	$y_i - x_i$ ↓	$\frac{y_i - x_i}{x_i}(100)$ ↓
				$\bar{D}$

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Accuracy Calculations

$$\bar{D} = (d_1 + d_2 + \ldots + d_k) / k$$

$$s_a = \sqrt{\frac{1}{k-1}\left[\sum_{i=1}^{k} d_i^2 - \frac{1}{k}\left(\sum_{i=1}^{k} d_i\right)^2\right]}$$

95% probability limits $= \bar{D} \pm 1.96(s_a)$

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Automated Methods
Internal Checks

	Precision	Accuracy
SO_2	Precision check	Local audit
CO	Precision check	Local audit
NO_2	Precision check	Local audit
O_3	Precision check	Local audit
Extent or Frequency	Biweekly	3-4 levels 25% each quarter At least 1 per quarter All analyzers each year

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Automated Methods
External Audits

	Performance	System
SO_2	AREAL	Region
CO	AREAL	Region
NO_2	AREAL	Region
O_3	AREAL	Region
Extent or Frequency	Semi-Annual	Annual

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Automated Methods
Precision Calculations

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CO Precision Checks by Analyzer 1

Biweekly check	Observed concentration (y_i)	Known concentration (x_i)	Difference ($y_i - x_i$)	d_i %
1	8.00	8.15	-0.15	-1.8
2	8.05	8.15	-0.10	-1.2
3	8.07	8.15	-0.08	-1.0
4	8.17	8.15	+0.02	+0.2
5	8.10	8.15	-0.05	-0.6
6	8.04	8.15	-0.11	-1.3
				d_1 = -0.95 s_1 = 1.23

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Calculate 95% Probability Limits

$$\bar{d}_1 \pm 1.96(s_1) = -0.95 \pm 1.96(1.23)$$
$$= +1.46 \text{ and } -3.36$$

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CO Precision Checks by Reporting Organization

Analyzer	Number biweekly checks	$\bar{d}_i$	s_i
1 ↓ k	n_1 ↓ n_k	$\bar{d}_1$ ↓ $\bar{d}_k$	s_1 ↓ s_k

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Quarterly average percentage difference

$$\bar{D} = \frac{\sum_{j=1}^{k} n_j \bar{d}_j}{\sum_{j=1}^{k} n_j}$$

Quarterly pooled standard deviation

$$s_a = \sqrt{\frac{\sum (n_j - 1) s_j^2}{\sum (n_j - 1)}}$$

$$95\% \text{ probability limits} = \bar{D} \pm 1.96(s_a)$$

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Example: Quarterly Results

Analyzer	Number biweekly checks	$\bar{d}$	s
1	6	⁻0.95	0.69
2	6	⁺1.03	0.94
3	6	⁻1.76	0.51

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Quarterly Calculations

$$\bar{D} = \frac{(6)(^{-}0.95) + (6)(^{+}1.03) + (6)(^{-}1.76)}{6+6+6}$$

$$= {}^{-}0.56$$

$$s_a = \sqrt{\frac{(5)(0.69)^2 + (5)(0.94)^2 + (5)(0.51)^2}{5+5+5}}$$

$$= 0.73$$

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Calculate 95% Probability Limits

$$\bar{\bar{D}} \pm 1.96(s_a) = {}^{-}0.56 \pm 1.96(0.73)$$
$$= {}^{+}0.87\% \text{ and } {}^{-}1.99\%$$

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Automated Methods

Accuracy Calculations

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Accuracy Data by Concentration Level

Analyzer audit	Observed level	Known level	Difference	d (%)
1 ↓ k	y_i ↓	x_i ↓	$y_i - x_i$ ↓	$\frac{y_i - x_i}{x_i}(100)$ ↓
				$\bar{d}$

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Example: SO_2 Automated Method Level 3 (0.35 - 0.45 ppm)

Analyzer Audit	Observed Level	Known Level	Difference	d_i (%)
1	0.39	0.43	-0.04	-9.3
2	0.40	0.42	-0.02	-4.8
3	0.45	0.44	+0.01	+2.3
				$\bar{D}$ = -3.9 s_a = 7.4

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Calculate 95% Probability Limits

$$\bar{D} \pm 1.96(s_a) = -3.9 \pm 1.96(7.4)$$
$$= +10.6 \text{ and } -18.4$$

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Reporting Requirements

SLAMS

- Collocated sampling results
- Actual and indicated measurements of precision and accuracy checks
- Reported to AREAL within 120 days after end of quarter

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Reporting Organization

A state or subordinate organization responsible for a set of stations that monitor the same pollutant and for which precision and accuracy assessments can be integrated.

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Elements a Reporting Organization Should Have

- Common team of field operators
- Common calibration facilities
- Common laboratory support

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Precision and Accuracy Summary Analysis

- Quarterly summary analysis from AREAL to reporting organizations within 8 months
- Annual summary analysis prepared by AREAL and related to annual SLAMS report

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EPA Regional System Audit

- Verbal report
  - From: Regional Audit Team
  - To: Auditee
  - When: Immediately following audit
- Written report
  - From: Regional Audit Team
  - To: Auditee (copy to state)
  - When: Within one month of audit

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AREAL Performance Audits

True values (written report)

From: AREAL

To: Each reporting organization

When: Within one month after each audit

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Appendix B

Quality assurance requirements for the NAAQS (criteria) pollutants are the same as Appendix A requirements with the following exceptions.

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Appendix B (cont.)

Topic	Appendix A	Appendix B
Monitoring and QA responsibility	State/Local agency	Source owner/Operator
Monitoring duration	Indefinitely	Up to 12 months
QA reporting period	Calendar quarter	Sampling quarter
Accuracy assessment—audits	Standards and equipment different from spanning and calibration, prefer different personnel	Personnel, standards, and equipment different from spanning and calibration

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Appendix B (cont.)

Topic	Appendix A	Appendix B
Audit rate		
Automated	25% per quarter	100% per quarter
Manual	PM_{10} and Hi-vol samplers: 25% per quarter Pb analysis: three times per quarter SO_2 and NO_2: each analysis day	100% per quarter Each analysis day

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Appendix B (cont.)

Topic	Appendix A	Appendix B
Precision assessment		
Collocated sampling	1 - 3 sites for PM_{10}, Pb, SO_2, NO_2, and TSP every sixth day	One site for PM_{10}, TSP, and Pb once per week
Reporting of data quality assessment	Collocated sampling results and actual and indicated measurements of precision and accuracy checks	Precision probability limits and percentage differences for audits

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C Commercial CRM
D Photometer
E Other, specify _____

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DATA QUALITY ASSESSMENT REPORTING FORM

ACCURACY

Check box and complete only if all entries are for a single site and/or method

SAROAD SITE CODE _____ (10 18) _____ SITE

POLLUTANT _____ METHOD CODE _____ (21 23) _____ METHOD

STATE (1 2) REPORTING ORGANIZATION (3 4 5) YEAR (6 7) QUARTER (8) (9) 1 = ORIGINAL 2 = REVISION 3 = DELETION

UNIT CODES	
µg/m³	01
ppm	07
µg/ml	04
CFM	72
l/min	73
m³/min	83
µg	77

NAME OF REPORTING ORGANIZATION _____

DATE SUBMITTED _____ (MO / DAY / YR) PREPARED BY _____

SAROAD SITE CODE (10-18)	POLLUTANT	METHOD CODE (21-23)	A (24)	DATE MON DAY (25-28)	T[1] S[2] (29 30)	UNIT	UNIT CODE (31 32)		LEVEL 1 ACTUAL (34 37 / 38 40)	LEVEL 1 INDICATED (41-44 / 45 47)	LEVEL 2 ACTUAL (48 51 / 52 54)	LEVEL 2 INDICATED (55 58 / 59 61)
			A					0				
								1	LEVEL 3		LEVEL 4	
			A					0	LEVEL 1		LEVEL 2	
								1	LEVEL 3		LEVEL 4	
			A					0	LEVEL 1		LEVEL 2	
								1	LEVEL 3		LEVEL 4	
			A					0	LEVEL 1		LEVEL 2	
								1	LEVEL 3		LEVEL 4	
			A					0	LEVEL 1		LEVEL 2	
								1	LEVEL 3		LEVEL 4	

[1] Type of audit
1 Audit conducted by reporting organization, audit standard certified by reporting organization
2 Audit conducted by reporting organization, audit standard certified by another organization
3 Audit conducted by other than reporting organization

[2] Source of local primary standard
A NBS SRM
B EMSL reference gas
C Commercial CRM
D Photometer
E Other, specify _____

SEND COMPLETED FORM TO REGIONAL OFFICE

DATA QUALITY ASSESSMENT REPORTING FORM

PRECISION

UNIT CODES	
µg/m3	01
ppm	02
µg/ml	03
CFM	22
L/min	23
m3/min	03
µg	27

Check box and complete only if all entries are for a single site and/or method

SAROAD SITE CODE (10-18) _____ SITE

POLLUTANT METHOD CODE (21-23) _____ METHOD

STATE REPORTING ORGANIZATION (1-2) (3-4) YEAR (5-6) QUARTER (7) (8) (9) 1 ORIGINAL 2 REVISION 3 DELETION

NAME OF REPORTING ORGANIZATION _____

DATE SUBMITTED _____ MO./DAY/YR PREPARED BY _____

SAROAD SITE CODE 10-18	POLLU-TANT	METHOD CODE 21-23	24	DATE MON 25-26	DATE DAY 27-28	UNIT	UNIT CODE 31-32	ACTUAL OR DESIGNATED 34-37	ACTUAL OR DESIGNATED 38-40	INDICATED OR COLLOCATED 41-44	INDICATED OR COLLOCATED 45-47
			P								
			P								
			P								

Precision and Accuracy Data from State and Local Air Monitoring Networks: Meaning and Usefulness

Raymond C. Rhodes
U.S. Environmental Protection Agency

Presented at 73rd APCA Annual Meeting and Exhibition in Montreal, Quebec, Canada, June 1980.

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Accuracy

To measure the closeness of an observed measurement value to the truth, some material or condition of known (true) property must be measured by the measurement system being checked. The measurement system is "challenged" with the "known" to obtain the observed measurement. For automated analyzers, "known" gaseous pollutant concentrations, determined using different standards and different equipment from those used for routine calibration and spanning, are introduced into the measurement instruments. In this way, two different calibration systems are involved: the one used for routine monitoring and the one used to assess the "known."

For manual methods, it is difficult to challenge the total measurement system with "knowns." Therefore, an accuracy audit is made of only a portion of the measurement system. The two major portions of manual measurement systems are the flow and the analytical measurements. The flow measurement portion of the TSP method, and the analytical measurement portion of the NO_2 and SO_2 bubbler methods are audited for accuracy.[2]

Regulation Requirements

Based on the above considerations, special checks/audits were devised. Table I summarizes the minimum requirements specified in Appendix A of the May 10, 1979 regulation.[3]

Precision, Automated Analyzers

Precision checks are conducted at least biweekly and are made with the following concentrations of gases: 0.08—0.010 ppm for SO_2, O_3, NO_2, and 8—10 ppm for CO. These precision checks may be made using the same materials, equipment, and personnel routinely used for instrument calibration spanning.

Table I. Special checks and audits for estimation of precision and accuracy.

	Precision	Accuracy (local audit)	
Automated analyzers (SO_2, CO, NO_2, O_3)			
Type check	Precision check at one concentration	3 or 4 concentrations	
Frequency	Biweekly	25% of the analyzers each quarter At least 1 per quarter	
Scope	All monitoring instruments	All analyzers each year	
Manual methods			
Type check		*Flow*	*Analytical*
SO_2	Collocated samplers 2 sites	—	3 levels
NO_2		—	
TSP		1 level	—
Frequency	Each monitoring day	25% of the sites each quarter At least 1 per quarter	Each analysis-day At least twice per quarter
Scope	2 sites (of high concentration)	All sites each year	(Not applicable)

[2] Checks for PM_{10} and Pb methods have been added.

[3] These checks and audits were revised in 1986 and 1987.

Precision, Manual Methods

Precision checks are made using collocated samplers at least two sites (of high concentration).[4] One of the collocated samplers will be randomly designated as the official sampler for routine monitoring; the other shall be considered the duplicate. Results from the duplicate are to be obtained each day the designated sampler is operated unless the samplers are operated more frequently than every sixth day, in which case at least one duplicate is required each week.

Accuracy, Automated Analyzers

Automated analyzers are challenged (audited) with known pollutant concentrations at three levels (or four levels, in the case of episode analyzers), in accordance with Table II:

Table II. Automated analyzer audit concentrations (ppm)

Audit level	Concentration range	
	SO_2, NO_2, O_3	CO
1	0.03—0.08	3— 8
2	0.15—0.20	15—20
3	0.40—0.45	40—45[5]
4	0.80—0.90	80—90

Twenty-five percent of the automated analyzers of each type in the monitoring network are to be audited once each calendar quarter so as to represent a random sample for the entire network. Thus, for each quarter, the results represent a random sample from all of the analyzers. However, at least one analyzer shall be audited each quarter and all analyzers shall be audited each year. Since the audits are to be conducted with standards and equipment different from that used for calibration and spanning (the analyst should also be different), when the audit is performed within the quarter is not critical.

Accuracy, Manual Methods

For manual methods an accuracy audit is made of only a portion of the measurement system. For TSP, only the flow measurement portion is audited; for NO_2 and SO_2, only the chemical analytical portion is audited.

The flow rate audits for TSP are made at the normal operating level. Twenty-five percent of the sites shall be audited each quarter, so as to represent a random sample for the entire network. However, at least one site shall be audited each quarter and all sites shall be audited each year.

For the NO_2 and SO_2 methods, audit samples in the following ranges are used: 0.2—0.3 μg/ml; 0.5—0.6 μg/ml; 0.8—0.9 μg/ml. An audit at each concentration level shall be made on each day of analysis of routine monitoring samples, and the audits shall be made at least twice each quarter.

[4] Presently 1 to 3 collocated sites are required.

[5] These ranges are now 0.35-0.45 ppm and 35-45 ppm.

Computations

Signed Percentage Differences

The general form for computing individual signed percentage differences, d_i, whether for precision checks or for accuracy audits, is:

$$d_i = \frac{Y_i - X_i}{X_i}(100), \qquad (1)$$

where, for accuracy audits (both automated analyzers and manual methods) and for automated analyzer precision checks, Y represents the observed value and X represents the known value. For manual method precision estimates (collocated samplers), Y represents the duplicate sampler value and X represents the designated sampler value.[6]

Percentage differences instead of actual differences are used because errors in precision and errors in accuracy are generally proportional to concentration levels.

Signed percentage differences instead of absolute percentage differences are used to reveal or highlight any systematic errors that may need to be investigated and corrected to further improve the precision and accuracy of the monitoring data. Absolute percentage differences would not enable a separation of the systematic errors from the random errors.

Data Summarization

Precision and accuracy data are summarized and reported for each calendar quarter.

Precision. For each analyzer or site, the individual signed percentage differences are summarized by calculating an arithmetic average, $\bar{d}_j$, and a standard deviaion, S_j. Ninety-five percent probability limits can be calculated for each instrument or site for local network information, using the following formula:

$$\bar{d}_j \pm 1.96\ S_j. \qquad (2)$$

Although the regulations do not require such limits to be computed, they should be of particular interest and value for the local network as a supplement to their routine internal quality control. However, for reporting to EPA, a consolidated set of 95 percent probability limits,

$$\bar{D} \pm 1.96\ S_*, \qquad (3)$$

are computed for automated analyzers: where the $\bar{D}$ is the weighted average of the $\bar{d}_j$, and S_* is the pooled, weighted valued computed from the S_j.[7]

The expression for the probability limits for precision for collocated samplers is:

$$\bar{D} \pm 1.96\ S_*/\sqrt{2}. \qquad (4)$$

This $\sqrt{2}$ factor is introduced to correct for the statistical accumulation of imprecision of results from both the duplicate and the designated samplers. The probability limits are thereby put in terms of individual reported values, the same as for the other probability limits.

Accuracy. From the d_i values obtained from the accuracy audit checks at a given concentration (or flow) level, an average $\bar{D}$ and S_* are computed. For reporting to EPA, 95 percent probability limits are computed using Equation 3.

Meaning of Probability Limits

Average Value, Precision

Automated Analyzers. The $\bar{d}_j$ values for each instrument represent the average bias of results due to instrument drift. The $\bar{D}$ simply represents, for the network, the average of the $\bar{d}_j$.

[6] The present equation for collocated samplers is $d_i = \frac{Y_i - X_i}{(Y_i + X_i)/2}(100)$,

[7] Presently, EPA calculates 95% probability limits for all reported precision and accuracy check data.

Various control charts can be used for plotting the results of the precision and accuracy data. As indicated above, the results of the precision and accuracy checks, if used in a timely way, can provide a valuable supplement to normal routine internal quality control checks.

Quality Control Charts. Although the prime objective of the precision and accuracy audits is to obtain an assessment of data quality, a number of statistical control charts can be maintained to provide some long-term internal control. With control limits established on the basis of past history (at least one quarter for precision, at least one year for accuracy), future data values can be plotted to detect any significant change from past experience.

In general, the control chart limits will be similar to the computed probability limits except that the 1.96 value will be replaced by a 3. (The 1.96 corresponds to an expected 95 percent probability—the 3 corresponds to an expected 99.7 percent probability.) In the case of manual method precision, the $\sqrt{2}$ factor is not included because the points to be plotted will be the percentage differences, which include variability from the imprecision of both samplers. Also, since the intuitively expected value for $\bar{d}_i$ is zero for precision and accuracy, the centerline for the control charts should be zero. Table III summarizes the various control charts which can be plotted for the individual precision checks and accuracy audits.

Table III. Recommended control charts and limits for state and local agencies.

Pollutant Measurement Method	Control Charts	Number of Control Charts	Control Limits	Frequency of Plotting and Values to be Plotted	Variability or Bias to be Controlled
Automated methods for SO_2, NO_2, O_3, and CO	Precision-Single Instrument	One control chart for each instrument	Zero ± 3 S.	After each biweekly precision check, plot each individual d_i value	Excessive variability and drift of each instrument
	Accuracy-Single Instrument, each audit level	One control chart for each audit level	Zero ± 3 S.	After each audit check, plot each individual d_i value	Excessive bias of each instrument
Manual methods TSP SO_2 NO_2 TSP (flow rate)	Precision-Single Site	One control chart for each collocated site	Zero ± 3 S.	Each day, plot d_i for each site	Excessive lack of agreement between collocated samplers
	Accuracy-Single Site	One control chart per agency	Zero ± 3 S.	After each audit, plot each individual d_i	Excessive bias of each instrument
SO_2 (analysis) NO_2 (analysis)	Accuracy for each audit level	One for each audit level	Zero ± 3 S.	After each audit, plot each individual d_i	Excessive bias for each audit

Lesson 14A

Precision Work Session

Lesson 14A

Precision Work Session

Lesson Goal

To ensure that students can perform precision and accuracy calculations as described in Lesson 14, "Quality Assurance Requirements for SLAMS and PSD."

Lesson Objectives

At the conclusion of this lesson, each student will be able to calculate the 95% probability limits for the precision of air monitoring data collected by a reporting organization using collocated samplers.

I. Problem

Under the conditions described below, calculate the upper and lower 95% probability limits for the precision of PM_{10} monitoring data collected by the reporting organization.

Given:

Collocated PM_{10} Sampling Data for the Reporting Organization

Sampling Site 1

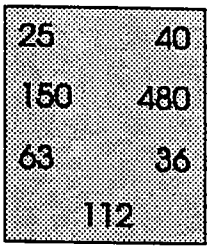
Sampling period	Duplicate sampler results (µg/std m³)	Official sampler results (µg/std m³)
1	227	236
2	268	275
3	258	256

Sampling Site 2

Sampling period	Duplicate sampler results (µg/std m³)	Official sampler results (µg/std m³)
1	245	257
2	227	240
3	164	166
4	212	221

Lesson 15

Data Validation



Data Validation

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Questions Answered in This Lesson

- What is data validation?
- What are the nine characteristics of a data-validation system?
- What are the factors that affect the selection of data-validation techniques?
- What are the levels of data validation for State Implementation Plan (SIP) air monitoring data?
- Why is it important to have data validation performed by the organization that generates the data?

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Data Validation

A systematic procedure of reviewing a body of data against a set of criteria to provide assurance of its validity before its intended use

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Terms Related to Data Validation

- Data editing
- Data screening
- Data auditing
- Data verification
- Data evaluation
- Data qualification
- Data quality assessment

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Characteristics of a Data Validation System

- An after-the-fact review
- Applied to blocks of data
- Systematically and uniformly applied
- Uses set of criteria
- Checks for internal consistency

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Characteristics of a Data Validation System (cont.)

- Checks for temporal/spatial continuity
- Checks for proper identification
- Checks for transmittal errors
- Flags/Rejects questionable data

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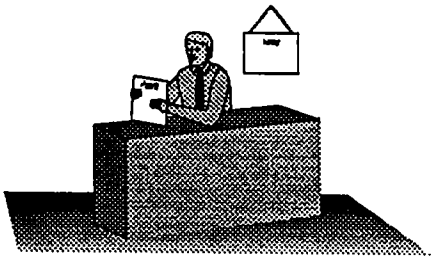
An After-the-Fact Review



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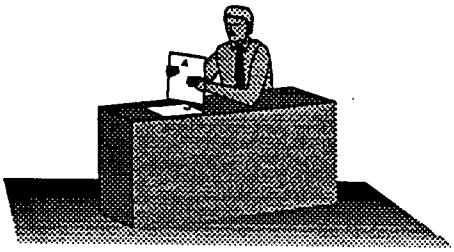
Applied to Blocks of Data



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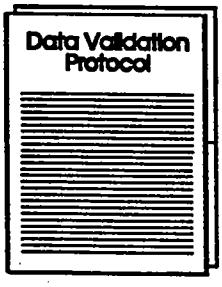
Systematically and Uniformly Applied



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Uses Set of Criteria

Criteria

- Maximum span drift
- Maximum temperature
- Concentration limit

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Checks for Internal Consistency

- Uniform sampling methodology
- Uniform monitor siting
- Uniform data reduction and reporting
- Pollutant relationships
- Pollutant /Meteorological relationships

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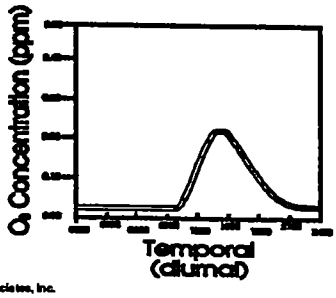
Checks for Temporal/Spatial Continuity



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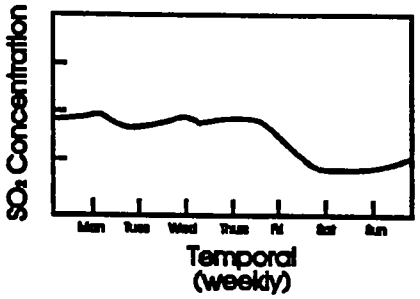
Temporal



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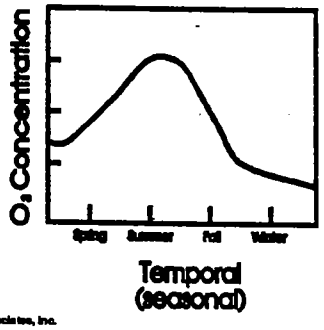
Temporal (cont.)



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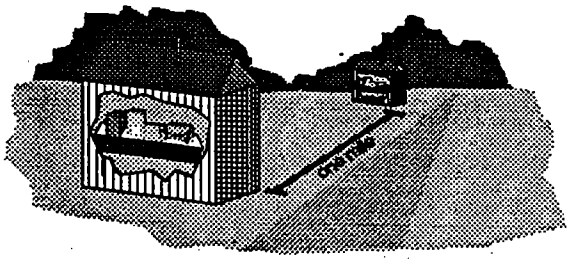
Spatial



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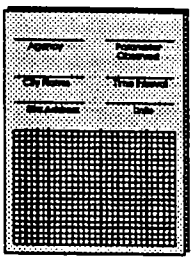

Checks for Proper Identification

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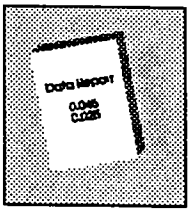
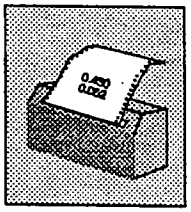
Checks for Transmittal Errors





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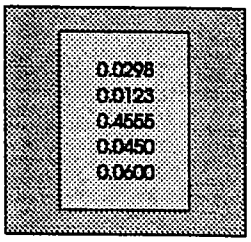
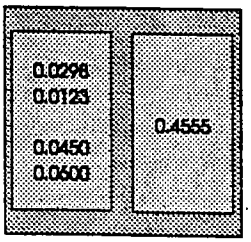
Flags/Rejects Questionable Data





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Techniques Employed

Monitoring Network Characteristics

- Nature of response output
- Data reduction methods
- Data transmittal methods
- Types and amount of ancillary data
- Computing/Plotting capability
- Intended uses of data
- Amount of data

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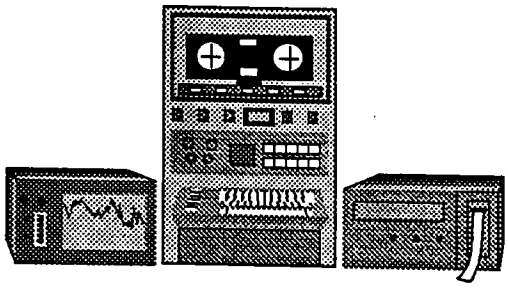
Nature of Response Output



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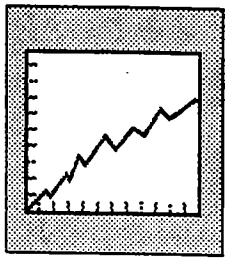
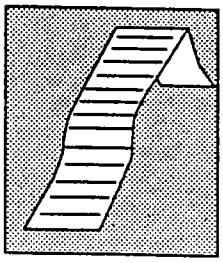
Data Reduction Methods





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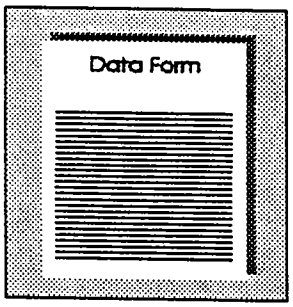
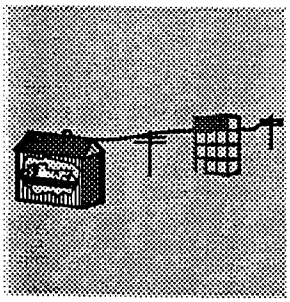
Data Transmittal Methods





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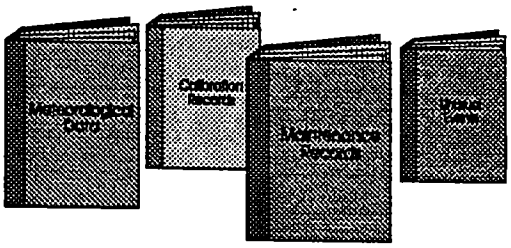
Types and Amount of Ancillary Data



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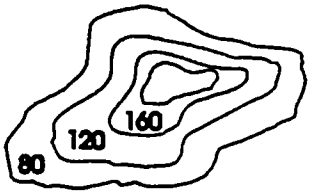
Computing/Plotting Capability



Concentration Isopleths Plot

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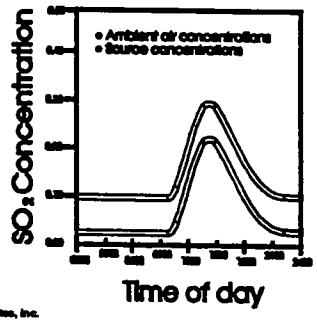
Computing/Plotting Capability (cont.)



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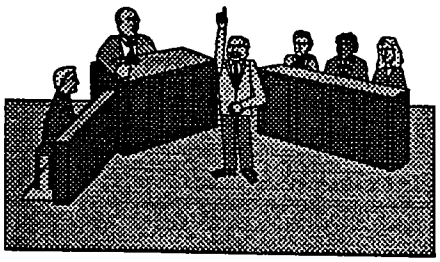
Intended Uses of Data



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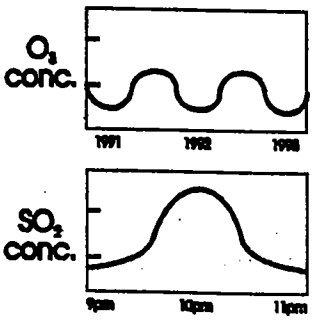
Intended Uses of Data (cont.)



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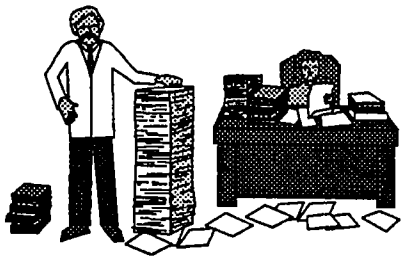
Amount of Data



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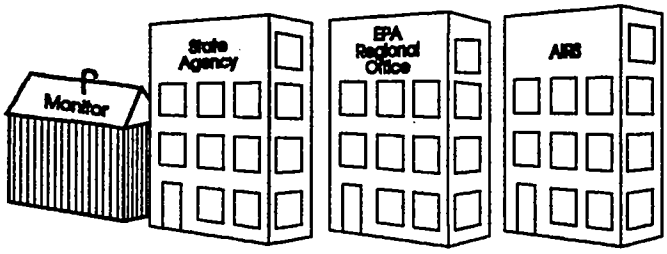
Levels of Data Validation



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Validation by Originating Organization

They have more information concerning:

- Local meteorology
- Local emissions sources
- Unusual events
- Site/instrument logbooks

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Validation by Originating Organization (cont.)

They have more information concerning:

- Personnel
- Equipment/Supplies
- Operating procedures
- Calibration materials

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Validation should be performed by someone other than the person who collected or reported the data

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QUALITY ASSURANCE AND DATA VALIDATION FOR THE REGIONAL AIR MONITORING SYSTEM OF THE ST. LOUIS REGIONAL AIR POLLUTION STUDY

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The success of model development and evaluation from a body of monitoring data depends heavily upon the quality of that data. The quality of the monitoring data in turn is dependent upon the various quality assurance (QA) activities which have been implemented for the entire system, commencing with the design, procurement, and installation of the system and ending with validation of monitoring data prior to archiving. Of the many sources of aerometric and emissions data that exist, the St. Louis Regional Air Pollution Study (RAPS) is the only known study specifically designed for model development and evaluation on an urban/rural scale.[1,2]

The prime objective of RAPS is to develop and evaluate mathematical models which will be useful in predicting air pollution concentrations from information of source emissions and meteorology. In addition to detailed emissions and meteorological data, an extensive base of high quality pollutant monitoring data is required to verify and to refine the models.

The Regional Air Monitoring System (RAMS) is the ground-based aerometric measurement system of RAPS and consists of 25 automated data acquisition sites situated in and about the St. Louis metropolitan area. Data from these 25 stations are transmitted over telephone lines to a central computer facility for processing and then sent to Research Triangle Park for archival. Details of RAMS have been described by Meyers and Reagan.[3] The complex air pollution, meteorological, and solar radiation measurements that are made at RAMS sites are shown in Table 1. Also shown are the recording intervals and the number of recording stations for each instrument.

Two main challenges exist for an effort of the magnitude of the St. Louis study:

1. To efficiently and effectively handle the large quantity of monitoring data; and

2. To obtain high quality monitoring data.

In general, data validity results from: (1) A quality assurance system aimed at acquiring acceptable data, and (2) A screening process to detect spurious values which exist in spite of the quality control process.

*On assignment from the National Oceanic and Atmospheric Administration, U.S. Department of Commerce.

Table 1. RAMS NETWORK MEASUREMENTS

		MEASUREMENT INTERVAL (min)	NUMB STAT.
AIR QUALITY:	SULFUR DIOXIDE	5	13
	TOTAL SULFUR	1	12
		5	13
	HYDROGEN SULFIDE	5	13
	OZONE	1	25
	NITRIC OXIDE	1	25
	OXIDES OF NITROGEN	1	25
	NITROGEN DIOXIDE	1	25
	CARBON MONOXIDE	5	25
	METHANE	5	25
	TOTAL HYDROCARBONS	5	25
METEOROLOGICAL:	WIND SPEED	1	25
	WIND DIRECTION	1	25
	TEMPERATURE	1	25
	TEMPERATURE GRADIENT	1	7
	PRESSURE	1	7
	DEW POINT	1	25
	AEROSOL SCATTER	1	25
SOLAR RADIATION:	PYRANOMETER	1	6
	PYRHELIOMETER	1	4
	PYRGEOMETER	1	4

QUALITY ASSURANCE SYSTEM

The following list includes the elements of a total quality assurance system for aerometric monitoring:

- Quality policy
- *Quality objectives
- *Quality organization and responsibility
- QA manual
- *QA plans
- Training
- *Procurement control
  - Ordering
  - Receiving
  - Feedback and corrective action
- *Calibration
  - Standards
  - Procedures
- Internal QC checks
- Operations
  - Sampling
  - Sample handling
  - Analysis
- Data
  - Transmission
  - Computation
  - Recording
  - * Validation
- *Preventive maintenance
- *Reliability records and analysis
- *Document control
- *Configuration control
- *Audits
  - On-site system
  - Performance
- Corrective action
- Statistical analysis
- Quality reporting
- Quality investigation
- Interlab testing
- Quality costs

Detailed definition and discussion of the elements of quality assurance for air pollution measurement systems have recently been published.[4]

The elements of particular concern to RAMS[5] fall into three general categories:

1. Procurement and management, those activitie which need to be established or accomplished early in the program;

2. Operation and maintenance, those activities which need to be performed routinely to assure continued operation of the system; and

*These particular elements, of major concern to data screening, are discussed herein.

3. Specific data quality control activities, those activities which involve the calibration and data output from the meteorological and pollutant measurement instruments and are explicitly involved in acquiring quality data.

Procurement and Management

Data Quality Objectives. A requirement of the initial contract stated that 90% valid data were to be achieved. Valid data for pollutant measurements were defined as the data obtained during periods when the daily zero and span drifts were less than 2 per cent, with an allowance for the time required to perform daily zero/span checks and periodic multi-point calibrations.

Procurement. In planning to achieve the objectives very stringent requirements were placed on the suppliers of the various instruments of the system and extensive performance tests (with numerous rejections) were conducted prior to final acceptance.

First Article Configuration Inspection (FACI). The first remote station was installed and performance tested by the contractor under EPA review. Various indicated corrections were made before proceeding with the installation of the entire network.

System Acceptance Test (SAT). After installation of the entire network, a one-month system performance demonstration was required to assure satisfactory operation with respect to obtaining data of adequate quantity and quality. The SAT was completed in December 1974.

Incentive Contract. The current contract has introduced award fee performance incentives for management, schedule, and for quality. The quality portion of the award fee provides a continual motivation for obtaining and improving data quality.

Quality Assurance Plans. An extensive QA plan has been developed by the contractor. A point of emphasis is that the QA plan (and its implementation) is dynamic --continually being revised and improved based upon experience with the system. The QA plan outlines in detail the activities of the various QA elements previously mentioned.

Organization. To implement the QA plan, one full-time employee is assigned to overall QA responsibilities reporting directly to the Program Manager. In addition, two persons are assigned for QA on a half-time basis, one for the remote monitoring stations, and the other for the central computer facility.

Operation and Maintenance

Document Control. Detailed operation and maintenance manuals have been prepared for the remote stations and for the central computer facility. These manuals are issued in a loose-leaf revisable and document-control format so that needed additions and/or revisions can be made. Also, a complete history of changes are kept so that traceability to the procedures in effect for any past period of time can be made. A document control system also exists for the computer programs.

Preventive Maintenance. Record-keeping and appropriate analysis of the equipment failure records by instrument type and mode of failure have enabled more efficient and effective scheduling of maintenance and optimum spare parts inventory with resultant improvement in instrument performance. RAMS station preventive maintenance is completed twice each week. Normally, the remote stations are unattended except for the weekly checks, for other scheduled maintenance, or for special corrective maintenance.

Central Computer Monitors. Central computer personnel, using a CRT display, periodically monitor the output from all stations to detect problems as soon as possible. To maximize the satisfactory operation of the network equipment, the assigned QA personnel review the following activities associated with preventive maintenance:

1. remote station logbook entries,

2. remote station corrective maintenance reports,

3. laboratory corrective maintenance reports, and

4. central computer operator log.

Additionally, the QA individuals are in frequent verbal communication with field and laboratory supervisors to discuss quality aspects of the operations.

Reliability Records and Analysis

Telecommunications Status Summaries. Each day, a summary of telecommunications operations is prepared to determine which stations and/or telephone lines are experiencing significant problems that might require corrective action.

Daily Analog/Status Check Summaries. Each day, the central computer prepares a summary of analog/status checks by station so that major problems can be corrected as soon as possible by available field technicians. These analog/status checks are explained in the section on data validation.

Configuration Control. Histories are kept of the station assignment of specific instruments, by serial number, so that possible future problems with specific instruments can be traced back to the stations. A logbook for each instrument is maintained for recording in a systematic manner the nature and date of any changes or modifications to the hardware design of the instruments.

Specific Data Quality Control Activities

Calibration

Calibration References for Gaseous Pollutants. NBS standard reference materials are used for calibration standards if available. Otherwise, commercial gases are procured and certified at NBS for use as standards.

Multipoint Calibrations. As a check on the linearity of instrument response, an on-site, 5-point calibration is scheduled at each station at 8-week intervals. Originally, acceptability was determined by visual evaluation of the calibration data plots; more recently, quantitative criteria are being established for linearity.

Measurement Audits. Independent measurement audits for pollutant instruments are performed by the contractor using a portable calibration unit and independent calibration sources at each station once each calendar quarter. Similar audits are performed on the same frequency for temperature, radiation, and

mass flowmeters; and independent checks are made on relative humidity, windspeed, and wind direction instruments. In addition to the internal audits performed by the contractor on his own operation, a number of external audits have been performed by EPA and other contractors[5] to check the entire measurement system.

On-Site System Audit. A thorough, on-site quality system audit of RAMS was performed for EPA by an independent contractor.[6] The results of this audit pointed out several areas of weakness for which corrective actions have been implemented.

Data Validation. As a part of the overall QA system, a number of data validation steps are implemented. Several data validation criteria and actions are built into the computer data acquisition system:

Status Checks. About 35 electrical checks are made to sense the condition of certain critical portions of the monitoring system and record an on-off status. For example, checks are made on power on/off, valve open/shut, instrument flame-out, air flow. When these checks are unacceptable, the corresponding monitoring data are automatically invalidated.

Analog Checks. Several conditions including reference voltage, permeation tube bath temperature, and calibration dilution gas flow are sensed and recorded as analog values. Acceptable limits for these checks have been determined, and, if exceeded, the corresponding affected monitoring are invalidated.

Zero/Span Checks. Each day, between 8-12 pm, each of the gaseous pollutant instruments in each station are zeroed and spanned by automatic, sequenced commands from the central computer. The results of the zero/span checks provide the basis for a two-point calibration equation, which is automatically computed by the central computer and is used for converting voltage outputs to pollutant concentrations for the following calendar day's data. In addition, the instrument drift at zero and span conditions between successive daily checks are computed by the central computer and used as a basis for validating the previous day's monitoring data. Originally, zero and span drifts were considered as acceptable if less than 2 per cent, but the span drift criterion has recently been increased to 5 per cent, a more realistic level. If the criteria are not met, the minute data for the previous day are flagged. Hourly averages are computed during routine data processing only with data which have not been flagged as invalid.

DATA SCREENING IN RAMS

The tests which are used to screen RAMS data are summarized in Table 2. Specific tests and associated data base flags are listed. The types of screens that have been employed or tested will be detailed, the mechanisms for flagging will be reviewed, and then the implementation of screening within RAMS will be discussed.

Table 2. SCREENING CATEGORIES AND ASSOCIATED FLAGS FOR RAMS DATA

Category	Flag
I. Modus Operandi	
No instrument	10^{37}
Missing measurement	10^{37}
Status	Value + 10^{-35}
Calibration	10^{35}
II. Continuity and Relational	
A. Intrastation	
Gaseous analyzer drift	- Value
Gross limits	10^{34}
Aggregate frequency distributions	Being implemented
Relationships	Value + 10^{32}
Temporal continuity	
Constant output	Value + 10^{31}
Successive difference	Being implemented
B. Interstation	
Meteorological network uniformity	Value + 10^{16}
Statistical outliers	
Diamm Ratio	Value + 10^{-30}
III. A Posteriori	
Review of station log	Invalidate 10^{38}
Unusual events or conditions	Validate + Comm flag
Visual inspection of data	

For descriptive purposes, the tests are divided into three categories. The first category, "Modus Operandi," contains checks which document the networ instrument configuration and operating mode of the recording system. Included are checks for station instrumentation, missing data, system analog and status sense bits, and instrument calibration mode. These checks, which have been described above, are part of the quality control program incorporated in the data acquisition system and central facility dat processing, and are an important data management function used to document system performance.

The second category, "Continuity and Relational contains temporal and spatial continuity checks and relational checks between parameters which are based on physical and instrumental considerations or on statistical patterns of the data. A natural subdivision can be made between intrastation checks, those checks which apply only to data from one stati and interstation checks, which test the measured parameters for uniformity across the RAMS network.

Intrastation checks include tests for gaseous analyzer drift, gross limits, aggregate frequency distributions, relationships, and temporal continuit The drift calculations, which are part of the qualit control program, have been discussed above.

Gross limits, which are used to screen impossib values, are based on the ranges of the recording instruments. These, together with the parametric relationships which check for internal consistency between values, are listed in Table 3. Setting limi for relationship tests requires a working knowledge noise levels of the individual instruments. The relationships used are based on meteorology, atmospheric chemistry, or on the principle of chemical ma balance. For example, at a station for any given minute, TS cannot be less than SO_2 + H_2S with allowances for noise limits of the instruments.

Table 3. GROSS LIMITS AND RELATIONAL CHECKS

PARAMETER	INSTRUMENTAL OR NATURAL LIMITS		INTERPARAMETER CONDITION
	LOWER	UPPER	
Ozone	0 ppm	5 ppm	$NO^{*}O_3 \leq 0.04$
Nitric Oxide	0 ppm	5 ppm	$NO + NO_2 \pm$ Noise (NO)
Oxides of Nitrogen	0 ppm	5 ppm	$NO + NO_2 \pm$ Noise (NO_2)
Carbon Monoxide	0 ppm	50 ppm	
Methane	0 ppm	50 ppm	CH_4 - THC $\pm$ Noise (CH_4)
Total Hydrocarbons	0 ppm	50 ppm	CH_4 - THC $\pm$ Noise (THC)
Sulfur Dioxide	0 ppm	1 ppm	SO_2 - TS $\pm$ Noise (SO_2)
Total Sulfur	0 ppm	1 ppm	SO_2 - TS $\pm$ Noise (TS)
Hydrogen Sulfide	0 ppm	1 ppm	H_2S - TS $\pm$ Noise (H_2S)
Aerosol Scatter	$0.000001m^{-1}$	$0.00099m^{-1}$	
Wind Speed	0 m/s	22.2 m/s	
Wind Direction	0°	360°	
Temperature	-20°C	45°C	
Dew Point	-30°C	45°C	DP - 0.5 $\leq$ T
Temperature Gradient	- 5°C	5°C	
Barometric Pressure	950 mb	1050 mb	
Pyranometers	- 0.50	2.50 Langleys/min	
Pyrgeometers	0.30	0.75 Langleys/min	
Pyrheliometers	-0.50	2.50 Langleys/min	

A refinement of the gross limit checks can be made using aggregate frequency distributions. With a knowledge of the underlying distribution, statistical limits can be found which have narrower bounds than the gross limits and which represent measurement levels that are rarely exceeded. A method for fitting a parametric probability model to the underlying distribution has been developed by Dr. Wayne Ott of EPA's Office of Research and Development.[7]. B.E. Suta and G.V. Lucha[8] have extended Dr. Ott's program to estimate parameters, perform goodness-of-fit tests, and calculate quality control limits for the normal distribution, 2- and 3-parameter lognormal distribution, the gamma distribution, and the Weibull distribution. These programs have been implemented on the OSI computer in Washington and tested on water quality data from STORET. This technique is being studied for possible use in RAMS as a test for potential recording irregularities as well as a refinement of the gross limit check currently employed.

Under intrastation checks are specific tests which examine the temporal continuity of the data as output from each sensor. It is useful to consider, in general, the types of atypical or erratic responses that can occur from sensors and data acquisition systems. Figure 1 illustrates graphically examples of such behavior, all of which have occurred to some extent within RAMS. Physical causes for these reactions include sudden discrete changes in component operating characterisitcs, component failure, noise, telecommunication errors and outages, and errors in software associated with the data acquisition system or data processing. For example, it was recognized early in the RAMS program that a constant voltage output from a sensor indicated mechanical or electrical failures in the sensor instrumentation. One of the first screens that was implemented was to check for 10 minutes of constant output from each sensor. Barometric pressure is not among the parameters tested since it can remain constant (to the number of digits recorded) for periods much longer than 10 minutes. The test was modified for other parameters which reach a low constant background level during night-time hours.

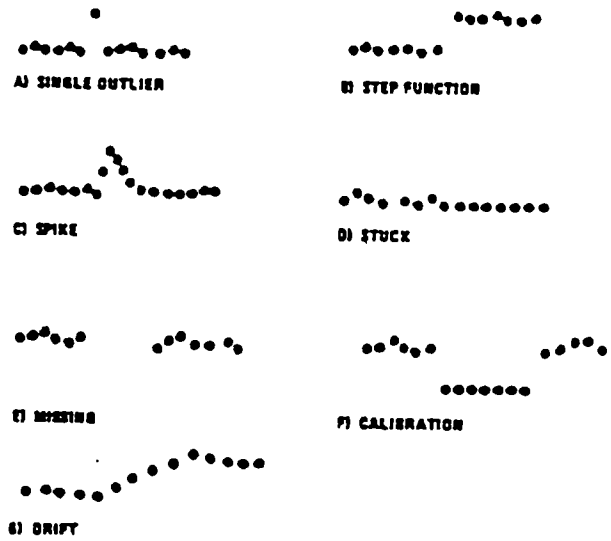

Figure 1. Irregular instrument response.

A technique which can detect any sudden jump in the response of an instrument, whether it is from an individual outlier, step function or spike, is the comparison of minute successive differences with predetermined control limits. These limits are determined for each parameter from the distribution of successive differences for that parameter. These differences will be approximately normally distributed with mean zero (and computed variance) when taken over a sufficiently long time series of measurements.

Exploratory application of successive differences, using 4 standard deviation limits which will flag 6 values in 100,000 if the differences are truly normally distributed, indicate that there are abnormal occurrences of "jumps" within certain parameters. Successive difference screening will be implemented after further testing to examine the sensitivity of successive difference distributions to varying computational time-periods and to station location.

The type of "jump" can easily be identified. A single outlier will have a large successive difference followed by another about the same magnitude but of opposite sign. A step function will not have a return, and a spike will have a succession of large successive differences of one sign followed by those of opposite sign.

The interstation or network uniformity screening tests that have been implemented in RAMS will now be described. Meteorological network tests are performed on hourly average data and are based on the principle that meteorological parameters should show limited differences between stations under certain definable conditions typically found in winds of at least moderate speeds (>4 m/sec). Each station value is compared with the network mean. The network mean is defined as the average value for a given parameter from all stations having reported valid data. (If more than 50% are missing, a network mean is not

Figure 3. Generalized data flow for environmental measurement systems.

Data screening should take place as near to data acquisition as possible either in data processing which is traditionally concerned with laboratory analysis, conversion to engineering units, transcribing intermediate results, etc., or in a separate module, as illustrated, designed specifically for the screening process. Screening data soon after data acquisition permits system feedback in the form of corrective maintenance, changes to control processes, and even to changes in system design. This feedback is essential to minimize the amount of lost or marginally acceptable data.

The RAMS screening tests, which have been developed at Research Triangle Park (RTP), are now part of the data processing carried out at the RAPS central facility in St. Louis. Slow computation speeds of the St. Louis PDP 11/40 computer required restricting the intrastation screening tests to hourly average data. RAMS data is still passed through the RTP screening module before archiving.

SUMMARY

The experiences gained in RAMS and applicable to other monitoring systems are:

1. Data validity is a function of quality assurance and data screening.

2. A QA plan and data screening rules should be established initially and maintained throughout the program.

3. The QA plan and screening rules are dynamic, being improved as additional knowledge and experience is gained.

4. Applied during data acquisition or shortly thereafter, quality control and screening checks constitute an important feedback mechanism, indicating a requirement for corrective action.

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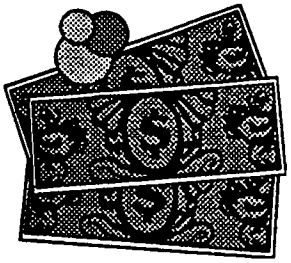
Lesson 16

Quality Costs

Quality Costs



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Questions Answered in This Lesson

- What are the three types of cost that compose the total cost per measurement result of an air-quality measurement system?
- What is the relationship between unacceptable data cost and quality assurance cost?
- What is the purpose of a quality-cost system?

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Questions Answered in This Lesson (cont.)

- What are the three cost categories of a quality-cost system?
- What are two groups of activities that are related to each of the three cost categories?
- What is the procedure for establishing a quality-cost system?

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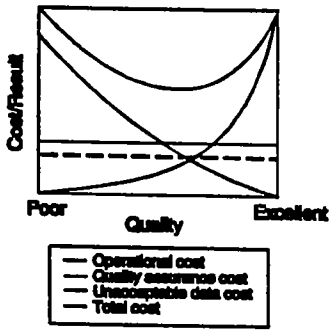
Quality Pays



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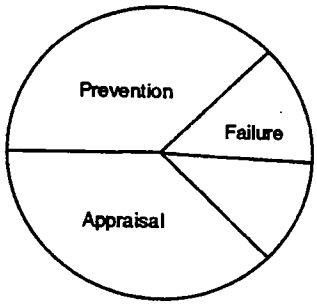
Quality-Related Costs



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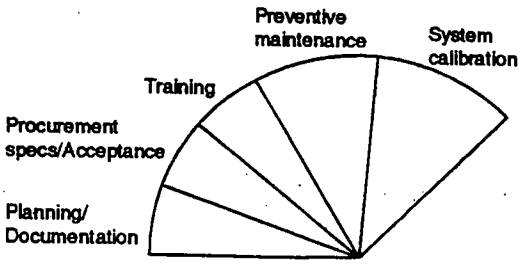
Prevention Cost Groups



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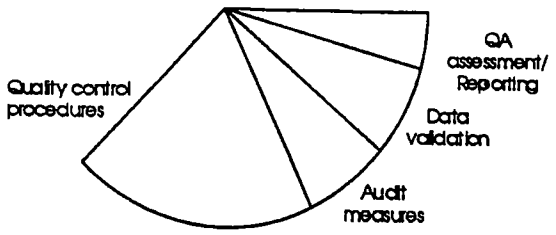
Appraisal Cost Groups



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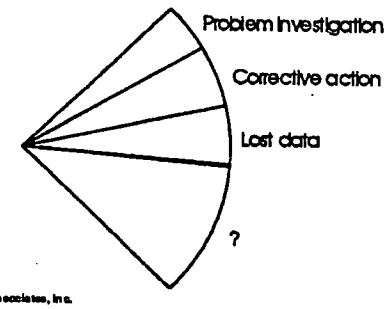
Failure Cost Groups



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Accumulation of Costs

- Lost data costs
- Other costs

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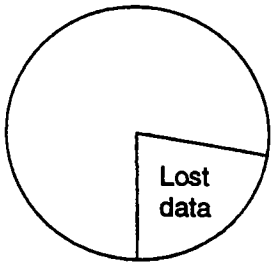

$$F_d = f \times B$$

Where:

F_d = lost data cost

f = % lost data

B = part of network budget associated with lost data

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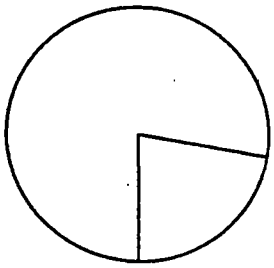
Prorate Personnel Salaries



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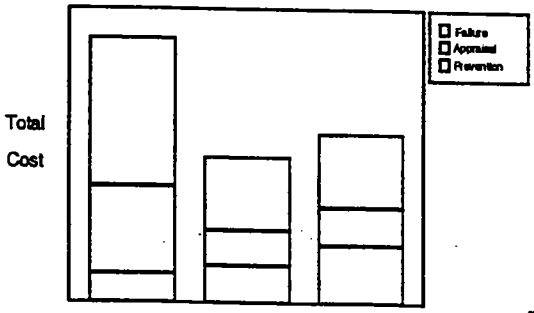
Cost Effectiveness



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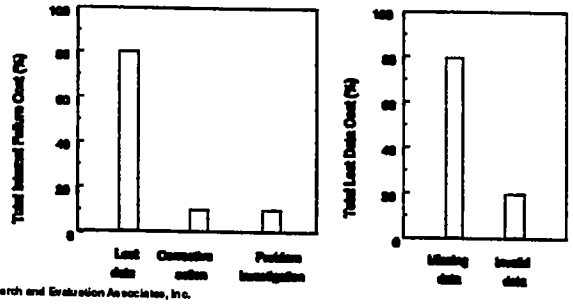
Pareto Analysis of Quality Cost Data



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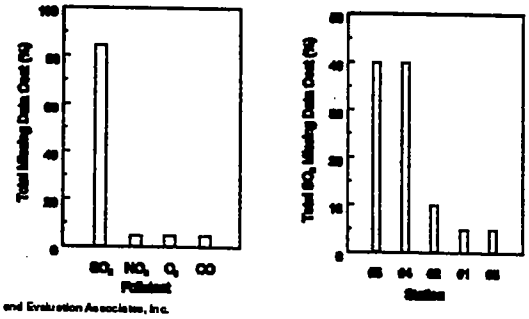
Pareto Analysis of Quality Cost Data (cont.)



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Quality Cost Reporting

- Data obtained from source documents
- Reports understandable at a glance
  - Data summarized
  - Graphs preferred

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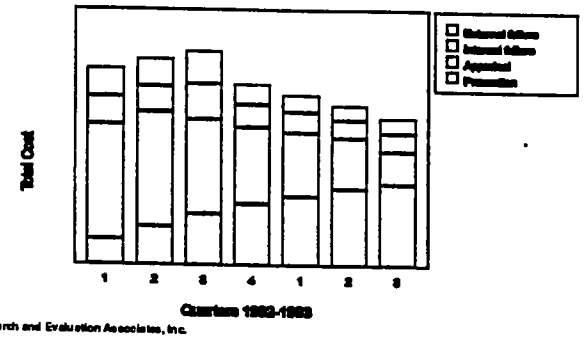
Quality Cost Trend Chart



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GUIDELINES FOR IMPLEMENTING A QUALITY COST SYSTEM FOR ENVIRONMENTAL MONITORING PROGRAMS

Presented at 73rd APCA Annual Meeting
and Exhibition in Montreal, Quebec,
Canada, June 1980

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GUIDELINES FOR IMPLEMENTING A QUALITY COST SYSTEM FOR ENVIRONMENTAL MONITORING PROGRAMS

Introduction

Program managers with Governmental agencies and industrial organizations involved in environmental measurement programs are concerned with overall program cost-effectiveness including total cost, data quality and timeliness. There are several costing techniques designed to aid the manager in monitoring and controlling program costs. One particular technique specifically applicable to the operational phase of a program is a quality cost system.

The objective of a quality cost system for an environmental monitoring program is to minimize the cost of those operational activities directed toward controlling data quality while maintaining an acceptable level of data quality. The basic concept of the quality cost system is to minimize total quality costs through proper allocation of planned expenditures for the prevention and appraisal efforts in order to control the unplanned correction costs. That is, the system is predicated on the idea that prevention is cheaper than correction.

There is no pre-set formula for determining the optimum mode of operation. Rather, the cost effectiveness of quality costs is optimized through an iterative process requiring a continuing analysis and evaluation effort. Maximum benefits are realized when the system is applied to a specific measurement method in a stable long term monitoring program. For example, a monitoring program with a fixed number of monitoring sites, scheduled to operate for a year or more, would be a desirable candidate for a quality cost system.

Quality costs for environmental monitoring systems have been treated by Rhodes and Hochheiser[1]. The purpose of this paper is to present guidelines for the implementation of a quality cost system. The contents of this paper are based on work performed by the Research Triangle Institute under contract to the U.S. Environmental Protection Agency[2].

Structuring of Quality Costs

The first step in developing a quality cost system is identifying the cost of quality-related activities, including all operational activities that affect data quality, and dividing them into the major cost categories.

Costs are divided into category, group, and activity. Category, the most general classification, refers to the standard cost subdivisions of prevention, appraisal, and failure. The category subdivision of costs provides the basic format of the quality cost system. Activity is the most specific classification and refers to the discrete operations for which costs should be determined. Similar types of activities are summarized in groups for purposes of discussion and reporting.

Cost Categories

The quality cost system structure provides a means for identification of quality-related activities and for organization of these activities into prevention, appraisal, and failure cost categories. These categories are defined as follows:

- Prevention Costs—Costs associated with planned activities whose purpose is to ensure the collection of data of acceptable quality and to prevent the generation of data of unacceptable quality.
- Appraisal Costs—Costs associated with measurement and evaluation of data quality. This includes the measurement and evaluation of materials, equipment, and processes used to obtain quality data.
- Failure Costs—Costs incurred directly by the monitoring agency or organization producing the failure (unacceptable data).

Cost Groups

Quality cost groups provide a means for subdividing the costs within each category into a small number of subcategories which eliminates the need for reporting quality costs on a specific activity basis. Although the groups listed below are common to all environmental measurement methods, the specific activities included in each group may differ between methods.

Groups within prevention costs. Prevention costs are subdivided into five groups:

- Planning and Documentation—Planning and documentation of procedures for all phases of the measurement process that may have an effect on data quality.
- Procurement Specification and Acceptance—Testing of equipment parts, materials, and services necessary for system operation. This includes the initial on-site review and performance test, if any.
- Training—Preparing or attending formal training programs, evaluation of training status of personnel, and informed on-the-job training.
- Preventive Maintenance—Equipment cleaning, lubrication, and parts replacement performed to prevent (rather than correct) failures.
- System Calibration—Calibration of the monitoring system, the frequency of which could be adjusted to improve the accuracy of the data being generated. This includes initial calibration and routine calibration checks and a protocol for tracing the calibration standards to primary standards.

Groups within appraisal costs. Appraisal costs are subdivided into four groups:

- Quality Control (QC) Measures—QC-related checks to evaluate measurement equipment performance and procedures.
- Audit Measures—Audit of measurement system performance by persons outside the normal operating personnel.
- Data Validation—Tests performed on processed data to assess its correctness.
- Quality Assurance (QA) Assessment and Reporting—Review, assessment, and reporting of QA activities.

Groups within failure costs. Under most quality cost systems, the failure category is subdivided into internal and external failure costs. Internal failure costs are those costs incurred directly by the agency or organization producing the failure.

Internal failure costs are subdivided into three groups:

- Problem Investigation—Efforts to determine the cause of poor data quality.
- Corrective Action—Cost of efforts to correct the cause of poor data quality, implementing solutions, and measures to prevent problem reoccurrence.
- Lost Data—The cost of efforts expended for data which was either invalidated or not captured (unacquired and/or unacceptable data). This cost is usually prorated from the total operational budget of the monitoring organization for the percentage of data lost.

External failure costs are associated with the use of poor quality data external to the monitoring organization or agency collecting the data. In air monitoring work these costs are significant but are difficult to systematically quantize. Therefore, this paper will only address failure costs internal to the monitoring agency. However, external failure costs are important and should be considered when making decisions on additional efforts necessary for increasing data quality or for the allocation of funds for resampling and/or reanalysis.

Examples of failure cost groups are:

- Enforcement actions—Cost of attempted enforcement actions lost due to questionable monitoring data.
- Industry—Expenditures by industry as a result of inappropriate or inadequate standards established with questionable data.
- Historical Data—Loss of data base used to determine trends and effectiveness of control measures.

Cost Activities

Examples of specific quality-related activities which affect data quality are presented in Table I. These activities are provided as a guide for implementation of a quality cost system for an air quality program utilizing continuous monitors. Uniformity across agencies and organizations in the selection of activities is desirable and encouraged, however, there are variations which may exist, particularly between monitoring agencies and industrial/research projects.

Agencies should make an effort to maintain uniformity regarding the placement of activities in the appropriate cost group and cost category. This will provide a basis for future "between agency" comparison and evaluation of quality cost systems.

Development and Implementation of the Quality Cost System

Guidelines are presented in this section for the development and implementation of a quality cost system. These cover planning the system, selecting applicable cost activities, identifying sources of quality cost data, tabulating, and reporting the cost data.

Planning

Implementation of a quality cost system need not be expensive and time consuming. It can be kept simple if existing data sources are used wherever possible. The importance of planning cannot be overemphasized. For example, implementation of the quality cost system will require close cooperation between the quality cost system manager and other managers or supervisors. Supervisors should be thoroughly briefed on quality cost system concepts, benefits, and goals.

System planning should include the following activities:

- Determining scope of the initial quality cost program.
- Setting objectives for the quality cost program.
- Evaluating existing cost data.
- Determining sources to be utilized for the cost data.
- Deciding on the report formats, distribution, and schedule.

To gain experience with quality cost system techniques, an initial pilot program could be developed for a single measurement method or project within the agency. The unit selected should be representative, i.e., exhibit expenditure for each cost category: prevention, appraisal, and failure. Once a working system for the initial effort has been established, a full-scale quality cost system can then be implemented.

Activity Selection

The first step for a given agency to implement a quality cost system is to prepare a detailed list of the quality-related activities most representative of the agencies monitoring operation and to assign these activities to the appropriate cost groups and cost categories. Worksheets and cost summaries for collecting and tabulating cost data for specific measurement methods will then need to be assigned and methods developed to accumulate the costs as easily as possible. Ultimately and most important is the analysis of the accumulated costs, discussed in the next section.

The general definitions of the cost groups and cost categories, presented in the previous section, are applicable to any measurement system. Specific activities contributing to these cost groups and categories, however, may vary significantly between agencies, depending on the scope of the cost system, magnitude of the monitoring network, parameters measured, and duration of the monitoring operation. The activities listed in Table I are provided as a guide only, and they are not considered to be inclusive of all quality-related activities. An agency may elect to add or delete certain activities from this list. It is important, however, for an agency to maintain uniformity regarding the cost groups and categories the activities are listed under. As indicated previously, this will provide a basis for future cost system comparison and evaluation.

Quality Cost Data Sources

Most accounting records do not contain cost data detailed enough to be directly useful to the operating quality cost system. Some further calculation is usually necessary to determine actual costs which may be entered on the worksheets. The cost of a given activity is usually estimated by prorating the person's charge rate by the percentage of time spent on that activity. A slightly rougher estimate can be made by using average charge rates for each position instead of the actual rates.

Failure costs are more difficult to quantize than either prevention or appraisal costs. The internal failure cost of lost data (unacquired and/or unacceptable data), for example, must be estimated from the total budget.

Cost Accumulation and Tabulation

Cost collection and tabulation methods should be kept simple and conducted within the framework of the agency's normal reporting format whenever possible. During initial system development, a manual approach will allow needed flexibility, whereas, automatic quality cost data tabulation would be complicated, since many of the quality-related activities are not typical in existing accounting systems. Automatic tabulation of costs may be practical after the basic quality cost system has been developed.

Also, an effective cost system does not require precise cost accounting. Reasonable cost estimates are adequate when actual cost records are not available.

Worksheets and summaries used to collect and tabulate the cost data should be designed to represent expenditures by activity.

Quality Cost Worksheets

Worksheets for collecting and tabulating quality cost data should be prepared for each specific measurement method. The worksheet should be designed to allow cost tabulation for each quality-related activity performed and to accomodate more than one personnel level per activity. In addition, activities should be organized into appropriate cost groups and cost categories so that when total costs are computed, they can be transferred directly to cost summaries later.

Quality Cost Analysis Techniques

Techniques for analyzing and evaluating cost data range from simple charts comparing the major cost categories to sophisticated mathematical models of the total program. Common techniques include trend analysis and Pareto analysis.

Trend analysis. Trend analysis compares present to past quality expenditures by category. A history of quality cost data, typically a minimum of 1-year, is required for trend evaluation. (An example is given in Figure 1 of the next section).

Cost categories are plotted within the time frame of the reporting period (usually quarterly). Costs are plotted either as total dollars (if the scope of the monitoring program is relatively constant) or as "normalized" dollars/data unit (if the scope may change). Groups and activities within the cost categories contributing the highest cost proportions are plotted separately.

Pareto analysis. Pareto analysis identifies the areas with greatest potential for quality improvement by:

- Listing factors and/or cost segments contributing to a problem area.
- Ranking factors according to magnitude of their contribution.
- Directing corrective action toward the largest contributor.

Pareto techniques may be used to analyze prevention, appraisal, or failure costs. They are most logically applied to the failure cost category, since the relative costs associated with activities in the failure category indicate the major source of data quality problems. Typically, relatively few contributors will account for most of the failure costs.[3,4] (An example is given in Figure 3 of the next section.)

Quality Cost Reports

Quality cost reports prepared and distributed at regular intervals should be brief and factual, consisting primarily of a summary discussion, a tabulated data summary, and a graphic representation of cost category relationships, trends, and data analysis. The summary discussion should emphasize new or continuing problem areas and progress achieved during the reporting period.

Written reports should be directed toward specific levels of management. Managers and supervisors receiving reports should be thoroughly briefed on the concepts, purpose, and potential benefits of a quality cost system, i.e., identification of quality-related problems, potential input into problem solution, and quality cost budgeting.

Quality Cost System Example

A hypothetical case history of a quality cost system is presented in this section. In this example, a cost system is developed for an agency operating sixteen sulfur dioxide monitoring stations. The stations are located within a 50-mile radius and each is equipped with a continuous sulfur dioxide monitor. The monitoring network has been in operation for 2 years.

The QA Coordinator is given the responsibility for implementing the quality cost system. The QA Coordinator plans the implementation of the pilot cost system. Planning for the system includes selecting cost activities, determining cost methods, and establishing procedures for maintaining the system.

To establish an historical basis quality costs are estimated for the past year. This allows for trend observation over an adequate period of time. These costs are shown (see Figure 1) and discussed in the following paragraphs.

Unacceptable data costs are a major cost group in the failure category. In order to establish the value of "lost data", the overall monitoring budget is determined from contracts, accounting documents, and other source documents. Table II summarizes total monitoring costs for the criteria pollutants and the sulfur dioxide costs are used in this example quality cost system. The cost data includes the maximum possible number of data units and cost per data unit.[5]

Quality-related costs are estimated for each quarter over the preceding year. The estimated costs are subject to the following considerations:

- Estimates of time spent by an operator performing a specific activity takes into account the capability of the operator to perform several activities simultaneously. For example, an operator performing daily analyzer zero/span will have time to simultaneously perform other duties while the analyzers stabilize to the zero/span inputs.
- The activities are performed by three personnel types: manager, supervisor, and operator. The cost per hour for each level is consistent with "Cost of Monitoring Air Quality in the United States."[5]

Analysis and evaluation of the collected cost data will determine several facts about the example agency's quality effort. The cost data should reflect the present status of the quality program, where major problem areas exist, and what immediate goals should be established.

A graph of the expenditures for each cost category is shown in Figure 2. Throughout the preceding year prevention costs were relatively small, appraisal costs were moderate, and failure costs were significant. Also, failure costs showed an increasing trend throughout the year.

A Pareto distribution of the failure costs (Figure 3) shows that the major cost contributor is "lost" data. The "lost" data cost represents over 80 percent of the total failure costs. Although the "lost" data cost represents less than 20 percent of the total data possible, the cost of this loss is significant.

An investigation determines the major cause of the problem to be a shortage of station operators. The workload of the one fulltime operator does not allow adequate time for an effective preventive maintenance program. The lack of proper preventive maintenance increases the frequency of analyzer/equipment failure resulting in an additional workload for the station operator, i.e., equipment repair.

The quality manager prepares a quality cost report covering the initial study results. The report presents several recommendations, including:

- Hire and train an additional operator.
- Increase prevention efforts for the monitoring operation.
- Reduce failure costs 50% by the end of the next reporting period.

During the following quarter, an additional operator was hired and trained. Preventive maintenance procedures were reviewed and modified as required. At the end of this reporting period, quality costs were collected, analyzed, and evaluated. The quality cost report covering this reporting period shows that failure costs were reduced 37%, prevention costs were increased 81% and appraisal costs increased 32%. A net decrease in total quality cost, amounting to $2,584 (11%) was experienced for the quarter as seen in Figure 1 when comparing the first quarter of 1979 with the fourth quarter of 1978.

The changes in category expenditures (Figure 2) reflect specific corrective measures initiated during the reporting period. These measures included hiring and training an additional operator and increasing the preventive maintenance effort.

Although the unacceptable data costs were decreased significantly, these costs are still excessive and a preliminary analysis of the last sulfur dioxide data indicates that additional effort in preventive maintenance is necessary to further reduce the networks operating costs.

TOTAL QUALITY COST SUMMARY
(Combined network costs, 1978-79)

COST GROUP	2nd Quarter	3rd Quarter	4th Quarter	1st Quarter
PREVENTION				
Planning & documentation	—	—	—	179
Procurement	—	—	—	179
Training	—	—	—	459
Preventive maintenance	588	559	587	1,046
System calibration and operation	1,254	1,317	1,386	1,713
TOTAL PREVENTION COSTS	1,842	1,876	1,973	3,576
APPRAISAL				
QC measures	768	806	742	1,631
Audits	1,308	1,508	1,470	1,913
Data validation	1,468	1,668	1,868	1,887
QA assessment & reporting	1,748	1,839	1,686	2,179
TOTAL APPRAISAL COSTS	5,292	5,821	5,766	7,610
FAILURE				
Problem investigation	1,579	1,886	1,760	704
Corrective action	1,361	1,334	1,365	546
Lost data (unacquired data)	12,430	13,893	13,162	9,506
TOTAL FAILURE COSTS	15,370	17,113	16,287	10,256
TOTAL QUALITY COSTS	22,504	24,810	24,026	21,442

MEASUREMENT BASES
Total program cost per quarter 48,304
Total data units per quarter 33,792

Figure 1. Total quality cost summary.

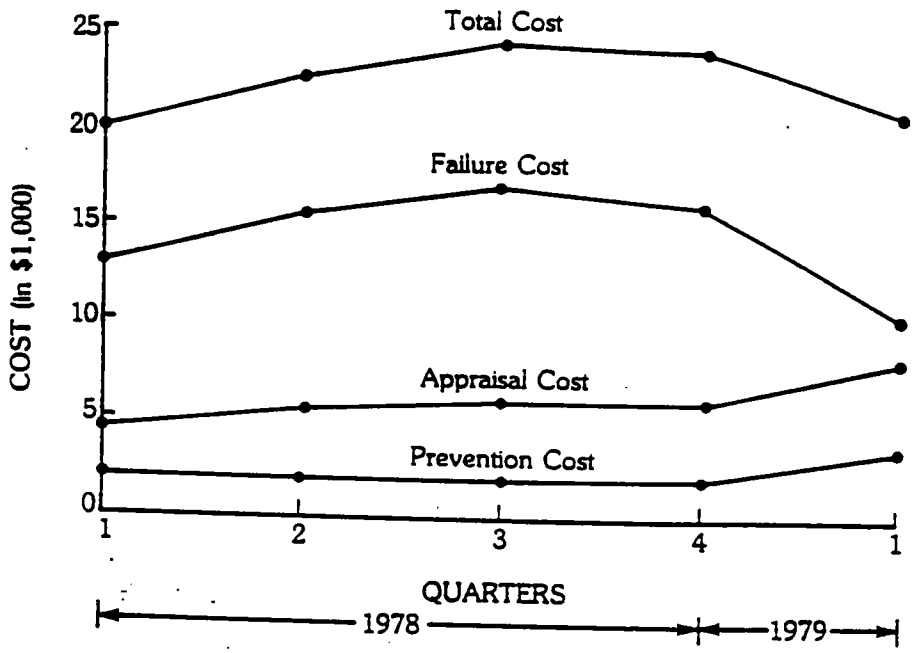

Figure 2. Quality cost trends.

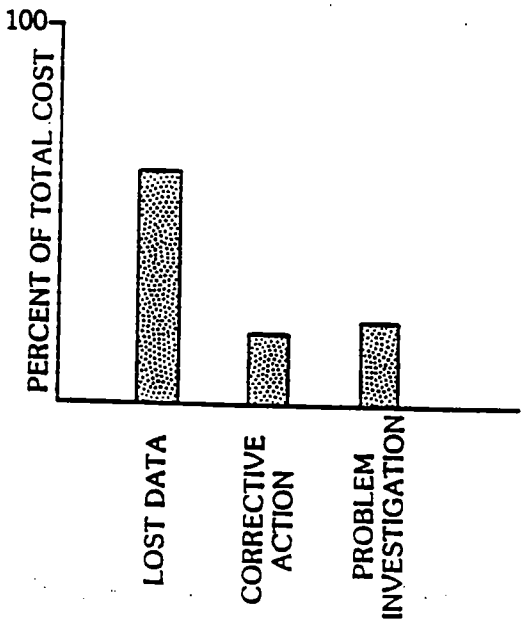

Figure 3. Failure cost distribution.

TABLE 1. EXAMPLE COST ACTIVITIES GUIDE FOR AMBIENT AIR MEASUREMENT METHODS

APPRAISAL COST CATEGORY	ACTIVITY	PREVENTION COST CATEGORY	ACTIVITY	FAILURE COST CATEGORY	ACTIVITY
QC Measures	Intercomparison of calibration teams. Operation of collocated samplers. Special activities performed by station operator strictly for obtaining data to evaluate accuracy and precision.	Planning and Documentation	Planning and preparation of: • Audit schedules and procedures • Operating standards • Apparatus configuration • Control procedures • Special tests • Operating procedures • Preventive maintenance procedures	Problem Investigation	Special tests and information collection to detect source and characteristics of problem Data and procedure review to identify problem area
Audits	Independent performance and system audits.	Procurement	Specification and acceptance testing of: • Calibration gases/devices • Equipment	Corrective Action	Cost of correcting data Cost of changing established procedures to prevent reoccurrence of problems
Data Validation	• Statistical evaluation of data • Review of data handling procedures • Comparison of data with historical data and data from nearby stations Review and evaluation of: • Control tests • Audit schemes • Audit results • Special interference tests	Training	Operator training/proficiency evaluation. Certification of calibration personnel and equipment.	Lost Data (unacquired and/or unacceptable data)	Estimated value of data which was lost or invalidated
QA Assessment and Reporting	Assessment of overall data quality. Generation of data quality reports. Maintenance of quality cost system records.	Preventive Maintenance	Maintenance of records, logs operation, calibration, and maintenance). Performance of preventive maintenance procedures.		
		System Calibration	Routine multipoint calibration of analyzers. Zero/span checks. Special checks on calibration system (voltage, temperature, variation, etc.)		

TABLE II. Total monitoring cost (dollars).

Pollutant	Annualized Total Cost Per Station	Maximum Data Units Per Station*	Cost Per Data Unit
CO	9,969	8448	1.18
SO_2	12,076	8448	1.43
O_3	8,713	8448	1.03
TSP	1,535	61	25.10
NO_2	8,757	8448	1.04
THC	9,231	8448	1.09
TOTAL FOR SO_2 = $12,076 × 16 = $193,216			

*Maximum data units for continuous analyzers based on total possible hourly averages per year.

Summary

The first step in implementing a quality cost system for an environmental monitoring program is to categorize quality-related activities into prevention, appraisal, and correction categories. An example listing for measurement methods involving continuous gaseous analyzers is given in this paper. Major items to be considered when implementing a system have been discussed along with an example quality cost system. Emphasis should be placed by management on preventive activities to decrease total cost of quality related activities.

References

1. Rhodes, Raymond C. and Seymour Hochheiser. "Quality Costs for Environmental Monitoring Systems," American Society for Quality Control, Technical Conference, 1977, p. 328.
2. Strong, R. B., J. H. White and F. Smith "Guidelines for the Development and Implementation of a Quality Cost System for Air Pollution Measurement Programs", Research Triangle Institute, Research Triangle Park, North Carolina, 1980, EPA Contract No. 68-02-2722.
3. American Society for Quality Control, Quality Costs Technical Committee. "Guide for Reducing Quality Costs," Milwaukee, Wisconsin, 1977.
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5. PEDCo Environmental, Inc. "Cost of Monitoring Air Quality in the United States," December 1978.

Lesson 17

Quality Assurance Guidance for PM2.5 Ambient Air Monitoring- Part I

[redacted]

Office of Air Quality Planning and Standards
U.S. Environmental Protection Agency

APTI 470
Quality Assurance for Air Pollution
Measurement Systems

Introduction

- Scope
- Applicability
- Conventions
- Reference and Equivalent Methods

470-17-1

Scope

- Formal specifications for $PM_{2.5}$ samplers in 40 CFR Part 50, Appendix L
- Clarifications and supplemental QA guidance in Section 2.12 of QA Handbook, Vol. II, Part II, as well as Vol. II, Part I
- Emphasis of Section 2.12 on operational aspects
- QA guidance provided to achieve data quality objectives (DQOs)
- DQO process is driving force for QA system
- Goals for acceptable measurement uncertainty

470-17-2

Measurement Uncertainty Goals

- < 10% coefficient of variation (CV) for total precision
- ± 10% of the audit value for total bias
- Accuracy goals for the sampler's flow rate

470-17-3

Accuracy Goals for Sampler's Flow Rate

- ± 2% of the audit device during multipoint verification/calibration
- ± 4% during one-point verification checks and audits
- ± 5% of the sampler's design flow rate of 16.67 L/min

470-17-4

Applicability of Section 2.12

- State and Local Air Monitoring Stations (SLAMS)
- Other organizations conducting SLAMS or SLAMS related $PM_{2.5}$ monitoring
- Recommendations and guidance are non-mandatory or nonbinding

470-17-5

Conventions

- *Shall* and *must* refer to mandatory method requirements.
- *Should* refers to an activity or procedure strongly recommended.
- *May* refers to an activity or procedure that is optional or discretionary.

470-17-6

Reference Methods

- Sampler design and performance specifications are in Appendix L of 40 CFR Part 50.
- Sampler design specifications
- Sampler performance specifications

470-17-7

Sampler Design Specifications

- Sampler inlet
- Downtube
- Impactor
- Impactor filter
- Filter holder assembly
- Flow rate measurement adaptor
- Internal surfaces finish
- Sampling height

470-17-8

Sampler Performance Specifications

- Sample flow rate
- Sample air flow rate control system
- Sampler flow rate regulation
- Flow rate cut off
- Flow rate measurement
- Leak test capabilities

470-17-9

Sampler Performance Specifications
(continued)

- Ambient temperature
- Relative humidity
- Pressure operational requirements
- Clock/timer system
- Data reporting requirements

470-17-10

Equivalent Methods

- More flexible in design, configuration, and operating principle
- New measurement technologies allowed
- Must demonstrate comparability to reference method
- Requirements specified in 40 CFR Part 53
- Three classes acceptable
  - Class I
  - Class II
  - Class III

470-17-11

Limitations of Reference and Class I Equivalent Methods

- Air flow rate must be maintained.
- Changes in weight are affected by sample mishandling, chemical reactions, and volatilization.
- Buildup of electrostatic charge on filters during their manufacture or during sampling can cause error.

470-17-12

Personnel Qualifications, Training, and Health and Safety Warnings

- Personnel Qualifications
- Training
- Health and Safety Warnings

470-17-13

Personnel Qualifications

- Laboratory personnel
- Field personnel

470-17-14

Laboratory Personnel Qualifications

Laboratory personnel should be able to

- measure temperature, relative humidity, and pressure.
- operate microbalance and antistatic devices.
- use common methods to determine temperature, pressure, flow rate, and relative humidity in the field.
- record and enter data into AIRS and other databases.

470-17-15

Field Personnel Qualifications

Field personnel should be able to

- operate the $PM_{2.5}$ sampler.
- calibrate, audit, maintain, and troubleshoot the sampler.
- use common methods to determine temperature, pressure, flow rate, and relative humidity in the field.
- record and enter data into AIRS and other databases.

470-17-16

Training Program for $PM_{2.5}$ Measurements

- Train field personnel familiar with PM_{10} and TSP measurements.
- Train laboratory personnel familiar with weighing room techniques.
- Study Section 2.12 of QA Handbook, and Federal Register $PM_{2.5}$ rulemaking.
- Develop a training manual and operations checklist.

470-17-17

Training Program for $PM_{2.5}$ Measurements
(continued)

- Develop local detailed SOPs and QAPP.
- Attend State/Regional workshops and EPA sponsored training programs.
- Use on-the-job training.
- Administer hands-on testing procedures.

470-17-18

Health and Safety Warnings

- Electrical
- Chemical
- Equipment placement and stability

470-17-19

Summary Information

- Principles of reference method
- Calculation of mass concentration
- Sampler design illustration
- Field QA/QC checks
- Laboratory QA/QC checks
- Sampling procedures

470-17-20

Principles of $PM_{2.5}$ Reference Method

- Sample is drawn at a flow rate of 16.67 L/min through specially designed inlet.
- Particles greater than 10 µm in diameter are removed.
- Remaining particles are sent to next stage.
- Particles less than 10 µm but greater than 2.5 µm are removed.
- Particles ≤ 2.5 µm are collected on PTFE filter.

470-17-21

Sample Volume

$$V_a = \frac{Q_{avg} \times t}{10^3}$$

Where:

V_a = total sample volume, actual m^3

Q_{avg} = average sample flow rate over the sample collection period, L/min

t = total elapsed sample collection time, min

10^3 = units conversion

470-17-22

Net $PM_{2.5}$ Mass Calculation

$$M_{2.5} = (M_f - M_i) \times 10^3$$

Where:

$M_{2.5}$ = total mass of $PM_{2.5}$ collected during the sampling period, µg

M_f = final mass of the equilibrated filter after sample collection, mg

M_i = initial (tare) mass of the equilibrated filter before sample collection, mg

10^3 = units of conversion, (µg/mg)

470-17-23

$PM_{2.5}$ Concentration Calculation

$$PM_{2.5} = M_{2.5}/V_a$$

Where:

$PM_{2.5}$ = $PM_{2.5}$ mass concentration, µg/m³

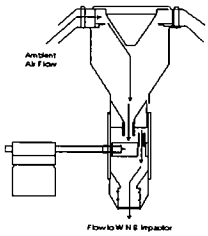
$M_{2.5}$ = total mass collected, µg

V_a = total sample volume, m³

470-17-24

Sampler Design Illustration

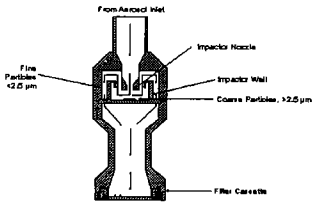
Inlet of the $PM_{2.5}$ sampler



470-17-25

Sampler Design Illustration

Impactor and filter holder assembly



470-17-26

Field QA/QC Checks

- Requirements (such as calibration standards, calibration/verifications)
- Frequency of checks
- Acceptance criteria
- CFR reference
- QA Handbook Vol. II, Part II, of Section 2.12

470-17-27

Calibration Standard for Flow Rate Transfer Standard

- Frequency - 1/yr
- Acceptance criteria - ± 2% of NIST traceable std.
- CFR reference - Part 50, App. L, Sec. 9.1, 9.2
- Section 2.12 reference - Sec. 6.3.3
- Information provided - Certification of traceability

470-17-28

Calibration/Verification for a Flow Rate Calibration

- Frequency - If multipoint failure
- Acceptance criteria - ± 2% of transfer standard
- CFR reference - Part 50, App. L, Sec. 9.2
- Section 2.12 reference - Sec. 6.3
- Information provided - Calibration drift and memory effects

470-17-29

Laboratory QA/QC Checks

- Requirements (such as blanks, accuracy, precision)
- Frequency of checks
- Acceptance criteria
- CFR reference
- Section 2.12 reference

470-17-30

Laboratory Blanks

- Frequency - 10% or 1 per weighing run
- Acceptance criteria - ± 15 µg difference
- CFR reference - Part 50, App. 1, Sec. 8.2
- Section 2.12 reference - Subsection 7.7
- Information provided - Laboratory contamination

470-17-31

Balance Audit

- Frequency - 1 per year
- Acceptance criteria - ± 15 µg for unexposed filters
- CFR reference - none
- Section 2.12 reference - Subsection 7.2
- Information provided - Verification of equipment operation

470-17-32

Summary of Sampling Procedures

- Prepare for site visit.
- Assemble equipment and supplies.
- Install filter cassette and begin sample run.
- End sample run and remove filter cassette.

470-17-33

Procurement and Acceptance Testing of Equipment and Supplies

- Field operation equipment
- Laboratory operation equipment

470-17-34

Field Operation Equipment Acceptance Test

- Equipment - Reference or equivalent method sampler
- Acceptance checks
  - Sampler and accessories must be complete with no evidence of damage.
  - Model must be designated as reference or equivalent method sampler.
  - Pump and display must work.

470-17-35

Field Operation Equipment Acceptance Test
(continued)

- Acceptance limits - Specifications outlined in 40 CFR Part 50, Appendix L
- Action if requirements are not met - Reject sampler

470-17-36

Laboratory Operation Equipment

- Filter media
- Filter cassettes and containers
- Shipping and filter-handling container
- Analytical microbalance
- Mass reference standards

470-17-37

Filter Media Acceptance Testing

- Equipment - filters, Teflon®
- Acceptance check - Must be correct type and undamaged
- Acceptance limits - Type as described in 40 CFR Part 50, Appendix L
- Action if requirements are not met - Reject filters

470-17-38

Analytical Microbalance

- Capacity - 100 to 200 µg
- Size (pan/opening) - sufficient room to weigh 46.2 mm diameter filters
- Minimum readability - ± 1 µg
- Repeatability - 1 µg

470-17-39

Sampler Installation

- Spatial and temporal scales
- Probe siting
- Safety, electrical, and security considerations
- Installation procedures

470-17-40

Spatial and Temporal Scales

- Most $PM_{2.5}$ monitoring in urban areas should be representative of a neighborhood scale.
- Regional transport should be characterized by urban or regional scales.
- Microscale sites may be used.
- However, core SLAMS on this scale should be limited to urban sites that are representative of long-term human exposure and many such microenvironments in the area.

470-17-41

Siting Requirements

- Unobstructed air flow must be minimum of 2 m in all directions.
- Sufficient room for a collocated sampler is required.
- Vertical placement must be 2 to 15 m above ground level.
- Microscale sites sampler inlet must be 2 to 7 m above ground level.

470-17-42

Siting Requirements
(continued)

- If collocated $PM_{2.5}$ sampler, spacing must be ≥1m.
- Spacing between inlets must be no more than 4m.
- Inlet heights of both samplers should be within 1mm of each other.

470-17-43

Considerations for Sampler Installation

- Safety
  - Must be located where operator can reach it safely regardless of weather
  - If on rooftop, must be slip proof during inclement weather
  - Must be relatively easy to transport all necessary supplies and equipment

470-17-44

Considerations for Sampler Installation

- Electrical
  - Must operate at 105 to 125 volts, AC, and frequency of 59-61 Hz
  - May require slow blow fuse
  - Should have stable power source
  - Must have enough power to run collocated sampler and an FRM performance evaluation sampler simultaneously

470-17-45

Considerations for Sampler Installation

- Security
  - Depends on location
  - Rooftop sites - locked access
  - Ground-level sites - fence
  - Fences - chain-linked or similar to avoid obstruction of air flow
  - Sampler inlet - extend above fencetop

470-17-46

Installation Procedures

- Sampler receipt
- Laboratory evaluation
- Sampling site setup
- Field evaluation

470-17-47

Calibration Procedures

- General aspects
- Calibration of flow rate
- NIST traceability and certification
- Generic calibration procedure
- Calibration of temperature and pressure sensors
- Leak checks
- Calibration and verification frequencies

470-17-48

Overview of Calibration Procedure

- Multipoint calibration
- Multipoint verification
- Single point verification

470-17-49

Calibration of Flow Rate Measurement System

- General requirements and guidance
- Flow rate calibration standards

470-17-50

General Requirements and Guidance

- Measurements must be based on actual volumetric units.
- Q_a is actual volumetric flow rate measured at existing conditions of temperature and pressure.
- V_a is the air volume measured or expressed at ambient (actual) conditions of temperature and pressure.

470-17-51

Flow Rate Calibration Standards

- Bubble flowmeters
- Piston flowmeters
- Mass flowmeters
- Orifice devices
- Laminar flow elements
- Wet test meters
- Dry test meters

470-17-52

National Institute of Science and Technology (NIST) Traceability and Certification

- Flow rate standard should have its own certification and should be traceable to other standards, which are traceable to an NIST standard.
- Other standards should be checked for accuracy and stability.
- Recertification should be conducted annually.

470-17-53

Generic Flow Rate Calibration Procedure

- Ensure sampler temperature and sensors are calibrated.
- Equilibrate flow rate calibration device to ambient conditions.
- Install filter cassette with an unused filter.
- Warm up sampler for 10 to 15 minutes.
- Remove sampler inlet and replace with flow calibration device.

470-17-54

Generic Flow Rate Calibration Procedure (continued)

- Follow multipoint flow calibration instructions in operator's manual.
- After completing calibration, turn off sampler pump, remove filter and filter cassette, remove flow calibration device, and replace the sampler inlet.

470-17-55

Calibration of Sampler Temperature Sensors

- General requirements
- Temperature calibration standards
- NIST traceability and certification
- Generic calibration procedure

470-17-56

General Calibration Requirements for Temperature Sensors

- A multipoint (at least three temperature points) calibration followed by a single point verification must be performed annually.
- Three separate temperature measurements must be evenly spaced over operational ambient temperature range.
- Ambient air and filter temperature are monitored.

470-17-57

General Calibration Requirements for Temperature Sensors
(continued)

- Ideally temperature calibrations should occur at the field; however indoor location may be preferable.
- Monthly verification should consist of one temperature measurement made at sampler's operating temperature.
- One point verification may be substituted for a three point calibration, if three-point calibration is conducted upon initial installation and at least annually thereafter.

470-17-58

General Calibration Requirements for Temperature Sensors
(continued)

- Complete three-point calibration must be conducted if one-point verification shows difference of ± 4°C from standard temperature.
- One-point verification should be done following the three-point calibration.

470-17-59

Temperature Calibration Standards

- Insulated vacuum bottles (thermos bottles)
- Solid cylinders of aluminum metal
- ASTM or NIST traceable mercury-in-glass thermometer

470-17-60

NIST Traceability and Certification

- Temperature standard must have its own certification traceable to NIST primary standard.
- Calibration relationship to temperature standard is established accurate to within 0.5°C over range of ambient temperatures.
- Temperature standard must be reverified and recertified at least annually.

470-17-61

Generic Temperature Calibration Procedure

- Remove ambient temperature sensor from radiation shield and place in constant temperature bath while still connected to the sampler's signal conditioner.
- Prepare a container for the ambient temperature water bath and ice slurry bath.
- Wrap sensor(s) and a thermometer with rubber band and immerse both in ambient temperature bath.

470-17-62

Generic Temperature Calibration Procedure
(continued)

- Allow temperatures to equilibrate.
- For each thermal mass, make five measurements.
- Accurately read meniscus of thermometer avoiding parallax errors.
- Average the five readings and record all readings.

470-17-63

Calibration of Sampler Pressure Sensors

- General Requirements
- Calibration Procedure

470-17-64

General Requirements

- Sampler should have the capability to measure the barometric pressure of the ambient air over a range of 600 to 800 mm Hg.
- Resolution must be to within 1 mm Hg with a NIST traceable accuracy of ± 5 mm Hg.

470-17-65

General Requirements
(continued)

- Barometer can be calibrated by comparing it with a secondary standard traceable to a NIST primary standard.
- Field barometer used to calibrate the sampler's pressure sensor must have a resolution to within 1 mm Hg with an accuracy of ± 5 mm Hg.

470-17-66

General Requirements
(continued)

- Fortin mercurial barometer is best employed as a higher quality laboratory standard for certification of the aneroid barometer.
- Precision aneroid barometer, though less accurate than the Fortin mercurial barometer, can be transported with less risk and presents no hazard form mercury spills.

470-17-67

General Requirements
(continued)

- Sampler pressure sensor can be left in the sampler during the comparison.
- Protect all barometers from violent mechanical shock and sudden changes in pressure.

470-17-68

Calibration Procedures for Fortin Type Barometer

- Read temperature from thermometer to nearest 0.1°C.
- Lower mercury level in cistern until index pointer is cleared, and raise level until dimple barely appears on the surface of mercury.
- Tap barrel, adjust vernier so base just cuts off light at the highest point of the meniscus, and avoid parallax errors.
- Read height of the mercury column.

470-17-69

Calibration Procedures for Aneroid Type Barometer

- Always use and read an aneroid barometer when it is in the same position (vertical or horizontal) as it was when calibrated.
- Locate the portable aneroid barometer next to the laboratory's primary standard.
- If the aneroid barometer has mechanical linkages, tap its case to overcome bearing drag.
- Read the aneroid barometer to the nearest 1 mm Hg.

470-17-70

Leak Checks

- External checks - sampler components to be subjected to this leak test include all components and their interconnections.
- Internal filter bypass check - determine if any portion of the sample flow that leaks past the sample filter without passing through the filter is significant relative to the design flow rate for the sampler.

470-17-71

Frequency of Calibrations and Verifications

- Flow rate measurement system
- Temperature
- Pressure

470-17-72

Flow Rate Calibration/Verification Frequency

- Multipoint verification should take place on installation, then at least annually, or when out of specification or following any major electrical or mechanical maintenance.
- Multipoint calibration is required upon failure of flow rate multipoint verification.
- Single point flow rate verification should take place every 4 weeks.

470-17-73

Temperature Calibration/Verification Frequency

- Temperature multipoint verification is recommended on installation, then annually or when out of specifications.
- Temperature multipoint calibration for both ambient air inlet and filter temperature sensors is required upon failure of multipoint verification.
- Temperature single point verification of ambient air inlet sensor and filter temperature sensor should be done every 4 weeks.

470-17-74

Pressure Calibration/Verification Frequency

- Pressure multipoint calibration is recommended on installation, then annually or when out of specifications.
- Pressure single point verification is recommended every 4 weeks.

470-17-76

Filter Preparation and Analysis

- Microbalance
- Microbalance environment
- Mass reference standards
- Filter handling
- Filter integrity checks
- Filter blanks
- Other checks

470-17-76

Microbalance

- Resolution of 1 µg
- Repeatability of 1 µg

470-17-77

Microbalance Environment

- Climate controlled
- Draft free room or chamber
- Clean area
- Proper grounding to reduce static

470-17-78

Mass Reference Standards

- Range is from 100 to 200 mg.
- Bracket weight of filter.
- Standards tolerance is less than 25 µg.
- Handle with smooth, nonmetallic, clean forceps.
- Verify working standards against NIST traceable primary standards every three to six months.

470-17-79

Filter Handling

- Powder-free gloves
- Smooth, clean forceps
- Clean filter handling container
- Unique identification number
- ^{210}Po antistatic strips, replaced every six months

470-17-80

Filter Integrity Checks

- No pinholes, separation, chaff, loose material
- No filter discoloration
- Uniformity

470-17-81

Filter Blanks

- Lot blanks
- Laboratory blanks
- Field blanks

470-17-82

Other Checks

- Presampling filter conditioning
- Pre- and post- sampling filter weighing
- Internal QC
- Postsampling filter storage
- Postsampling inspection, documentation, and verification
- Postsampling filter equilibration

470-17-83

Lesson 18

Quality Assurance Guidance for PM2.5 Ambient Air Monitoring- Part II

[redacted]

Office of Air Quality Planning and Standards
U.S. Environmental Protection Agency

APTI 470
Quality Assurance for Air Pollution
Measurement Systems

Field Operations

- Site visit activities
- Field operation activities to perform every five days
- Field operation activities to perform every four weeks

470-18-1

Site Visit Activities

- Beginning a run
- Ending a sampling period
- Validating samples
- Sample handling

470-18-2

Beginning a Run

- Complete sampling data sheet.
- Ensure sampler is not operating.
- Inspect filter for cleanliness or damage.
- Install filter cassette after O-ring check.
- Program controls for proper start time.
- Visually inspect site and equipment; note any changes in surroundings.

470-18-3

Beginning a Run
(continued)

- Visually inspect records of sampler.
- Check, measure, and record ambient temperature and pressure.
- Ensure that independent measurement of ambient temperature (inlet temperature) and pressure readings taken by sampler are within 4.0°C and 10 mm Hg of the independent readings.

470-18-4

Ending a Sampling Period

- Visually inspect sampler readouts for proper operation.
- Check for problems and record on data sheet.
- Download sampler data.
- Record date, stop time, total time, temperature, pressure, etc.

470-18-5

Validating Samples

- Sampler operating time
- Flow rates
- Filter quality
- Filter temperature
- Exceptional events - field qualifiers

470-18-6

Sample Handling

- Valid samples
- Questionable samples

470-18-7

Field Operation Activities to Perform Every Five Operating Days

- Clean impactor wells.
- Check water trap and empty if necessary.
- Clean interior of sampler.
- Inspect seals.
- Reinstall trap.

470-18-8

Field Operation Activities to Perform Every Four Weeks

- Conduct flow rate verification check.
- Conduct temperature and pressure verification check.
- Conduct leak check.

470-18-9

Sampler Maintenance

- Five sampling day procedure
- Monthly procedure
- Miscellaneous procedures
- Quarterly procedures
- Other periodic maintenance procedures

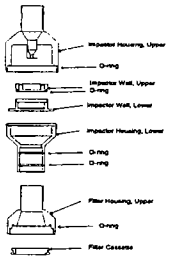
470-18-10

Five Sampling Day Procedure

- Inspect water collector bottle.
- Remove accumulated water.
- Replace bottle.
- Replace impactor well.
- Disassemble and clean impactor well.

470-18-11

$PM_{2.5}$ Impactor Well and Filter Holder



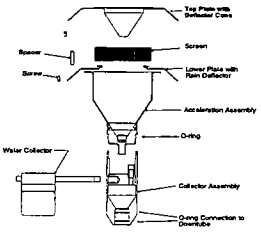
470-18-12

Monthly Procedure

- Sampler inlet
  - Dismantle and clean sampler inlet.
  - Reassemble and reinstall inlet.
- Impactor housing and well
  - Open impactor assembly and inspect interior.
  - Clean and dry assembly.
  - Check O-rings and replace if necessary.

470-18-13

Disassembled Sampler Inlet



470-18-14

Miscellaneous Procedures

- Clean interior of sampler case.
- Inspect and clean cooling air intake.
- Check internal clock.
- Check foam disks used to transport filter cassettes.
- Check filter cassettes and backing.

470-18-15

Quarterly Procedures

- Remove the O-rings in the aerosol inlet and condition them with a very light coating of vacuum grease.
- Remove the O-rings in the impactor assembly and apply a light coating of vacuum grease.
- Perform examinations of vacuum tubing, tubing fittings, and air intake filter and fans.

470-18-16

Other Periodic Maintenance

- Rebuild vacuum pump.
- Conduct leak check and recalibrate flow system.
- Refurbish sampler.

470-18-17

Performance Evaluation Procedures

- Performance evaluations
- Systems audits

470-18-18

Performance Evaluations

- Sampler Flow Rate Audit
- Temperature Audit
- Pressure Audit
- Assessment of Precision
- FRM Performance Evaluation
- Balance Accuracy Assessment

470-18-19

Sampler Flow Rate Audit

- One measurement is made at the sampler's operational flow rate.
- Flow rate standard used for the audit must not be the same standard used to verify or calibrate the sampler.
- Audit is conducted by the operator or by personnel from the QA unit of the reporting organization.
- Percentage difference should not be greater than ± 4%.

470-18-20

Temperature Audit

- Ambient temperature and filter temperature audits recommended based on the organization's QAPP.
- Audit conducted by reporting organization on a frequency specified in the organization's QAPP.
- Performance goals are those in QAPP.
- Use a bath of water, oil, or other suitable liquid.

470-18-21

Temperature Audit
(continued)

- Thermos bottle or Dewar flask should be used to insulate the bath.
- Three temperatures should be used.
- Any deviation greater than 2°C should be reported for corrective action.

470-18-22

Pressure Audit

- Use calibrated sensor such as an aneroid barometer.
- Audit is conducted by reporting organization.
- Audit frequency is specified in QAPP.
- Performance goals are specified in QAPP.
- Deviations greater than 10 mm Hg should be reported for corrective action.

470-18-23

Assessment of Precision

- Collocated sampler results are used to assess measurement system precision.
- Part 58 requires that 25% of primary samplers have another sampler collocated.
- Collocated samplers must collect a sample every sixth day.

470-18-24

Assessment of Precision
(continued)

- Collocated sampler results used by EPA to calculate quarterly and annual precision for each primary sampler and for each designated method used by each reporting organization.
- Data quality objective of 10% coefficient of variation or better is the goal for operational precision of $PM_{2.5}$ monitoring data.

470-18-25

FRM Performance Evaluation

- Accuracy of field $PM_{2.5}$ measurements is defined in a relative sense, by referencing the field measurements to a collocated Federal Reference Method (FRM) sampler.
- Accuracy is defined as the degree of agreement between a field $PM_{2.5}$ sampler and a collocated FRM sampler operating simultaneously and totally independent of site operations.

470-18-26

FRM Performance Evaluation
(continued)

- EPA Regional Offices may be conducting the FRM performance evaluations to assess total measurement system bias.
- 25% of the SLAMS $PM_{2.5}$ primary samplers within each reporting organization will be assessed with an FRM performance evaluation every calendar year.

470-18-27

FRM Performance Evaluation
(continued)

- Every designated FRM or FEM within a reporting organization must
  - have at least 25% of each method designation evaluated.
  - have at least one sampler evaluated.
  - be audited at a frequency of four evaluations per year.

470-18-28

FRM Performance Evaluation
(continued)

- Results from the primary sampler and the duplicate FRM sampler are used by EPA to calculate accuracy of the primary sampler on a quarterly basis, the bias of the primary sampler on an annual basis, and the bias of a single reporting organization on an annual basis.

470-18-29

Balance Accuracy Assessment

- Each State or reporting organization should conduct an internal accuracy assessment of each microbalance on an annual basis.
- Assessment requires the use of an independent set of ASTM Class I standard weights traceable to NIST.
- Weights of 100 and 200 mg are suggested for the audit and must not be the same ones used as working standards.
- Balance display should agree with designated weight of the audit weight to within ± 0.050 mg.

470-18-30

System Audits

- System audit activities
- Internal systems audits by State or Reporting Organizations
- External systems audits by EPA Regional Offices

470-18-31

System Audit Activities

- Initial equilibration, weighing, and transportation of the filters to the sampler
- Site selection criteria assessment
- Equipment installation
- Site security
- Equipment maintenance
- Calibration procedures
- Handling and placement of the filters

470-18-32

System Audit Activities
(continued)

- Proper operation of the sampler and sample collection
- Removal, handling, and transportation of the filter from the sampler to the laboratory
- Weighing, storage, and archival of the sampled filter
- Data analysis and reporting

470-18-33

Calculations, Validations and Reporting of $PM_{2.5}$ Monitoring Data

- Calculations
- Verification of manual calculations and data entry
- Validation of software
- Data reporting

470-18-34

Calculations

- Sample volume
- Net $PM_{2.5}$ mass calculation
- $PM_{2.5}$ concentration calculation

470-18-35

Sample Volume

$$V_a = (Q_{avg} \times t) / 10^3$$

Where:

V_a = total sample volume, actual m^3

Q_{avg} = average sample flow rate over the sample collection period, L/min

t = total elapsed sample collection time, min

10^3 = units conversion, m^3/L

470-18-36

Net $PM_{2.5}$ Mass Calculation

$$M_{2.5} = (M_f - M_i) \times 10^3$$

Where:

$M_{2.5}$ = total mass of $PM_{2.5}$ collected during the sampling period, µg

M_f = final mass of the equilibrated filter after sample collection, mg

M_i = initial (tare) mass of the equilibrated filter before sample collection, mg

10^3 = units of conversion, µg/mg

470-18-37

$PM_{2.5}$ Concentration Calculation

$$PM_{2.5} = M_{2.5}/V_a$$

Where:

$PM_{2.5}$ = $PM_{2.5}$ mass concentration, $\mu g/m^3$

$M_{2.5}$ = total mass collected, µg

V_a = total sample volume, m^3

470-18-38

Verification of Manual Calculations and Data Entry

- Manual Calculations
- Manual Data Entry
- Data Validations

470-18-39

Verification of Manual Calculations

- Gather the raw data sources to be checked.
- Obtain a copy of the resulting data report.
- Independently verify the results based on the raw data.
- Verify that the correct formulas, conversion constants, and reporting units were used.

470-18-40

Verification of Manual Data Entry

- Duplicating keying
- Proofing

470-18-41

Data Verification Purpose

- To verify that the data have been recorded, entered, and calculated correctly
- To screen potential outliers

470-18-42

Data Verification Techniques

- Graphing and visually examining time-series of operating parameter data such as flow checks
- Graphing and visually examining scatter plots of data
- Range checking
- Statistical checking
- Evaluation of goodness-of-fit and linearity
- Review of operators' notes and communication with operators to identify problems

470-18-43

Validation of Software Used to Process $PM_{2.5}$ Data

- Validation is to ensure that there are no incorrectly coded calculations and errors.
- A structured approach to software development, testing, and validation is recommended.
- Software testing is performed.

470-18-44

Example Areas to be Tested

- Correctness of calculations
- Correct assignment of input and output values
- Calculation of statistics
- Operation at the start of the year 2000
- Application of validation procedures, range checks, etc.

470-18-45

Validation of Software Used to Process $PM_{2.5}$ Data

- Use of spreadsheets for processing and managing large data sets is strongly discouraged.
  - Difficult to test thoroughly
  - Can develop new problems as data are added, i.e. predefined data range overflows
- Use of relational databases is preferred.

470-18-46

Data Reporting

- Rounding for data reporting
- Rounding rules for NAAQS comparison

470-18-47

Data and Records Management

- Methodology
- Records to create and retain
- Quarterly reporting requirements

470-18-48

Reasons for Data and Records Management

- Provide information on mechanical problems that occur and document how the problems were corrected.
- Provide a history of warranty repairs.
- Provide a history of in-house repairs and preventive maintenance servicing.
- Document date and site placement details.

470-18-49

Reasons for Data and Records Management
(continued)

- Be a useful source of information at the time of the annual network review .
- Provide evidence to support the quality of PM data submitted to regional and national $PM_{2.5}$ databases.

470-18-50

Methodology for Data and Records Management

- Personnel
- Quality assurance
- Facilities and equipment

470-18-51

$PM_{2.5}$ Records to Create and Retain

- Sampler siting and maintenance records
- Analytical laboratory installation
- Field sampling operation
- Weighing laboratory operation
- QA records

470-18-52

Quarterly Data Reporting Requirements

- Siting documentation
- $PM_{2.5}$ concentration data or sample weight and volume
- Information calculated and provided by the sampler
- Results of all valid precision, bias, and accuracy tests

470-18-53

Assessment of Measurement Uncertainty

- Flow rate audit
- Bias assessment
- Precision

470-18-54

Flow Rate Audit

- Flow rate must be audited each calendar quarter.
- Audit should be scheduled to avoid interference with the regularly scheduled sampling period.
- Times should be selected randomly.
- Accuracy of sampler's flow rate should be within ± 4% of the audit value.
- Audit measured flow rate accuracy should be within ± 5% of the design inlet flow rate (16.67 L/min).

470-18-55

Bias Assessment

- Assessment made from an FRM performance evaluation accomplished in AIRS
- Goal for acceptable bias is between -10% and +10%
- Performance evaluation requirements for SLAMS reporting organizations

470-18-56

FRM Performance Evaluation Requirements for SLAMS Reporting

- At least one sampler must be audited annually.
- At least 25% of each reference and equivalent method designation must be evaluated each year.
- 25% includes collocated sites, including those collocated with FRM samplers.
- Evaluations of the selected monitors must occur at least four times a year.
- All samplers must be evaluated at least once every four years.

470-18-57

FRM Performance Evaluation Requirements for SLAMS Reporting (continued)

- Should emphasize assessing sites with concentrations around the NAAQS.
- Individual sampler and audit measurements must be reported to EPA.
- EPA will use data to calculate single sampler bias and quarterly average bias for a reporting organization.

470-18-58

Precision

- Assessed by collocating samplers
- Number of collocated samplers
- Location of collocated samplers
- Schedule for operation of collocated samplers

470-18-59

Location of Collocated Samplers

- Place at sites having the highest $PM_{2.5}$ concentrations.
- Emphasize sites expected to be in violation of the NAAQS.

470-18-60

Location of Collocated Samplers (continued)

SLAMS reporting organizations that have areas in violation of the NAAQS should place their collocated samplers as follows:

- With sites reporting $PM_{2.5}$ concentrations equal to or exceeding 90% of the NAAQS,
  - 80% of the collocated samplers should be located at those sites that have concentrations that equal or exceed 90% of the NAAQS.
  - the remaining 20% of the collocated samplers should be located at sites that have concentrations less than 90% of the NAAQS.

470-18-61

Location of Collocated Samplers (continued)

- Without sites reporting concentrations exceeding 90% of the NAAQS,
  - 60% of the collocated samplers should be located at sites that rank in the top 25% of the highest $PM_{2.5}$ concentration sites.
  - the remaining 40% of the collocated samplers should be distributed among the remaining 75% of the sites.

470-18-62

Number of Collocated Samplers

- At least one reporting sampler within a reporting organization must have a collocated sampler.
- At least one of the collocated samplers must be an FRM sampler.
- At least 25% of all reporting samplers must have collocated samplers.

470-18-63

Number of Collocated Samplers
(continued)

- Collocated samplers for FRM designated reporting samplers shall always be of the identical FRM designation.
- If the reporting sampler is an FEM, half of the collocated samplers must have the identical equivalency designation while the other half are FRM designated samplers.

470-18-64

Schedule for Operation of Collocated Samplers

- Collocated samples should be collected to reflect the normal operation of the primary reporting sampler.
- Collocated samples should be evenly distributed across seasons and days of the week.
- Both the collocated and reporting samplers should be started and stopped at the same time.

470-18-65

Lesson 19

Quality Assurance Procedures for Monitoring PM10 in Ambient Air Using a High-Volume Sampler

[redacted]

Office of Air Quality Planning and Standards
U.S. Environmental Protection Agency

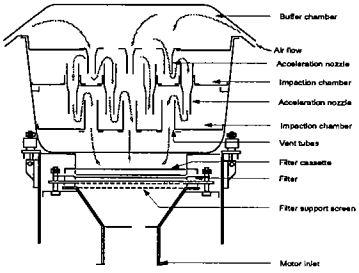
APTI 470
Quality Assurance for Air Pollution
Measurement Systems

Sampler Basic Components

- Sampler inlets
  - Impaction inlet
  - Cyclonic inlet
- Flow control systems
  - Mass flow control (MFC) system
  - Volumetric flow control (VFC) system

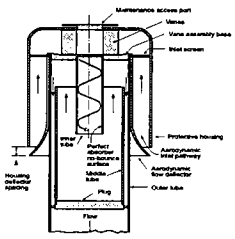
470-19-1

Impaction Inlet



470-19-2

Cyclonic Inlet



470-19-3

Flow Control Systems

- Mass flow control system
- Volumetric flow control system

470-19-4

Procurement of Equipment and Supplies

- Field Operations
- Laboratory Operations

470-19-5

Field Operations

- HV Samplers
- Calibration QA/QC Supplies
- QC Flow-Check Device
- Audit Equipment

470-19-6

HV Samplers

- Must meet EPA operational standards
- Requirements in 40 CFR Part 50, Appendix M

470-19-7

Calibration QA/QC Supplies

- In-house inventory
- Thermometer, range 0 to 50°C, scale 0.1°C, NIST certified
- Barometer, range 500 to 800 mm Hg
- Orifice transfer standard, calibration relationship referenced annually, ± 2%
- Manometer, range 0 to 400 mm H_2O, scale 2 mm

470-19-8

QC Flow-Check Device

- Routine operation
- NIST-traceable
- Volumetric flows of 1.02 to 1.24 m^3/min
- Calibrated annually, within ± 2%
- Same one used for calibration

470-19-9

Audit Equipment

- MUST be a different device

470-19-10

Laboratory Operations

- Filter media
- Filter protection
  - Filter cassettes
  - Protective covering
- Analytical balance
- Mass reference standards

470-19-11

Calibration Procedures

- Overview
- Flow Rate Measurement and General Aspects of PM_{10} Sampler Calibration
- Certification of an Orifice Standard
- Basic Calibration Procedure for MFC Sampler
- Basic Calibration Procedure for VFC Sampler
- HV Sampler Calibration Frequency

470-19-12

Overview

- All sampling and analysis equipment must be properly calibrated.
- PM_{10} reference standards are not available; individual components must be calibrated instead.

470-19-13

Flow Rate Measurement and General Aspects of PM_{10} Sampler Calibration

- Average T_{av} and P_{av} for 24 hr period
- If T_{av} and P_{av} not obtained, use seasonal averages
- Calibration in actual volumetric flow

470-19-14

Certification of an Orifice Standard

- Orifice Calibration Procedure
- Orifice Transfer Standard Calibration Frequency

470-19-15

Orifice Calibration Procedure

- Assemble equipment.
- Record equipment identification numbers.
- Record barometric pressure and temperature.
- Connect orifice transfer standard to inlet.
- Level meter and check for leaks.
- Zero water and mercury manometers.
- Adjust to first flow rate.

470-19-16

Orifice Calibration Procedure
(continued)

- Record initial volume, pass at least 3 m^3.
- Record volume meter's inlet pressure.
- Record elapsed time.
- Calculate volume measured.

$\Delta Vol = Final\ volume - Initial\ volume$

470-19-17

Orifice Calibration Procedure
(continued)

- Correct to ambient atmospheric pressure.

$$V_a = \Delta Vol\ (P_a - \Delta Hg) / P_a$$

Where: V_a = actual volume at ambient barometric pressure, m^3

ΔVol = actual volume measured by the standard volume meter, m^3

P_a = ambient barometric pressure during calibration, mm Hg (or kPa)

ΔHg = differential pressure at inlet to volume meter, mm Hg (or kPa)

470-19-18

Orifice Calibration Procedure
(continued)

- Calculate actual volumetric flow rate.

$$Q_a = V_a / \Delta Time$$

Where: Q_a = actual volumetric flow rate through the orifice, m^3/min

V_a = actual volume at ambient barometric pressure, m^3

$\Delta Time$ = elapsed time, min

470-19-19

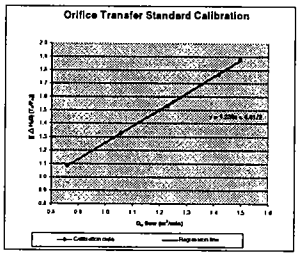
Orifice Calibration Procedure
(continued)

- Repeat for at least four additional flow rates.
- Compute $[\ (\Delta H_2O)\ (T_a/P_a)\ ]^{1/2}$ for each flow rate.
- Draw orifice transfer standard calibration curve.
- Calculate the slope (m), intercept (b), and correlation coefficient (r) of the linear least-squares regression.

470-19-20

Orifice Calibration Procedure
(continued)

- Plot regression line on same graph as calibration data
- Readable to 0.02 m^3/min
- Within 2% of line



470-19-21

Orifice Calibration Procedure
(continued)

- For future use of the orifice standard, calculate Q_a.

$$Q_a \text{ (orifice)} = \{ (\Delta H_2O)\,(T_a / P_a)^{1/2} - b \}\,(1/m)$$

470-19-22a

Orifice Calibration Procedure
(continued)

Where:

Q_a (orifice) = actual volumetric flow rate as indicated by the orifice transfer standard, m^3/min

ΔH_2O = pressure drop across the orifice, mm (or in.) H_2O

T_a = ambient temperature during use, K (K = °C + 273)

P_a = ambient barometric pressure during use, mm Hg (or kPa)

b = intercept of the orifice calibration relationship

m = slope of the orifice calibration relationship

470-19-22b

Orifice Transfer Standard Calibration Frequency

- Upon receipt
- At least annually
- When nicks or dents are visible

470-19-23

Basic Calibration Procedure for MFC Sampler

- Overview
- Calibration Equipment
- Multipoint Flow Rate Calibration Procedure
- Calibration Calculations

470-19-24

Overview

- Flow rates are determined by an orifice transfer standard.
- Recommended exit orifice plenum pressure is measured with a 25 cm water or oil manometer.
- Each sampler should have its own dedicated manometer.

470-19-25

Calibration Equipment

- Orifice transfer standard traceable to NIST
- An oil or water manometer with a 0 to 400 mm (0 to 16 in) range, scale division of 2 mm (0.1 in)
- A sampler oil and water manometer with a 0 to 200 mm (0 to 8 in) range, scale division of 2 mm (0.1 in) for measurement of sampler exit orifice plenum pressure

470-19-26

Calibration Equipment
(continued)

- Thermometer range of 0 to 50°C to the nearest 0.1°C traceable to NIST
- Portable aneroid barometer range of 500 to 800 mm Hg, sensitivity to nearest 1 mm Hg, referenced within 5 mm Hg of a barometer of known accuracy annually
- Miscellaneous handtools, calibration data sheets, and duct tape

470-19-27

Multipoint Flow Rate Calibration Procedure

- Set up calibration system.
- Disconnect motor from flow controller.
- Install orifice transfer standard.
- Check all gaskets and replace as needed.
- Select first calibration flow rate, install appropriate resistance plate or adjust the variable orifice valve.
- Conduct leak test.

470-19-28

Multipoint Flow Rate Calibration Procedure
(continued)

- Eliminate any leaks before proceeding.
- Inspect connecting tubing.
- Adjust manometer's sliding scales.
- Connect orifice transfer standard manometer to the orifice transfer standard.
- Connect sampler's exit orifice manometer to the exit orifice plenum port.

470-19-29

Multipoint Flow Rate Calibration Procedure
(continued)

- If a continuous recorder is used, record site location, sampler S/N, date, and operator's initials on the blank side of a clean recorder chart, and install recorder chart.
- Read and record date, location, and operator's signature; sampler S/N and model; ambient barometric pressure; ambient temperature, and orifice S/N and calibration relationship.
- Turn on sampler and allow it to warm up.

470-19-30

Multipoint Flow Rate Calibration Procedure
(continued)

- Install the other resistance plates or adjust the variable orifice value.
- Plot the calibration data.
- Turn off sampler and remove the orifice transfer standard.
- Reconnect the sampler motor to the flow controller.
- Perform calibration calculations.

470-19-31

Calibration Calculations

- Calculate and record Q_a for each calibration point.

$$Q_a\,(\text{orifice}) = \{ (\Delta H_2O)\,(T_a / P_a)^{1/2} - b \}\,(1/m)$$

Where:

Q_a (orifice) = actual volumetric flow rate as indicated by the orifice transfer standard, m^3/min

ΔH_2O = pressure drop across the orifice, mm (or in.) H_2O

T_a = ambient temperature during use, K (K = °C + 273)

P_a = ambient barometric pressure during use, mm Hg (or kPa)

b = intercept of the orifice calibration relationship

m = slope of the orifice calibration relationship

470-19-32

Calibration Calculations
(continued)

- Calculate and record ΔP_{ext} for each calibration point.

$$\Delta P_{ext} = [\,\Delta P_{ex}\,(T_a + 30) / P_a\,]^{1/2}$$

Where: ΔP_{ext} = transformed manometer reading

ΔP_{ex} = sampler manometer reading, mm (or in.) H_2O

T_a = ambient temperature, K (K = °C + 273)

P_a = ambient barometric pressure, mm Hg (or kPa)

470-19-33

Calibration Calculations
(continued)

- If a continuous flow recorder is used, calculate the quantity as follows:

$$It = I\,[\,(T_a + 30) / P_a\,]^{1/2}$$

Where: It = transformed flow recorder chart reading

I = flow recorder chart reading, arbitrary units on a square root scale

- Note: If recorder charts with linear scales are used, substitute $(I)^{1/2}$ for I

470-19-34

Calibration Calculations
(continued)

- Plot the calculated Q_a flow rates on the x-axis versus ΔP_{ext} on the y-axis.
- Because Q_a depends on ambient average temperature and pressure, the use of graphic plots is not recommended for future data reduction.
- Use plot to visually assess calibration points to see if any should be rerun.

470-19-35

Calibration Calculations
(continued)

- Plot the regression line.
- For the regression model $y = mx + b$, let $y = \Delta P_{ext}$ and $x = Q_a$ (orifice); therefore

$$\Delta P_{ext} = m [Q_a (orifice)] + b.$$

470-19-36

Calibration Calculations
(continued)

- For the flow recorder, the model is

$$It = m [Q_a (orifice)] + b.$$

470-19-37

Calibration Calculations
(continued)

- Determine m, b, and r.
- Correlation coefficient of $r > 0.990$, no point deviating more than 0.04 m^3/min from the predicted value.

470-19-38

Calibration Calculations
(continued)

- For subsequent sample periods, the flow rate is calculated as follows:

$$\overline{Q_a} = \{ [\overline{\Delta P_{ex}} (T_{av} + 30) / P_{av}]^{1/2} - b \} (1/m)$$

470-19-39a

Calibration Calculations
(continued)

Where: $\overline{Q_a}$ = the sampler's average actual flow rate, m^3/min

$\overline{\Delta P_{ex}}$ = average of initial and final sampler manometer readings $(\Delta P_{ex_i} + \Delta P_{ex_f}) / 2$, mm

T_{av} = average ambient temperature for the sample period, K (K = °C + 273)

P_{av} = average ambient pressure for the sample period, mm Hg (or kPa)

b = intercept of the sampler calibration relationship

m = slope of the sampler calibration relationship

470-19-39b

Calibration Calculations
(continued)

- For the flow recorder, the calculation is:

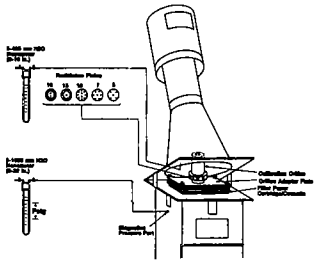
$$\overline{Q_a} = \{ \bar{I} \, (T_{av} + 30) / P_{av} \}^{1/2} - b\} \, (1/m)$$

Where: $\bar{I}$ = average flow recorder reading for the sample period

- If recorder charts with linear scales are used, substitute $(I)^{1/2}$ for I

470-19-40

Calibration Procedure -- VFC Sampler



Typical VFC PM_{10} HV sampler

470-19-41

HV Sampler Calibration Frequency

- At least quarterly or annually
- After relocation
- After repairs
- If field calibration flow check exceeds QC limits
- If field flow check audit shows sampler out of calibration

470-19-42

Field Operations

- Siting Requirements
- Sampler Installation Procedures
- Example Sampling Operations for an MFC Sampler
- Sample Validation and Documentation
- Field QC Flow Check Procedure

470-19-43

Siting Requirements

- Spatial scales
- Temporal scales
- Accessibility
- Electricity
- Security

470-19-44

Sampler Installation Procedures

- Inspect upon receipt.
- Perform operational check.
- Transport to field site.
- Follow manufacturer's instructions; bolt to a secure mounting surface.
- Assemble sampler inlet and install.
- Check tubing.
- Perform a multiunit flow rate calibration.

470-19-45

Example Sampling Operations for an MFC Sampler

- Presampling Filter Preparation
- Filter Installation
- Filter Recovery
- Postsampling Filter Handling

470-19-46

Presampling Filter Preparation

- Cassettes can be loaded at sampling site.
- Technicians should wear protective gloves.
- Filters should be kept in protective folders or boxes.
- Never bend or fold filters.
- Consistently label filters on one side.
- Put protective cover over filter cassette.

470-19-47

Filter Installation

- Access filter support screen.
- Examine filter support screen.
- Lower and inspect sample inlet.
- Examine flow recorder; remove any moisture.
- Record the sampler S/N, filter ID number, and site location on back of a clean chart and install the chart in the flow recorder.

470-19-48

Filter Installation
(continued)

- Advance the chart, verify pen rests on zero, adjust as necessary.
- Turn on sampler, equilibrate to operating temperature.
- Record the following parameters:
  - Site location
  - Sample date
  - Filter ID number
  - Sampler model and S/N
  - Operator's initials

470-19-49

Filter Installation
(continued)

- Inspect manometer.
- Measure ΔP_{ex}.
- Verify flow recorder is operational.
- Turn the sampler off.
- Reset the sampler timer.
- Close the sampler door.

470-19-50

Filter Recovery

- Turn on the sampler; equilibrate to operating temperature.
- Measure the final ΔP_{ex} and record.
- Turn off the sampler.
- Remove flow recorder chart, examine trace.

470-19-51

Filter Recovery
(continued)

- Record the following parameters:
  - Elapsed time of the sampling
  - Average recorder response
  - T_{av}
  - P_{av}

470-19-52

Filter Recovery
(continued)

- Calculate and record the average actual flow rate.

- For the flow recorder, the calculation is:

470-19-53a

Filter Recovery
(continued)

Where: $\overline{Q_a}$ = average sampler flow rate, actual m^3/min

$\overline{\Delta P}$ = average initial and final sampler manometer readings $(\Delta P_{ex_i} + \Delta P_{ex_f})/2$, mm (or in.) H_2O

$\overline{I}$ = average flow recorder response, arbitrary units

T_{av} = average ambient temperature for the run day, K

P_{av} = average ambient pressure for the run day, mm Hg or kPa

470-19-53b

Filter Recovery
(continued)

b = intercept of the MFC sampler calibration relationship

m = slope of the MFC sampler calibration relationship

[Note: If charts with linear-function scales are used, substitute $(I)^{1/2}$ for I.]

470-19-53c

Filter Recovery
(continued)

- Observe conditions around the monitoring site.
- Raise the sampler inlet; remove the filter cassette.
- Replace the cassette protective cover.
- Keep filter cassette level; transport it to the laboratory.

470-19-54

Postsampling Filter Handling

- Remove the top frame of the filter cassette.
- Conduct a secondary check.
- Slip folder underneath the edge of the exposed filter.
- Center the filter on folder.

470-19-55

Postsampling Filter Handling
(continued)

- Fold manila folder lengthwise.
- Insert folder into protective envelope.
- Deliver in its protective folder to the analytical laboratory.

470-19-56

Sample Validation and Documentation

- Field Validation
- Laboratory Validation
- Data Documentation

470-19-57

Field Validation

- Timing
- Flow rate

470-19-58

Laboratory Validation

- Check filter for signs of air leakage.
- Check for physical damage.
- Check appearance of particles.

470-19-59

Data Documentation

- Operator who starts the sample
- Operator who removes the sample

470-19-60

Field QC Flow Check Procedure

- MFC Sampler
- VFC Sampler

470-19-61

MFC Sampler

- Determine manometer reading.
- Record ambient temperature and pressure.
- Calculate actual sampler flow rate using sampler's calibration relationship.

470-19-62

VFC Sampler

- Determine relative stagnation pressure.
- Record ambient temperature and pressure.
- Calculate actual flow rate using sampler's calibration relationship.

470-19-63

Filter Preparation and Analysis

- Filter Handling
- Filter Integrity Check
- Filter Equilibration
- Initial Weighing Procedures
- Internal QC
- Post Sampling Documentation and Inspection
- Final Weighing Procedure (Gross Weight)
- Calculation of Net Mass Filter Loading

470-19-64

Filter Handling

- Package tare weighed filters in groups of 50 or less.
- Separate filters with a sheet of tracing paper.
- Assign a filter number to each filter.
- Stack filters in numerical order.
- Ship filters in reinforced envelopes and manila folders.

470-19-65

Filter Integrity Check

- Visually check each filter.
- Check for pinholes.
- Check for loose material.
- Check for filter nonuniformity.
- Check for other imperfections.

470-19-66

Filter Equilibration

- At least 24 hours before weighing
- Relative humidity between 20 and 45%
- Temperature between 15 and 30°C ± 3°C

470-19-67

Initial Weighing Procedures

- Ensure that the balance has been calibrated at least annually.
- Allow balance to warm up for five minutes.
- Begin with zero balance.
- QC supervisor performs "standard" filter QC check.
- Weigh filter.
- Record balance number, filter ID number, and tare weight.

470-19-68

Internal QC

- Standard weight check
- Zero and calibration checks
- Tare and gross weight checks
- QC supervisor responsibilities

470-19-69

Postsampling Documentation and Inspection

- Examine data sheet.
- Remove filter.
- Recover any material dislodged from filter.
- Match filter ID to recorded balance ID numbers.
- Remove filter and examine filter for any damage.
- Remove any embedded insects.

470-19-70

Postsampling Documentation and Inspection

- Place defect-free filters in protective envelope and forward to laboratory for weighing and analysis.
- File data sheets.
- Return defective filters to original protective envelopes.

470-19-71

Final Weighing Procedure (Gross Weight)

- Group filters.
- Place defect-free filters in conditioning environment.
- Repeat steps conducted in filter tare weighing procedure.
- Perform internal QC checks.
- Record gross weight on laboratory data/coding form.

470-19-72

Final Weighing Procedure (Gross Weight)

- Archive filter.
- If further analysis to be performed, return filter to protective covering and note.
- Asterisk to indicate further analysis.
- Forward to laboratory for further analysis.

470-19-73

Calculation of Net Mass Filter Loading

- Gross weight minus the tare weight is the net mass of the particulate.
- Each calculation must be independently validated.

470-19-74

Calculation, Validation, and Reporting

- Calculations
- Calculation Validation
- Data Reporting and Interpretation

470-19-75

Calculations

- Flow rate calculations MFC sampler
- Flow rate calculations VMC sampler
- PM_{10} concentration calculation

470-19-76

Flow Rate Calculations
MFC Sampler

- Determine initial and final manometer reading or average flow recorder trace.
- Determine average ambient temperature.
- Determine average barometric pressure.
- Apply these values to the calibration relationship.

470-19-77

Flow Rate Calculations
VMC Sampler

- Calculate average absolute stagnation pressure.
- Determine ratio of average absolute stagnation pressure to average barometric pressure.
- Determine ambient average temperature.
- Determine average flow rate from average stagnation pressure ratio and average temperature for sample period.

470-19-78

PM_{10} Concentration Calculation

- Determine total volume sampled.
- Determine the net mass in µg.
- Determine mass concentration in $\mu g/m^3$.

470-19-79

Calculation Validation

- Collect total sampling time data, average actual flow rate data, and tare and gross weights.
- Recalculate the total mass concentration.
- If errors found, values in that sample lot should be recalculated.
- Scan all total mass concentration values, investigate if necessary.

470-19-80

Calculation Validation
(continued)

- Recompute the total mass concentration.
- Correct errors found, initial them, and indicate date of correction.
- If exceedingly high or low values still exist, review all raw data.

470-19-81

Data Reporting and Interpretation

- Fully covered in 40 CFR Part 50 Appendix K

470-19-82

Maintenance

- Maintenance Procedures
- Recommended Maintenance Schedules

470-19-83

Recommended Maintenance Procedures

- Dismantling and cleaning impaction inlet as specified by the manufacturer
- Cyclonic inlet
- MFC base
- VFC base
- Refurbishment of HV PM_{10} samplers

470-19-84

Cyclonic Inlet

- Periodically wipe outer tube with disposable wiper.

470-19-85

MFC Base

- Check tubing and power lines.
- Inspect filter screen every sampler recovery day.
- Inspect filter cassette gaskets each time a cassette is loaded.
- Check motor and housing gaskets every 3 months.
- Replace blower motor brushes.

470-19-86

MFC Base
(continued)

- Replace motors as needed.
- Repair or replace recorder as needed.
- Replace recorder pens every 30 recording days.

470-19-87

VFC Base

- Check tubing and power lines.
- Inspect filter screen and throat every sampler recovery day.
- Inspect filter cassette gaskets each time a cassette is loaded.
- Check motor and housing gaskets every 3 months.

470-19-88

VFC Base
(continued)

- Replace blower motor brushes before they become worn.
- Replace motors as needed.

470-19-89

Refurbishment of HV PM_{10} Samplers

- Conduct major repairs or complete refurbishment as needed.
- Leak check and calibrate before resuming field operation.

470-19-90

Auditing Procedures

- Flow Rate Performance Audit for MFC PM_{10} Sampler
- Audit Data Reporting
- Flow Rate Performance Audit Frequency
- Systems Audit

470-19-91

Flow Rate Performance Audit for MFC PM_{10} Sampler

- Transfer audit equipment to field site.
- Instruct operator to install clean filter in the sampler.
- Install the audit orifice transfer standard with no resistance plate.
- Leak test the system.
- Inspect the audit orifice manometer connecting tubing.

470-19-92

Flow Rate Performance Audit for MFC PM_{10} Sampler (continued)

- Open manometer valves and adjust sliding scale.
- Connect audit manometer to pressure port.
- Turn on sampler and allow it to warm up to operating temperature.
- Observe and record sampler location, date, time, sampler model, sampler S/N, calibration relationship, ambient temperature, ambient pressure, unusual weather conditions, and audit orifice transfer standard S/N and calibration information.

470-19-93

Flow Rate Performance Audit for MFC PM_{10} Sampler (continued)

- Record pressure drop across the orifice.
- Instruct operator to read sampler exit orifice manometer reading.
- Turn off sampler, remove audit orifice transfer standard, turn sampler on again, repeat previous step for the normal operating flow rate.
- Collect all audit data and verify that correct readings have been recorded.

470-19-94

Flow Rate Performance Audit for MFC PM_{10} Sampler
(continued)

- Determine flow rate through the audit orifice transfers standard.

$$Q_a\ (\text{audit}) = \{ (\Delta H_2O)\ (T_a / P_a)^{1/2} - b \}\ \{1/m\}$$

470-19-95a

Flow Rate Performance Audit for MFC PM_{10} Sampler
(continued)

Where:

Q_a (audit) = actual volumetric flow rate as indicated by the audit orifice transfer standard, m^3/min

ΔH_2O = pressure drop across the orifice, mm (or in.) H_2O

T_a = ambient temperature, K (K = °C + 273)

P_a = ambient barometric pressure, mm Hg (or kPa)

b = intercept of the audit orifice transfer standard's calibration relationship

m = slope of the audit orifice transfer standard's calibration relationship

470-19-95b

Flow Rate Performance Audit for MFC PM_{10} Sampler
(continued)

- Instruct operator to calculate sampler's inlet flow rate with and without orifice installed.
- Calculate percentage difference between indicated flow rate and corresponding audit flow rate.

$$\text{Audit flow rate \% difference} = 100\ \frac{Q^*\ (\text{sampler}) - Q^*\ (\text{audit})}{Q^*\ (\text{audit})}$$

470-19-96

Flow Rate Performance Audit for MFC PM_{10} Sampler
(continued)

- Record audit flow percentage difference.
- Prior to invalidating any data, double check
  - sampler's calibration.
  - audit orifice transfer standard's certification.
  - all calculations.

470-19-97

Flow Rate Performance Audit for MFC PM_{10} Sampler
(continued)

- Calculate corrected sampler flow rate.

$$Q^*(\text{corrected sampler}) = [Q^*(\text{sampler})]\left[\frac{100 - \text{audit \% difference}}{100}\right]$$

- Calculate design flow rate percentage difference.

$$\text{Design flow-rate \% difference} = \left[\frac{100}{1.13}\right]\left[\frac{Q^*(\text{corrected sampler})}{Q^*}\right] - 1$$

470-19-98

Flow Rate Performance Audit for MFC PM_{10} Sampler
(continued)

- Record the design flow rate differences.
- Prior to invalidating any data, double check
  - audit orifice transfer standard's certification.
  - all calculations.

470-19-99

Audit Data Reporting

- Given to the operating agency at the completion of the audit and discussed as necessary
- Not used to make monitoring system modifications
- Post audit verification of the audit equipment and data essential
- Final audit results submitted to the operating agency as soon as possible

470-19-100

Flow Rate Performance Audit Frequency

- Frequency of flow rate audits depends on the use of the data
- For PSD monitoring, audits must be conducted once per sampler quarter
- For SLAMS monitoring audits conducted on at least 25% of the samplers each quarter
- If < 4 PM_{10} samplers, one or more randomly selected samplers readapted so at least one sampler is audited each quarter

470-19-101

Systems Audit

- Data processing
- Analytical process system

470-19-102

Assessment of Monitoring Data for Precision and Accuracy

- Precision
- Accuracy

470-19-103

Precision

- Requires duplicate collocated sampling sites
- 1 to 5 sites = 1 collocated sampling site
- 6 to 20 sites = 2 collocated sampling sites
- > 20 sites = 3 collocated sampling sites
- Collocated samplers should be the same type of sampler
- Must be within 4 m of each other, at least 2 m apart

470-19-104

Precision
(continued)

- Calibration, sampling, and analysis same for all other samplers in the network
- One sampler is primary sampler, other is duplicate sampler
- Duplicate sampler must be operated concurrently with primary at least once per week
- Data from both sites reported
- Precision calculated per 40 CFR Part 58, Appendix A

470-19-105

Accuracy

- Each quarter audit flow rate at least 25%
- Each sampler audited at least once per year
- Four samplers per reporting organization, randomly audit one sampler per quarter
- Auditing performance of the sampler at its specified flow rate
- Accuracy calculated as described in 40 CFR Part 59, Appendix A

470-19-106

Recommended Standards for Establishing Traceability

- ASTM Class 1, 1.1, or 2 weights
- Positive displacement primary standard
- Elapsed time meter checked semiannually
- Accuracy checks conducted at routine intervals traceable to NIST

470-19-107

Lesson 20

Quality Assurance Procedures for Monitoring PM10 in Ambient Air Using a Dichotomous Sampler

Office of Air Quality Planning and Standards
U.S. Environmental Protection Agency

APTI 470
Quality Assurance for Air Pollution
Measurement Systems

Introduction

Approved EPA PM_{10} Reference Methods

- High-volume (HV) PM_{10} sampler
- Dichotomous sampler

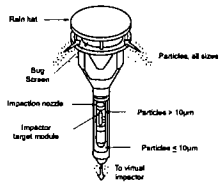
470-20-1

Sampler Basic Components

- Sampler inlet
- Size fractionating virtual impactor
- Flow control system

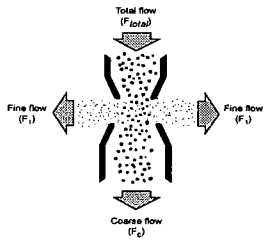
470-20-2

Sampler Inlet



470-20-3

Size Fractionating Virtual Impactor



470-20-4

Procurement of Equipment and Supplies

- Field Operations
- Laboratory Operations

470-20-5

Field Operations

- Dichotomous samplers
- Calibration QA/QC supplies
- QC flow-check device
- Audit equipment

470-20-6

Laboratory Operations

- Filter media
- Filter cassettes
- Filter conditioning
- Filter handling
- Analytical balance
- Mass reference standards

470-20-7

Calibration Procedures

- Overview
- Flow rate measurement and general aspects
- Sampling and analysis equipment calibration requirements
- Recommended standard and associated equipment

470-20-8

Overview

- All sampling and analysis equipment must be properly calibrated and recorded in a calibration logbook, on calculation data sheets, or appropriate recording files.

470-20-9

Flow Rate Measurement and General Aspects

- Critical: The flow rate through the sampler must be maintained at or near the sampler's design flow rates.
- Limits should be within ± 10%.

470-20-10

Sampling and Analysis Equipment Calibration Requirements

- Sampler calibration
- Flow-rate transfer standard
- On/off timer
- Elapsed-time meter
- Analytical balance
- Relative humidity indicator
- Mass reference standards

470-20-11

Sampler Calibration

- Frequency - Upon receipt, after maintenance on sampler, and any time audits or flow checks deviate more than ± 7% from the indicated flow rate or ± 10% from the design flow rate
- Acceptance limits - Indicated flow rate = true flow rate ± 4%
- Action if requirements not met - Recalibrate.

470-20-12

Flow-Rate Transfer Standards

- Frequency - Check upon receipt, at 1 yr intervals against primary standard, or if damaged.
- Acceptance limits - Indicated flow rate from previous calibration = actual flow rate ± 2%.
- Action if requirements not met - Adopt new calibration curve.

470-20-13

On/off timer

- Frequency - Check at purchase, routinely on sample recovery days
- Acceptance limits - ± 30 min / 24 hr
- Action if requirements not met - Adjust or repair.

470-20-14

Elapsed-time Meter

- Frequency - 6-month intervals
- Acceptance limits - ± 2 min / 24 hr
- Action if requirements not met - Adjust or replace.

470-20-15

Analytical Balance

- Frequency - Gravimetric test-weighing at purchase and during periodic calibration checks
- Acceptance limits - Sensitivity = ± 1 µg, Precision = 1 µg
- Action if requirements not met - Replace and/or recalibrate.

470-20-18

Relative Humidity Indicator

- Frequency - 6 month intervals
- Acceptance limits - Indicator reading = psychrometer reading ± 6%
- Action if requirements not met - Adjust or replace.

470-20-17

Mass Reference Standards

- Frequency - Check every 3 to 6 months against laboratory primary standards
- Acceptance limits - Standard's tolerance less than 25 µg, handle with smooth, nonmetallic forceps
- Action if requirements not met - Obtain proper standards or forceps.

470-20-18

Recommended Standards and Associated Equipment

- Total and fine flow rates
- Coarse flow rate

470-20-19

Total and Fine Flow Rates

- Laminar flow element (LFE)
- Mass flowmeter (MFM)
- Dry gas meter (DGM)
- Orifice

470-20-20

Laminar Flow Element (LFE)

- Optimum flow range Q_a - 12.0 to 19.0 L/min
- Equipment - LFE thermometer/barometer, manometer, filters, adapter
- Should have filtered air entering LFE
- Subject to fluctuations due to temperature changes
- Manometer used within its temperature range

470-20-21

Laminar Flow Element (LFE)
(continued)

- Must equilibrate

$$(\Delta H_2 0)\,(CF) = Q_a$$

Where: $\Delta H_2 0$ = pressure drop
CF = correction factor
Q_a = actual flow rate

470-20-22

Mass Flowmeter (MFM)

- Optimum flow range Q_a - 12.0 to 19.0 L/min
- Equipment - MFM, thermometer/barometer, filters, adapter
- Recommended liquid-crystal display (LCD)
- Must equilibrate in ambient conditions
- (Volts) (CF) = Q_{std}

470-20-23

Dry Gas Meter (DGM)

- Optimum flow range Q_a - 12.0 to 19.0 L/min
- Equipment - DGM thermometer/barometer, stopwatch, filters, adapter
- Should time through five revolutions
- Each timing repeated three times

$$\frac{\text{Volume}}{\text{Time}} = Q_a$$

470-20-24

Orifice

- Optimum flow range Q_a - 12.0 to 19.0 L/min
- Equipment - orifice thermometer/barometer, manometer, filters, adapter
- Good only in range $\Delta P \leq 8$ in.

470-20-25

Orifice
(continued)

$$\left[\frac{\Delta H_2O \cdot T_m}{M_m \cdot P_m}\right]^{1/2} (CF) = Q_a$$

Where:
- T_m = upstream absolute pressure
- P_m = upstream absolute pressure
- M_m = molecular weight of gas
- CF = correction factor
- Q_a = actual flow rate

470-20-26

Coarse Flow Rate

- Laminar flow element
- Mass flowmeter
- Dry gas meter
- Orifice
- Soap film flowmeter (SFFM)

470-20-27

Soap Film Flowmeter (SFFM)

- Optimum flow range Q_a - 0 to 2 L/min
- Equipment - SFFM stopwatch, plug with adapter, filters
- Caution - can break easily
- Flow in Q_a
- Three timings
- Flow rate in terms of actual conditions

470-20-28

Soap Film Flowmeter (SFFM)
(continued)

$$\text{Volume} \left[P^* - (1 - RH) P^{H_2O} \right] = Q^*$$

Where: RH = fractional relative humidity

P_{H_2O} = vapor pressure of water at T_a

Q_a = actual flow rate

P_a = average ambient pressure

470-20-29

Sampler Calibration Frequency

- Upon installation
- At least annually
- After repairs that might affect calibration
- Whenever field calibration flow check exceeds QC limits
- Whenever an audit shows sampler to be out of calibration

470-20-30

Field Operations

- Siting Requirements
- Sampler Installation Procedures
- Example Sampling Operations
- Field Calibration Check Procedure
- Documentation

470-20-31

Siting Requirements

- Spatial Scales
- Temporal Scales
- Minimum Site Guidelines
- Other Factors

470-20-32

Spatial Scales

- Range: small scale (0.1 to 0.5 km^2) to large scale (> 1×10^5 km^2)
- Based on potential impact of particulate pollution
- Should reflect expected impact

470-20-33

Temporal Scales

- Geometric mean concentration or 24 hr average concentration
- Consideration given to prevailing wind direction
- Not ideal for 24 hr concentrations

470-20-34

Minimum Site Guidelines

- Unobstructed air flow 2 m in all directions
- Inlet height of 2 to 15 m above ground
- Collocated minimum spacing 2 m, maximum spacing 4m
- Inlet heights within 1 vertical m

470-20-35

Other Factors

- Accessibility for all weather conditions
- Operator's safety
- Adequate electricity 3 to 5 A (120V a.c., 60 Hz)
- Security of monitoring equipment and operating personnel

470-20-36

Sampler Installation Procedures

- Perform visual inspection upon receipt.
- Perform operational check in laboratory.
- Carefully transport to site.
- Install control module and connect vacuum lines.
- Check tubing for crimps and cracks, and plug in power cord.
- Perform a multipoint flow rate calibration.

470-20-37

Example Sampling Operations

- Specific to the Particular Model
- Pre-sampling Procedures
- Filter Installation (General Procedures)
- Filter Recovery
- Sample Validation and Documentation
- Sample Handling

470-20-38

Example
Pre-Sampling Procedures

- Keep filter cassettes in protective petri dishes.
- Discard damaged filters.
- Mark petri dish with filter ID number, sample ID number, sample collection date, type of sample collected.

470-20-39

Example Filter Installation (General Procedures)

- Switch timer off.
- Unscrew (by hand) knurled filter holder assembly.
- Verify O-ring is in each filter holder.
- Insert cassette into appropriate filter holder.
  - Course-particulate filter centerline of the virtual impact head
  - Fine-particle filter holder offset

470-20-40

Example Filter Installation (General Procedures)
(continued)

- Tighten both knurled filter nuts by hand.
- Switch timer to ON.
- Turn on vacuum pump.

470-20-41

Example Filter Installation (General Procedures)
(continued)

- Record following info on sample data sheet:
  - Filter ID number
  - Sampler model number
  - Sampler serial number
  - Sample location
  - TSP
  - CSP
  - AIRS designation
  - Sample collection date

470-20-42

Example Filter Installation (General Procedures)
(continued)

- Set total flow rate.
- Record total vacuum gauge indication.
- Set course flow rate.
- Record course vacuum gauge indication.
- Turn off sampler.

470-20-43

Example Filter Installation (General Procedures)
(continued)

- Set master timer.
- Reset elapsed time to 0.
- Close front cover.
- Visually inspect monitoring site.

470-20-44

Example Filter Recovery

- Record the elapsed-time indicator value.
- Record following information:
  - final total rotameter reading
  - final coarse rotameter reading
  - final total vacuum gauge reading
  - final coarse vacuum gauge reading.
- Turn the sampler off.
- Reverse the filter installation procedure.
- Remove each filter, one at a time.

470-20-45

Example Filter Recovery
(continued)

- Put the filter cassettes in original marked plastic petri dishes.
- Calculate and record the total and coarse average rotameter readings.

470-20-46a

Example Filter Recovery
(continued)

$$\bar{I} = \frac{TSP \text{ or } CSP + IF}{2}$$

Where: $\bar{I}$ = average total or coarse rotameter response, arbitrary units

TSP, CSP = total or coarse rotameter set points, arbitrary units

IF = indicated final total or coarse rotameter response, arbitrary units

470-20-46b

Example Filter Recovery
(continued)

- Record average ambient temperature T_{av} and barometric pressure ~P_{av}.
- Calculate and record the total and coarse average actual flow rates.

470-20-47a

Example Filter Recovery
(continued)

$$\overline{TQ_a} \text{ or } \overline{CQ_a} = 1/m \{(\bar{I}(T_{av}/P_{av})^{1/2} - b\}$$

Where: $\overline{TQa}$, $\overline{CQa}$ = sampler total or coarse average flow rate, actual L/min

$\bar{I}$ = average total or coarse rotameter response, arbitrary units

T_{av} = average ambient temperature, K

P_{av} = average ambient pressure, mm Hg or kPa

m = slope of the dichotomous sampler total or coarse calibration relationship

b = intercept of the dichotomous sampler total or coarse calibration relationship

470-20-47b

Example Filter Recovery
(continued)

- Calculate the actual fine flow rate.
- Record calculations.
- Observe conditions around monitoring site.

470-20-48

Sample Validation and Documentation

- Timing
  - ON or OFF within 1/2 hr of midnight
  - Must operate ≥ 23 hr and ≤ 25 hr
- Flow rates
  - Total: 16.7 L/min ± 10%
  - Course: 1.67 L/min ± 10%
- Filter quality - damaged filter invalidated

470-20-49

Sample Handling

- Calculate the total, coarse, and fine flow rate and enter on data sheet.
- Valid sample - Promptly deliver in protective petri dish.
- Invalid sample -
  - Mark "VOID."
  - Do not discard the filter.
  - Promptly deliver to analytical laboratory.
- Questionable sample -
  - Complete as much of data sheet possible.
  - Record as "Questionable."
  - Promptly deliver to analytical laboratory.

470-20-50

Example QC Field Calibration Check Procedure

- Insert clean fine and coarse filters.
- Turn on the sampler to warm up.
- Read and record:
  - ambient temperature (T_a), °C and K.
  - ambient barometric pressure (P_a), mm Hg and kPa.
  - sampler S/N and model.
  - orifice S/Ns and calibration relationships.
  - date, location, and operator's signature.
  - sampler rotameter's target flow rates and target set points.

470-20-51

Example QC Field Calibration Check Procedure
(continued)

- Adjust total and coarse rotameters.
- Remove inlet, replace with flow check orifice device, recheck the rotameter set point.
- Observe the ΔH_2O.
- Determine corresponding flow rate.
- Record manometer deflection value and corresponding flow rate.

470-20-52

Example QC Field Calibration Check Procedure
(continued)

- Calculate total actual flow rate (TQ_a) and record.
- Turn the sampler off.
- Disconnect fine flow vacuum line.
- Install the coarse flow rate orifice.
- Turn the sampler on.
- Observe the ΔH_2O.
- Determine corresponding flow rate.

470-20-53

Example QC Field Calibration Check Procedure
(continued)

- Record manometer deflection value and corresponding flow rate.
- Calculate indicated coarse actual flow rate (CQ_a).
- Calculate the QC percentage difference.

$$\text{QC\% Difference} = 100 \left| \frac{(\text{Sampler TQ}^a \text{ or CQ}^a) - \text{Orifice flow rate}}{\text{Orifice flow rate}} \right|$$

470-20-54

Example
Field Calibration Procedure
(continued)

- Turn off sampler, remove orifice device, replace the inlet, reconnect the fine flow vacuum line.
- Remove filters from fine and coarse filter holders.
- Set up the sampler for the next sampling period.

470-20-55

Documentation

- Operator who starts the sample
- Operator who removes the sample

470-20-58

Filter Preparation and Analysis

- Filter Handling
- Filter Integrity Check
- Filter Equilibration
- Initial Weighing Procedures
- Internal QC
- Post Sampling Documentation and Inspection
- Final Weighing Procedure
- Calculation of Net Mass Filter Loading

470-20-57

Filter Handling

- Use nonserrated forceps, nylon gloves.
- Place filters in petri dishes.
- Number sequentially.

470-20-58

Filter Integrity Check

Visually check each filter for:

- pinholes.
- separation of filter ring.
- chaff or flashing.
- loose material.
- discoloration.
- other imperfections.

470-20-59

Filter Equilibration

- 24 hours before weighing
- Relative humidity between 20 and 45%
- Temperature between 15 and 30°C ± 3°C
- Filter conditioned in petri dishes

470-20-60

Initial Weighing Procedures

- Warm up for five minutes.
- Begin with zero balance.
- QC supervisor performs "standard" filter QC check.
- Weigh filter.
- Place tared filter in petri dish.
- Record assigned filter ID number.

470-20-61

Internal QC

- Analyst QC
- Supervisory QC procedure

470-20-62

Post Sampling Documentation and Inspection

- Examine data sheet.
- Examine petri dish for dislodged material.
- Recover any material dislodged from filter.
- Match filter ID with correct laboratory data coding form and group filters according to recorded balance ID numbers.
- Examine filters for damage.

470-20-63

Post Sampling Documentation and Inspection
(continued)

- Reject filters if defects are found.
- Return defective filters to original petri dish and submit to laboratory supervisor.

470-20-64

Final Weighing Procedure

- Group filters.
- Open petri dish.
- Cover open dish.
- Repeat filter tare weighing procedure.
- Ensure validity of reweighing.
- Record gross weight.

470-20-65

Final Weighing Procedure
(continued)

- Archive filter.
- If analyzing further, return filter to petri dish and place asterisk on laboratory data / coding form.
- Forward to laboratory for further analysis.

470-20-66

Calculation of Net Mass Filter Loading

- Gross weight minus the tare weight is the net mass of the particulate.

$$Mf \text{ or } Mc = W_g - W_t$$

Where: Mf, Mc = fine or coarse particulate net mass, mg
W_g = gross weight, mg
W_t = tare weight, mg

- Each calculation must be independently validated.

470-20-67

Calculation, Validations, and Reporting

- Calculations Using a Dichotomous Sampler
- Calculation Validation
- Data Reporting and Interpretation

470-20-68

Calculations Using a Dichotomous Sampler

- Flow rate calculations
- PM_{10} concentration calculation

470-20-69

Flow Rate Calculations

- Determine average total or coarse rotameter response.

$$\bar{I} = \frac{TSP \text{ or } CSP + IF}{2}$$

Where:

$\bar{I}$ = average total or coarse rotameter response, arbitrary units

TSP, CSP = total or coarse rotameter set points, arbitrary units

IF = indicated final total or coarse rotameter response, arbitrary units

470-20-70

Flow Rate Calculations

$$\overline{TQ_a}, \overline{CQ_a} = 1/m \left[\bar{I} \left(T_{av}/P_{av} \right)^{1/2} - b \right]$$

Where:

$\overline{TQ_a}, \overline{CQ_a}$ = sampler total or coarse average flow rate, actual L/min

$\bar{I}$ = average total or coarse rotameter response, arbitrary units

T_{av} = average ambient temperature, K

P_{av} = average ambient pressure, mm Hg or kPa

m = slope of the dichotomous sampler total or coarse calibration relationship

b = intercept of the dichotomous sampler total or coarse calibration relationship

470-20-71

PM_{10} Concentration Calculation

- Determine total volume sampled.

$$V = \left(\overline{TQ_a}\right) t$$

Where:

V = total sample volume, m^3

$\overline{TQ_a}$ = total flow rate corrected to standard conditions, m^3/min

t = elapsed total sampling time, min

470-20-72

PM_{10} Concentration Calculation

- Determine mass concentration.

$$PM_{10} = \frac{(M_f + M_c)10^3}{V}$$

Where:

PM_{10} = mass concentration of PM_{10}, $\mu g/m^3$

M_f = net mass of particulate of the fine filter, mg

M_c = net mass of particulate of the coarse filter, mg

10^6 = conversion factor for mg to µg and L to m^3

V = total sample volume, L

470-20-73

Calculation Validation

- Collect total sample time and average total flow rate data.
- Compute the total mass concentration for seven samples per 100.
- If calculation errors, all values in that sample lot should be recalculated.

470-20-74

Calculation Validation
(continued)

- Scan all total mass concentration values.
- Recompute the total mass concentrations.
- Correct any errors that are found.
- If mass concentration computations appear correct, and exceedingly high or low values still exist, review all raw data for completeness and correctness.

470-20-75

Maintenance

- Maintenance Procedures
- Refurbishing Dichotomous Samplers

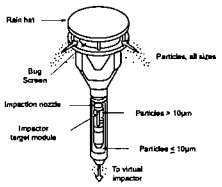
470-20-76

Maintenance Procedures Supplies

- Alcohol-based general-purpose cleaner
- Cotton swabs
- Small soft-bristle brush
- Paper towels
- Distilled water
- Miscellaneous hand tools
- Compressed air source (recommended, not required)

470-20-77

Sampler Inlet



470-20-78

Sampler Inlet
(continued)

- Mark "match marks."
- Dissassemble using manufacturer's instructions.
- Clean all interior surfaces.
- Reassemble using "match marks."

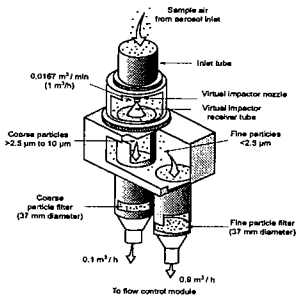
470-20-79

Virtual Impactor Assembly

- Inspect inlet tube every 3 to 4 months.
- Inspect and clean all remaining inner surfaces every 6 to 12 months.
- Use alcohol or water and soft-bristle brush for cleaning.
- Examine sample module vacuum tubing periodically.
- Examine connecting fittings for cross-threading.

470-20-80

Virtual Impactor Assembly



470-20-81

Control Module Cleaning Procedures

- Remove or open the front panel.
- Wipe down all surfaces.
- Take action to correct any obvious problems before completion of cleaning.
- Check rotameters for cleanliness.
- Remove and clean all filter jars.

470-20-82

Control Module Cleaning Procedures
(continued)

- Clean or replace any dirty filter elements.
- Clean the cooling fan blades.
- Clean exterior surfaces of the vacuum pump.
- Check all mounting brackets.

470-20-83

Vacuum Pump

- Diaphragm and flapper valves replaced routinely (at 1 year intervals) or if sudden reduction in sampler vacuum occurs

470-20-84

Refurbishing Dichotomous Samplers

- Refurbish after extended period of field operation.
- Refer to manufacturer's instructions.
- Leak check and calibrate before resuming field operation.

470-20-85

Auditing Procedures

- Audit Guidelines
- Types of Audits

470-20-86

Audit Guidelines

- No special preparation
- Conducted by another individual with thorough knowledge, not by routine operator
- Uses transfer standards that are completely independent of those used for routine calibration and QC flow checks
- Audit documentation information

470-20-87

Audit Documentation Information

Includes:

- Audit transfer standards and traceability
- Types of instruments, model and serial numbers
- Calibration information
- Collected audit data

470-20-88

Types of Audits

- Flow Rate Performance Audit
- Systems Audit of Data Processing
- Analytical Process System Evaluation

470-20-89

Flow Rate Performance Audit

- Audit Apparatus
- Total Flow Rate Audit Procedures
- Fine Flow Rate Audit Procedures
- Coarse Flow Rate Audit Procedures
- Audit Data Calculations
- Performance Audit Frequency

470-20-90

Audit Apparatus

- Any flow rate transfer device is acceptable as a routine calibrator for a dichotomous sampler.
- Transfer device must be different from one used to calibrate sampler.
- Audit device must be calibrated with a primary standard.
- Document audit information.

470-20-91

Document Audit Information

- Sampler and audit transfer standard type
- Model and serial numbers
- Transfer standard traceability and calibration information
- Ambient temperature and pressure conditions
- Collected audit data

470-20-92

Total Flow Rate Audit Procedures

- Install new filters in fine and coarse filter holders.
- Adjust the rotameter flow control to set the total and coarse rotameters to their operational set points for routine sampling.
- Allow the sampler to warm up.
- Complete data sheet with required information, ambient temperature (T_a), ambient barometric pressure (P_a), TSP and CSP values and corresponding flow rates.

470-20-93

Total Flow Rate Audit Procedures
(continued)

- Remove the sampler inlet and replace with transfer standard adaptive device.
- Connect the adapter to the transfer standard outlet.
- Recheck rotameter settings.
- Record TS readings (volts, ΔH_2O, timings, etc.).

470-20-94

Fine Flow Rate Audit Procedures

- Turn the sampler off and disconnect the coarse-flow 6.53 mm (1/4 in.) line.
- Cap the course flow outlet port.
- Turn the sampler on.
- Check the rotameter set points, record the total and coarse rotameter units and corresponding flow rate values.
- Record TS readings (volts, ΔH_2O, timings, etc.).

470-20-95

Coarse Flow Rate Audit Procedures

- Turn the sampler off and exchange the total and fine flow-rate transfer standard for the coarse flow transfer standard.
- Reconnect the coarse flow line and disconnect the fine flow line, cap the fine flow outlet port.
- Turn the sampler on.

470-20-96

Coarse Flow Rate Audit Procedures
(continued)

- Check rotameter set points, record the total and coarse rotameter units and their corresponding flow rate values.
- Record on TS readings (volts, ΔH_2O, timings, etc.).

470-20-97

Audit Data Calculations

- Calculate and record the audit total, fine, and coarse flow rates using the calibration curve.
- Correct audit flow rates to actual conditions.

470-20-98a

Audit Data Calculations
(continued)

$$Q_a = Q_{std} (T_a/P_a) (P_{std}/T_{std})$$

Where:
- Q_a = flow rate at actual conditions, L/min
- Q_{std} = flow rate corrected to standard temp and pressure, L/min
- T_a = ambient temp, K
- P_a = ambient pressure, mm Hg or kPa
- P_{std}, T_{std} = standard pressure and temperature

470-20-98b

Audit Data Calculations
(continued)

- Calculate the corresponding sampler flow rates and record.
- Determine the flow rate percentage difference.

$$\%\,\text{Difference} = \left|100\left[\frac{Q_a\,(\text{sampler}) - Q_a\,(\text{audit})}{Q_a\,(\text{audit})}\right]\right|$$

470-20-98c

Audit Data Calculations
(continued)

- Record percent difference.
- Before leaving site, make a comparison between flows.

470-20-98d

Performance Audit Frequency

- PSD monitoring requires audits once per quarter.
- SLAMS requires audits on 25% of samplers per network per quarter.

470-20-99

Systems Audit of Data Processing

- General Considerations
- Audit Procedures

470-20-100

General Considerations

- Systems audit conducted as soon as possible after the original calculations
- Minimum frequency of 7 samples per 100 - Recommended
- Minimum of 4 per lot - Recommended

470-20-101

Audit Procedures

- Use the operational flow rates.
- Independently compute the concentration.
- Compare it with the corresponding concentration originally reported.
- Record the audit values on a data sheet and report them, along with the original values.

470-20-102

Analytical Process System Evaluation

- General Considerations
- Procedures

470-20-103

General Considerations

- ASTM Class 1 standard weights
- Should not be operated by inexperienced personnel

470-20-104

Procedures

- Review the maintenance and calibration log.
- Review QC data records for the filter-weighing process.
- Have the balance operator randomly reweigh filters.
- Calculate the weight difference for each filter.

470-20-105

Review the Maintenance and Calibration Log

- Routine balance maintenance and calibrations are performed by the manufacturer's service representative at manufacturer-specified scheduled intervals.
- Calibration intervals should not exceed 1 year.

470-20-106

Review QC Data Records for the Filter-Weighing Process

- Zero and calibration checks after every 5 filter weighings
- Standard filter weighing every day of the balance operation
- Duplicate filter weighing for every five to seven filters
- If QC checks out of limits, note action taken

470-20-107

Have the Balance Operator Randomly Reweigh Filters

- Groups ≤ 50: 4 filters out of every group
- Groups of ≥ 50 and ≤ 100: 7 from each group

470-20-108

Calculate Weight Difference For Each Filter

- Difference = Original weight (mg) - Audit weight (mg)
- For unexposed filters, difference should be less than ± 20 µg
- For exposed filter, potential loss of volatile particles prohibits acceptance / rejection limits

470-20-109

Assessment of Monitoring Data for Precision and Accuracy

- Precision
- Accuracy

470-20-110

Precision

- Requires duplicate collocated sampling sites
- Number of collocated samplers
  - 1 to 5 sites = 1 site
  - 6 to 20 sites = 2 sites
  - More than 20 = 3 sites
- Same type of sampler
- Within 4 m of each other, at least 2 m apart
- Calibration, sampling, and analysis must be the same

470-20-111

Precision
(continued)

- One sampler designated as primary sampler, other designated as duplicate sampler
- Duplicate sampler must be operated concurrently with its primary sampler at least once per week
- Data from both sites are reported
- Percentage difference between the two samplers used to calculate precision as per 40 CFR Part 58, Appendix A

470-20-112

Accuracy

- Each quarter, audit flow rate of 25% of the samplers
- Each sampler audited at least once per year
- If fewer than four samplers per reporting organization, randomly audit one or more samplers so that one sampler is audited per quarter

470-20-113

Accuracy
(continued)

- Accuracy assessed by auditing performance of sampler
- Percentage difference between the flow rates is used to calculate accuracy as described in 40 CFR Part 58, Appendix A

470-20-114

Recommended Standards for Establishing Traceability

- ASTM Class 1, 1.1, or 2 weights for laboratory microbalance
- Positive displacement primary standard or laminar flow element for calibrating flow rate transfer standard
- Positive displacement primary standard for calibrating the transfer standard

470-20-115

Recommended Standards for Establishing Traceability
(continued)

- Elapsed time meter checked semiannually to within 15 min/day
- Accuracy checks of thermometers, barometers, stopwatches, etc., conducted at routine intervals and against standards of known accuracy and traceable to NIST

470-20-116