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.

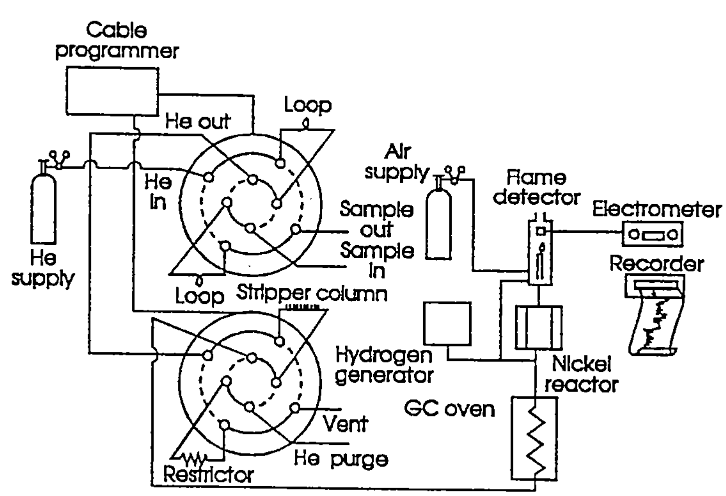


Figure 6A-1. Continuous Analyzer For Cyclolehmdone

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 x 10^{-12} to 1 x 10^{-7} amperes is recommended. For ambient air analysis, a logarithmic amplifier system set to amplify signals between 1 x 10^{-11} and 1 x 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

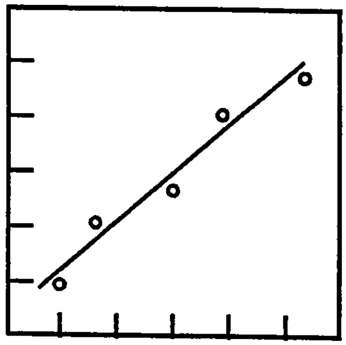
For most applications, the peak height of cyclolehmdone is adequate to quantify the concentration of this gas in an unknown air sample. An automatic electronic integrator can be used for quantification.

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?

470-7-2
6-30-93

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)?

470-7-3
6-30-93

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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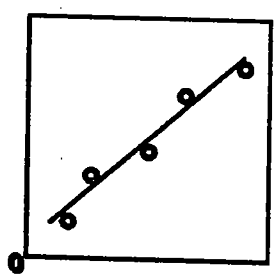

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Table III. Recommended control charts and limits for state and local agencies.

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	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
TSP (flow rate)	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

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^{33}
Temporal continuity	
Constant output	Value + 10^{23}
Successive difference	Being implemented
B. Interstation	
Meteorological network uniformity	Value + 10^{16}
Statistical outliers	
Dixon Ratio	Value + 10^{-20}
III. A Posteriori	
Review of station log	Invalidate 10^{38}
Unusual events or conditions	Validate - keep 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	$NO - NO_x \leq$ Noise (NO)
Oxides of Nitrogen	0 ppm	5 ppm	$NO - NO_x \pm$ Noise (NO_2)
Carbon Monoxide	0 ppm	50 ppm	
Methane	0 ppm	50 ppm	CH_4 - THC $\leq$ Noise (CH_4)
Total Hydrocarbons	0 ppm	50 ppm	CH_4 - THC $\leq$ Noise (THC)
Sulfur Dioxide	0 ppm	1 ppm	SO_2 - TS $\leq$ Noise (SO_2)
Total Sulfur	0 ppm	1 ppm	SO_2 - TS $\pm$ Noise (TS)
Hydrogen Sulfide	0 ppm	1 ppm	H_2S - TS $\leq$ Noise (H_2S)
Aerosol Scatter	$0.000001 m^{-1}$	$0.00099 m^{-1}$	
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.

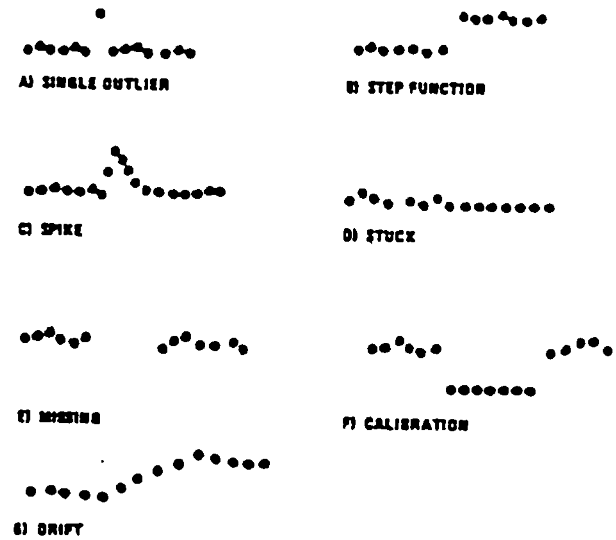

Figure 1. Irregular instrument response.

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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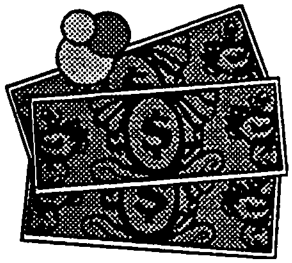
Lesson 16

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?

470-16-2
6-30-93

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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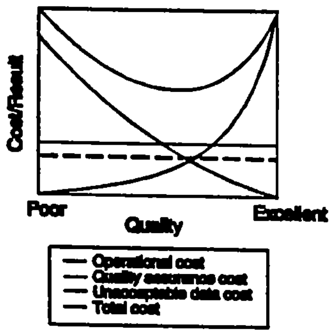
Quality Pays



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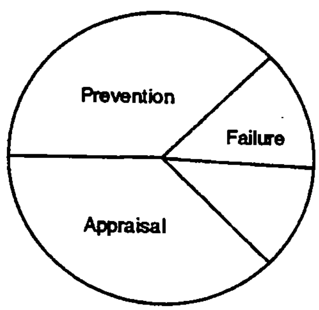
Quality-Related Costs



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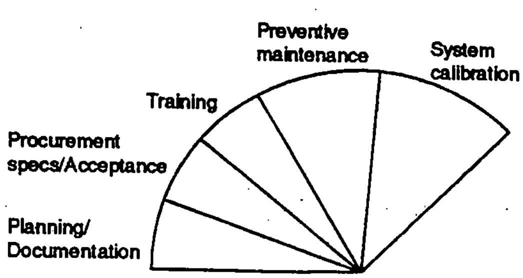
Prevention Cost Groups



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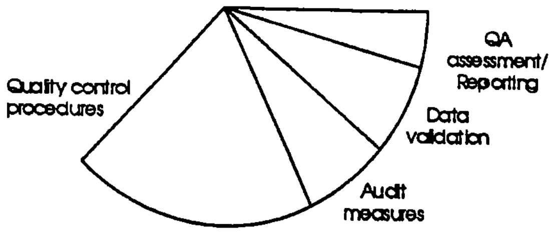
Appraisal Cost Groups



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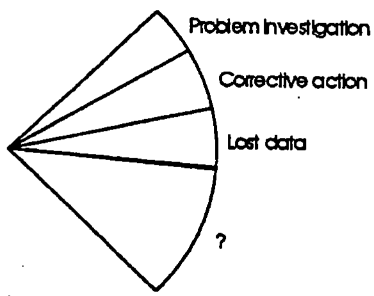
Failure Cost Groups



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Accumulation of Costs

- Lost data costs
- Other costs

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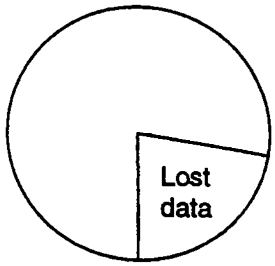
$$F_d = f \times B$$



Where:

F_d = lost data cost

f = % lost data

B = part of network budget associated with lost data

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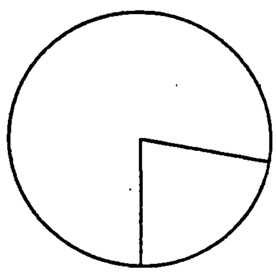
Prorate Personnel Salaries



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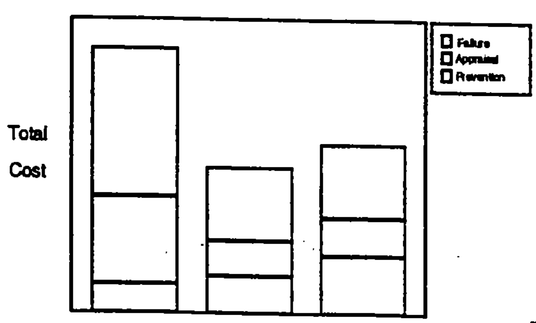
Cost Effectiveness



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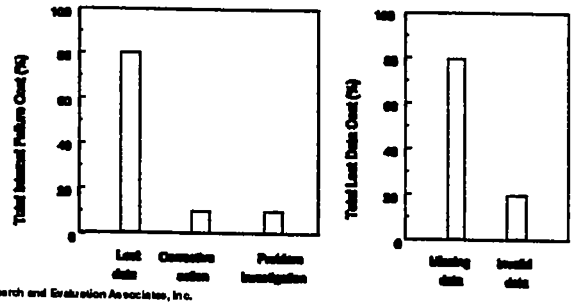
Pareto Analysis of Quality Cost Data



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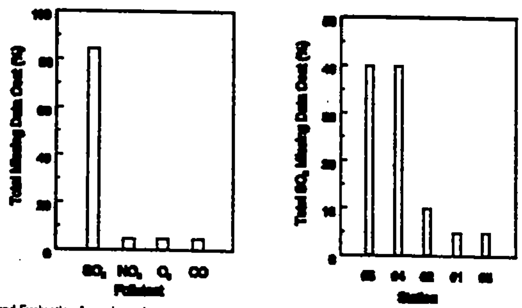
Pareto Analysis of Quality Cost Data (cont.)



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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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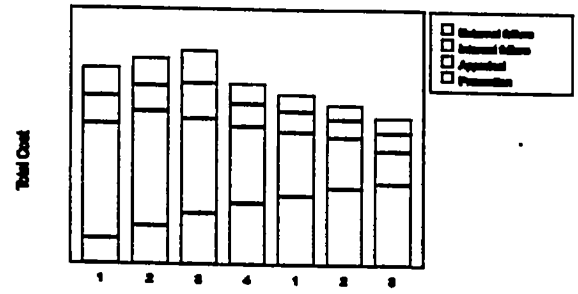
Quality Cost Trend Chart



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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

Ronald B. Strong
Research Triangle Institute

J. Harold White
Research Triangle Institute

Franklin Smith
Research Triangle Institute

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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.)

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.

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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.

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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.

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Temperature Calibration Standards

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

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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.

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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.

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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.

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Calibration of Sampler Pressure Sensors

- General Requirements
- Calibration Procedure

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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.

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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.

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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.

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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.

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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.

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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.

470-17-75

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

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Filter Blanks

- Lot blanks
- Laboratory blanks
- Field blanks

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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

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.

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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.

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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.

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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