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Standard Guide for Laboratories Engaged in Sampling and Analysis of Atmospheres and Emissions[1]

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ε[1] NOTE—Reference to Guide E 548 was editorially removed. As a result, Annex A1 was deleted and the definitions were transferred to the Terminology section. Annex A2 was editorially changed to Appendix X1. These changes were made April 2002.

INTRODUCTION

The utilization of well tested and uniform laboratory practices is essential to the production of reliable and defensible environmental data whose validity can be demonstrated at a later date through the use of written field and laboratory records. This document is intended to provide general guidelines for the elements of laboratory practices that are considered to be basic to the performance of laboratories that provide services in the sampling and analysis of atmospheres and emissions. This document is intended to stimulate an awareness of good laboratory and field practices.

1. Scope

1.1 This guide covers criteria to be used by those responsible for the selection, evaluation, operation, and control of laboratory organizations engaged in sampling and analysis of environmental atmospheres, including ambient, work space, and source emissions (stack gases), as well as atmospheric deposition samples.

1.2 This guide presents features of organizations, facilities, resources, and operations which by their selection and control affect the reliability and credibility of the data generated.

1.3 This guide presents the criteria for the selection and control of the features listed in 1.2 so that acceptable performance may be attained and sustained. Also, this guide presents recommendations for the correction of unacceptable performance.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[2]

D 1357 Practice for Planning the Sampling of the Ambient Atmosphere[2]

D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D-19 on Water[3]

D 3249 Practice for General Ambient Air Analyzer Procedures[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, see Terminology D 1356.

3.2 Other terms are defined as follows:

3.3 *accrediting authority*—a body that evaluates the capability of a testing agency, or an inspection agency, or both, in certain specific fields of activity.

3.4 *agency*—an organization or part of an organization, engaged in the activities of testing or inspection, or both.

3.5 *generic criteria*—common characteristics pertaining to organization, human resources, material resources, and quality systems which provide a basis for assessing the qualifications of testing or inspection agencies.

3.6 *human resources*—those elements of support or capability that are provided by humans using their mental and physical capabilities.

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[2] *Annual Book of ASTM Standards*, Vol 11.03.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

3.7 *inspection*—the process of measuring, examining, testing, gaging, or otherwise evaluating materials, products, services, systems, or environments.

3.8 *organizational component*—a portion of an organization with specific tasks and activities that constitutes a part of the total effort and accomplishment of the organization.

3.9 *quality*—the totality of features and characteristics of a product or service that bear on its ability to satisfy a given need.

3.10 *quality assurance*—a system of activities whose purpose is to provide assurance that the overall quality control job is in fact being done effectively. The system involves a continuing evaluation of the adequacy and effectiveness of the overall quality control program (see *quality control*) with a view to having corrective measures initiated where necessary. For a specific product or service, this involves verifications, audits, and the evaluation of the quality factors that affect the specification, production, inspection, and use of the product or service.

3.11 *quality control*—the overall system of activities whose purpose is to provide a quality of product or service that meets the needs of users; also, the use of such a system. The aim of quality control is to provide quality that is satisfactory, adequate, dependable, and economic. The overall system involves integrating the quality aspects of several related steps including: (*1*) the proper specification of what is wanted; (*2*) production to meet the full intent of the specification; (*3*) inspection to determine whether the resulting product or service is in accord with the specifications; and (*4*) review of usage to provide for revision of specification.

3.12 *testing*—the determination by technical means of properties, performance, or elements of materials, products, services, systems, or environments which involve application of established scientific principles and procedures.

4. Summary of Guide

4.1 This guide describes the criteria, practices, and recommendations for the physical resources, data validation, and mode of operation of the laboratory.

5. Significance and Use

5.1 Data on the composition and characteristics of environmental atmospheres, such as ambient air, work space air, and stack gas emissions, are frequently used to evaluate the health and safety of humans. Data on the composition of atmospheric deposition samples are often used for environmental impact assessment.

5.2 These data are frequently used to ascertain compliance with regulatory statutes that place limits on acceptable compositions and characteristics of these atmospheres.

5.3 Laboratories that produce environmental sampling and analysis data and those who have the responsibility of selecting a laboratory to perform air quality studies need to know what criteria, practices, and recommendations have been accepted by consensus within this field of endeavor.

5.4 Demonstration and documentation by a laboratory that there is judicious selection and control of organizational factors, facilities, resources, and operations enhance the reliability of the data produced and promote the acceptance of these data.

6. Responsibilities and Duties of the Laboratory

6.1 The purpose of the laboratory is to provide information that is factual, accurate, reliable, and adequate for its purpose. The procedure by which this is to be achieved is by the effective administration of a quality assurance (QA) plan by the management of the organization. The elements of a quality assurance plan are described in 6.1.1-6.1.6.1.

6.1.1 *Organization*—A table of organization which indicates the organizational structure and the lines of authority, areas of responsibility, and job descriptions should be available. Key personnel, including their workplace locations and phone numbers, should be identified for each organizational entity. Separate organizational charts for subcontractors might also be needed. QA managers should be identified along with their relationships to other project personnel. The QA managers should be organizationally independent of project management so that the risk of conflict of interest is minimized.

6.1.1.1 *Human Resources*—The key personnel of the organization should be described by means of personal résumés presenting the applicable education and work experience relative to his or her position in the table of organization and the requirements of that position.

6.1.1.2 *Physical Resources*—The laboratory facilities should provide a working environment that is clean, air-conditioned, heated, well-lighted, and safe. The instrumentation and equipment should be appropriate to the operational needs of the laboratory.

6.1.2 *Methodology*—Written procedures should be readily available to all personnel.

6.1.2.1 Sample collection and handling procedures, and storage requirements should be written.

6.1.2.2 Calibration and standardization procedures should be written.

6.1.2.3 Standard Operating Procedures (SOPs) and analytical methods should be written.

6.1.2.4 There should be a document control system to assure that the written procedures are current and complete.

6.1.2.5 All of the above should be periodically subjected to performance and system audits.

6.1.3 *Metrology Systems*—All systems for making measurements should have the following features:

6.1.3.1 Calibration and standardization procedures, including a description of a procedure for establishing traceability, description of calibration standards, and a schedule for calibration,

6.1.3.2 Preventative maintenance procedures including a schedule for maintenance intervals and documentation of their proper completion, and

6.1.3.3 Records of modification of configuration that may occur in any measurement system due to repair and servicing of equipment, replacement of components or reagents, or change of procedures.

6.1.4 *Data Recording*—The laboratory should keep records of submitted samples and completed analyses in a manner that provides for the retrievability, preservation and traceability of

the sample source, the procedures used, and the person or persons responsible for the sampling and analysis.

6.1.4.1 All laboratory data sheets should be dated and signed by the analyst.

6.1.4.2 A policy for the use of computers for data acquisition, archiving, and mathematical calculations should be implemented.

6.1.5 *Data Validation*—The laboratory should keep records of analytical performance by means of audit procedures, reference sample programs, and interlaboratory tests. Where applicable, quality control charts should be used to report results from these validation activities. Quality control procedures found in most current methods should be followed. **(1)**[4]

6.1.6 *Deficiency Correction*—The organizational system should provide the authority and the responsibility for a designated person or persons to investigate out of control procedures and to inform the laboratory management of the problems that occur. This is often the responsibility of the QA manager.

6.1.6.1 A current log should be maintained of such deficiencies and the action taken to correct them.

7. Organization

7.1 The production of reliable data is dependent upon the conscientious effort of everyone who has any involvement with the service. Therefore, it is important that each member of the organization have a clear-cut understanding of his or her duties and responsibilities, and their relationship to the total effort. The management of the laboratory has a prime responsibility in defining the policy goals in relation to the quality of performance and assigning the specific areas of responsibility to the individual. The human resources that are required for the operation of the laboratory will vary with the specific functions that are to be served, but the minimum personnel and their qualifications should generally be as follows:

7.2 *Human Resources* **(2)**

7.2.1 *The Director*—The laboratory director should be a full-time employee of the organization that operates the laboratory. He or she should have an earned baccalaureate degree in science or engineering from an accredited college or university or the equivalent (see Note 1) with a minimum of 5 years experience in sampling and analysis of atmospheres or in a related field. The director should have the following responsibilities:

7.2.1.1 Selection and approval of methods of sampling and analysis,

7.2.1.2 Implementation of a quality assurance program to describe the quality of technical data,

7.2.1.3 Development of standards of performance and evaluation of personnel by these standards, and

7.2.1.4 Training of personnel.

NOTE 1—The *equivalent* requirement is for the purpose of recognizing those persons who may have a comparable educational background that has been obtained through recognized and qualified educational resources but does not result in the award of a baccalaureate degree. The use of this term will necessarily require the judgement of the user of this guide. Certification by acknowledged professional boards is encouraged.

7.2.2 *The Laboratory Supervisor*—The laboratory supervisor should be a full time employee of the organization that operates the laboratory, and should have a minimum of an earned baccalaureate degree in science or engineering from an accredited college, university, or the equivalent (see Note 1), and a minium of one year analytical responsibility.

7.2.3 *The Senior Staff*—The senior staff of the laboratory should conduct the difficult and nonroutine sampling and analyses and should directly supervise the technical staff. Each member of the senior staff should have a baccalaureate degree in science or engineering from an accredited college or university or the equivalent (see Note 1).

7.2.4 *The Technical Staff*—The technical staff will normally consist of qualified personnel who conduct routine sampling and analyses and may also include highly trained and qualified people who specialize in difficult procedures.

7.2.4.1 Each member of the technical staff should have formal, on-the-job training in the analyses and areas of assigned responsibility. Training should be provided on-site, and in many cases should be supplemented by short courses offered by equipment manufacturers, professional organizations, universities, or other qualified training facilities.

7.2.4.2 After appropriate training, the staff member must demonstrate acceptable results in the analysis of an applicable quality control or performance evaluation sample.

7.2.5 *The Support Staff*—The support staff will normally consist of personnel who perform routine services such as: cleaning glassware, transportation and handling samples and equipment, maintenance of sampling equipment, and clerical and secretarial services.

7.2.5.1 Each member of the support staff should have sufficient on-the-job training for his or her level of responsibility as defined by the laboratory director.

7.3 *Physical Resources*

7.3.1 The laboratory environment can affect the results of analyses which are intended to describe the character of atmospheres and emissions; therefore, the laboratory facility should be carefully considered.

7.3.2 The specific items of equipment and apparatus that are needed for the performance of standard methods are described in those standards. If the laboratory proposes to perform a procedure, it should possess the specified items of equipment and apparatus.

7.3.3 The laboratory should be kept as free from interference as is necessary to avoid contamination of the samples. This may require such precautions as sweeping the floor carefully with a compound to suppress dust, periodically coating the floor with an inert material, vacuuming or scrubbing walls, floors, benches, and equipment, and wearing lint-free laboratory clothing. Care should be taken to avoid introducing potential contaminants when choosing cleaning products.

7.3.4 Samples that have been protected against the laboratory environment should be compared against samples that have been exposed to the laboratory environment.

[4] The boldface numbers in parentheses refer to the references at the end of this standard.

7.3.5 In order for the hoods to be effective in removing noxious, harmful, or interfering fumes and aerosols from the laboratory environment, the hoods must be operating at their designed capacity. They should not be located in areas of countervailing winds, such as between two open doors. Under usual operating conditions, hoods will require from 0.0236 m^3/s to 0.059 m^3/s (50–125 ft^2/min) per 0.093 m^3 of face area. Face velocities should be checked routinely by qualified personnel for compliance with specifications.

7.3.5.1 For a more detailed treatment of ventilation consult Ref. **(3)**.

7.3.6 In order to minimize the generation of noxious, harmful, or interfering fumes in the laboratory environment, potentially troublesome samples and reagents should be handled in properly operating hoods. Sinks should not be used for some sample or reagent disposal. The disposal area should be well separated from the laboratory area and meet applicable safety standards. The specific disposal methods are not covered in this guide. In some standards the disposal method for the reagent and sample will be defined. When this information is not supplied in the standard, useful information may be obtained from guides to control hazardous chemical spills, and manuals of laboratory safety which are available from various laboratory supply firms (see Ref. **(4)**). Disposal to the municipal sewers should be in accordance with applicable local, state, and federal regulations.

7.3.7 The mix-up of samples and the cross-contamination hazards are more easily avoided when there is adequate bench space or working area per analyst. As a general guideline there should be 150 to 300 ft^2 per analyst or 12 to 24 lineal ft of bench space per analyst. The space requirement per analyst depends upon the equipment or apparatus that is being used, the number of samples the analyst is expected to handle at any one time, and the number of operations that are to be performed by a single analyst. The laboratory may also have a requirement for specialized facilities, such as a perchloric acid hood. The lighting level may vary from 50 to 100 fc (538 to 1076 lx) depending upon the tasks being performed in the area. **(5)**

7.3.8 The reliability of the instruments is sometimes affected by electrical supply factors including voltage, frequency, and electrical fields. Some instruments may require a separate grounded circuit or a regulated power supply for stable operation. Such a supply is recommended as a good laboratory practice. A battery powered back-up supply is recommended, especially for computer controlled equipment and data acquisition devices.

7.3.9 The laboratory should be supplied with the following: *1)* Class ABC type fire extinguishers, *2)* spill control materials for acids, bases, and flammable materials, *3)* eye wash and safety shower facilities, *4)* eye protection, and other safety devices that may be consistent with the particular laboratory operation. The facility should provide for the safe disposal of reagents and samples with written instructions for the use of these procedures by utility or support personnel. The laboratory may be required to have an EPA waste generator number for compliance with the Resource Conservation and Recovery Act. **(6)**

7.3.10 In general, the physical conditions in the laboratory shall comply with applicable requirements of the Occupational Safety and Health Act. **(7)**

8. Methodology

8.1 The use of written procedures that should be periodically reviewed is essential to the systematic performance of operations. Procedures have a tendency to undergo a process of evolution over a period of time. Modifications of the procedures may be needed periodically and they should be noted. Such notations should be explicit, dated, and signed with the initials of the person responsible.

8.2 Although the laboratory should maintain a library of references for its methodology, it is inconvenient to use a reference book at the bench. Therefore, it is desirable to establish standard operating procedures (SOPs) for the laboratory. Each set of directions should be identified on each page, and contain the date of the document, date of the revision, if applicable, and the page number out of the total number of pages. This is to serve the purpose of providing a document control procedure or a means of maintaining the procedures in a complete and current condition.

8.3 A readily accessible office file of methods used should be maintained and kept current.

8.4 The detailed procedures of formerly used methods should be archived for reference and documentation of prior procedures.

9. Metrology Systems

9.1 The basic system of weights and measurements for a laboratory should be as comprehensive as required to conduct all the necessary measurements. National Institute of Standards and Technology (NIST) certified mass standards provide the standard against which the laboratory balances may be checked. This also provides the basic method of volumetric calibration of glassware. An NIST-certified thermometer provides a check on the temperature measurement systems. A set of color standards may be used to check the wavelength calibration and the stray light characteristics of a spectrometer or colorimeter. These systems should be common to most analytical laboratories. Many of these systems, such as balances and spectrometers, may be maintained and certified under an annual service contract.

9.2 Atmospheric sampling often involves the measurement of gaseous volumes. This means that the calibration of the variable-area meter (rotameter) that is used in the field should be traceable through the dry gas meter calibration or the wet meter calibration to the spirometer calibration which is considered to be a primary standard. The bubble flow meter is another primary volume measurement that should be available for low flow rates of gaseous volumes.

9.3 The laboratory should also be capable of making other physical measurements that may be necessary to the characterization of the atmosphere or emission, such as, but not necessarily limited to, wind velocity, barometric pressure, and relative humidity. The apparatus for making these measurements should have calibration procedures supplied by the manufacturer.

9.4 Whenever possible, metrology system calibration and metrological procedures should be traceable to standard procedures and standard weights and measures.

9.5 Periodic performance control of metrologic equipment should be instituted and records of performance maintained. The use of control charts may be an aid in maintaining a record of performance.

9.6 All metrology systems should have a record of calibration and maintenance schedules and there should be a notation of any configuration changes that may have occurred in any system. Also, records of significant changes in calibration should be noted and reviewed periodically for indication of needed modifications of systems or procedures.

10. Recommended Operational Practices

10.1 There are many good laboratory practices which, if followed, will tend to make a more reliable operation. Some of these practices are as follows:

10.1.1 A bound field logbook should be kept by the field sampling team for the purpose of recording field measurements and other pertinent information necessary to refresh the sampler's memory in the event that some information is lost, or if the sampler is called to testify concerning his field activities.

10.1.2 A log should be kept of incoming chemicals and reagents and upon the makeup of reagents, with an indication of their expected shelf life.

10.1.3 A reagent blank should be carried through all sampling and analytical procedures.

10.1.4 The colorimetric sample determinations should be performed against distilled water. The colorimetric values for the reagent blank should then be corrected, rather than blanking out the reagent. This technique allows the analyst to develop some knowledge of the usual value of a reagent blank which will serve as a warning against an unsuitable reagent.

10.1.5 When the data are obtained through the use of a standard curve, the points on the curve should be treated statistically and a regression line should be developed for the purpose of the analysis.

10.1.6 The utilization of reference materials, which are available from such sources as the National Institute of Standards and Technology, are encouraged for conformation of the adequacy of the technique and the analyst. This is also a useful tool for trouble-shooting deficiencies.

11. Data Recording

11.1 There are many systems for recording data, depending upon the means by which the data are generated. These systems are all acceptable as long as they meet the basic requirements that are outlined in 6.1.4.

11.1.1 For more detailed discussion of data recording and record keeping procedures, especially in evidentiary situations, see Ref. **(8)**.

11.2 The most commonly used and most functional method of recording data from the laboratory is the use of a laboratory notebook that is specifically printed for this purpose. The pages are serially numbered in pairs with a carbon between the pages to provide a matching serial numbered copy of the data. These books are permanently bound, but the duplicate page is perforated for easy removal. The duplicate page may then be filed in a system where it may be readily retrieved. The pages of the notebook are generally lined in a grid pattern with provision for such information as project identification, date, and signature of the analyst. The analyst should also record such information as the parameters that have been determined, a reference to the procedures that were used, and the observations that were made. There should also be a sample calculation that was used in the processing of the raw data. There should be a statement of the quality of those data or a warning on the limitations of the data.

11.3 When the data are generated by the use of an automated or semiautomated system, the data are generally displayed by means of a strip chart recorder, printed tape, or computer printout. Some chart paper makes provision for the signature of the analyst, the date, the sample identification, and the operational parameters of the instrument. If the chart paper, tape, or printout does not make provision for this information it should still be supplied by the analyst.

11.4 If the data are recorded by magnetic means or if the data are transmitted by telemetry, the preservation and retrievability of data will require secure systems.

11.5 Chart papers, tapes, and printouts should be retained as a part of the permanent record. Some laboratories may prefer to use microfilm for record retention.

11.6 The reporting of the data and the analytical results may require a format that is agreed upon by the laboratory and the user of the report. The criteria for the reporting of data are the same as the criteria for recording data. However, the laboratory should state its policy for reporting such items as the number of significant figures, the detection limits, non-detected results, the range of results, or the reliability of results.

11.7 For a suggested report format, see Appendix X1.

12. Data Validation

12.1 The validation of environmental data will require a variety of techniques due to the variety of ways in which data are generated and collected. The validation procedures should take into account the sampling, the sample handling, and the analytical procedures if the data are collected manually. If the data are generated by instrumental monitoring of the sampling, the calibration and the instrument performance statistics should be taken into account.

12.2 *Sampling*—The sampling of atmospheres or emissions, whether it is performed manually or by instrumental means, involves performing operations upon a heterogeneous mass under uncontrolled conditions. Reliable information can seldom be drawn from a single measurement or even a few measurements. The interpretation of the data should be based upon a statistical treatment of the data. The most basic statistical treatment of sampling data considers only the repeatability (precision) and would involve providing at least two replicate samples for analysis so that an average value could be determined. The user of this guide is advised to plan the sampling to provide an adequate number of samples.

12.2.1 The reasons for obtaining the information, the methods of obtaining it, and the levels of confidence that are desired in the information provide too many variables to be considered in this guide. The user is advised to consult Practice D 1357, and Ref. **(9)** for more comprehensive information on the

subject of sampling ambient atmospheres. Additional information on source sampling is available in Ref. **(10)**.

12.3 *Sample Handling and Identification*—To ensure that proper procedures are observed during sample collection, transportation, storage, and analysis, the following information should be available for all samples collected in an effort to protect against loss, misidentification, tampering, or other errors that may be introduced:

12.3.1 Date, time, and location of sample collection,

12.3.2 Sample identification number and the name of the sampler,

12.3.3 Sample preservation techniques utilized, if applicable, and

12.3.4 An indication of whether the samples are time-, light-, or temperature-sensitive, and an indication of the allowable holding time.

12.4 *Chain of Custody*—There should be an accountability of the time that the samples are in transit. If the samples are not maintained within the custody of the person performing the sampling until they are returned to the laboratory for analysis, they should be maintained in a tamper-proof condition by use of a lockable shipping container or a container that is secured by use of a nonpeelable seal. The user may consult the chapters on chain-of-custody procedures contained in Ref. **(8)** for a more detailed discussion.

12.5 *Control of Analytical Performance*—The control of the analytical performance in establishing reliability information on a standard would be primarily concerned with the production of precision and bias data on the analyst and on the analytical procedure. These data may be in reference to the true value of a constituent or characteristic. But in this guide the purpose may be to establish a statement of the quality of analytical performance in reference to a single value, determined by a single operator or analyst, for a constituent or characteristic for which there is no known true value. This condition provides a significant difference from the method of establishing a precision and bias statement for a standard test method. The system for producing these data should be flexible and reasonably economical to use, because it should be consistently and persistently applied on a daily basis if the laboratory is to claim to have a quality control program.

12.5.1 The individual laboratory operations will have different needs, but as a general guideline, the laboratory should perform quality control procedures on approximately 10 % of its work load. The system should not be dependent upon the presence of a statistician for its performance. The system that is presented here is a very basic approach to produce information on the quality of the analytical performance which may be described by the terms *repeatability* and *percent recovery*. If it is desired that precision and bias data be obtained, the user is advised to refer to Practice D 2777.

12.5.2 *Repeatability*—Repeatability refers to the agreement among replicate observations of the test by a single laboratory or analyst. The repeatability or the scatter of results about an average value may be expressed in a number of ways, but the most commonly accepted measure is the standard deviation. There are a number of ways of calculating the standard deviation, but either of the following equations provide a very adequate estimate of standard deviation:

$$S = \sqrt{\frac{\Sigma(x_i - \bar{x})^2}{n - 1}} \quad (1)$$

$$S = \sqrt{\frac{\Sigma x_i^{\,2} - (\Sigma x_i)^2/n}{n - 1}} \quad (2)$$

where:

x_i = value of the parameter found in each aliquot or replicate,
$\bar{x}$ = average value of parameter, and
n = number of aliquots, replicates, or observations.

12.5.3 *Percent Recovery*—Percent recovery is the term used to describe the ability of the analyst and the procedure or the analytical system to recover a known amount of constituent from the sample. This is the most serious and most common type of error to be dealt with in the laboratory. Most analysts can attain acceptable repeatability with practice, but it will take a more concerted effort to find and eliminate a systematic error.

12.5.3.1 The percent recovery may be determined by spiking the sample, which has previously been analyzed for that parameter and for which the average value has been determined, with a known amount of the parameter of interest. The spike material should be from a source other than the calibration materials. The percent recovery is calculated as follows:

$$\%\ Recovery = \left(\frac{C - A}{B}\right) \times 100 \quad (3)$$

where:

A = average amount of the constituent found in the aliquots of sample,
B = spike of a known amount of the constituent, and
C = average amount of the constituent found in spiked aliquots of sample.

NOTE 2—C should be approximately $2 \times A$, or approximately 10 to 20 times the Method Detection Limit concentration, whichever is greater.

12.5.3.2 The percent recovery is based upon the final concentration, taking into account the dilution of the sample by addition of the spike, if the change of volume is significant.

12.5.3.3 The analytical performance may be monitored by plotting the percent recovery on a chart with the norm being the 100 % recovery line and the upper and lower control limits being the lines that correspond to whatever the laboratory director determines is an acceptable performance, such as 90 and 110 % recovery, respectively. When the upper or lower control limits are exceeded, the need for an investigation into the cause is indicated. **(11)**

12.5.3.4 The spiking or known addition technique may not be the most applicable or the most feasible technique in all cases. The constituent may already be present in the sample at a high concentration.

12.5.3.5 An estimation of instrument performance may be determined by performing a 1 + 1 dilution of an aliquot of a previously analyzed sample and then repeating the analysis and observing the result in a similar manner as with the spiked sample. When the dilution is accounted for, the technique not only yields an estimate of the percent recovery, but also has the

advantage of detecting sample matrix interference or enhancement effects in the procedure by diluting out the effect of the matrix. This technique is limited by the possibility of diluting the constituent out of the range of the analytical procedure.

12.6 The sample size should be generally adequate for two trials so that two aliquots of the sample may be analyzed. The two aliquots may be analyzed as duplicates, or one aliquot is analyzed *as received* and the other aliquot is either spiked or diluted so that the two aliquots are analyzed at different levels of concentration.

12.6.1 The technique in 12.6 permits a convenient and reasonably rapid method of arriving at an average value. This technique is feasibly applied to a high percentage of the sample load. With a limited amount of experience, an analyst can recognize a pair of aliquot results that deviate excessively from the average value. It is possible to derive some quality performance data from these paired aliquots, such as repeatability and percent recovery if enough similar samples are tested. If a more detailed treatment of these data are desired, the user is referred to Youden. **(11)** The deviation between the paired aliquot results can provide adequate information to alert the analyst to an out-of-control process.

12.7 *Control of Instrumental Monitoring Performance*—The data validation problems involved with instrumental monitoring are twofold.

12.7.1 First, the instrument operating conditions must be established. The manufacturer should furnish the instructions for this process. The setting of the instrument conditions may consist of an electronic nulling of the instrument, or it may use a zero gas and a span gas to adjust the instrument response range.

12.7.2 Second, the instrument response should be validated. Since the span gas is a mixture of gases containing the constituent of interest, the zero gas/span gas calibration procedure also provides an indication of the instrument response. The response may also be checked by using a spiking technique in which a permeation tube adds a known amount of the constituent of interest to the sample stream; alternately, a reference gas or a zero gas may be used to perform a dilution of the sample. The user may find more specific information on calibration by referring to *ASTM STP 598.* **(12)**

12.7.3 Another alternative which may be used in some situations is to perform parallel manual sampling and analytical procedures for comparison to the results obtained with the instrumental monitoring technique. The user is referred to Practice D 3249 for the procedure of validating instrumental monitoring by manual techniques.

12.7.4 A log should be maintained on the instrument used, the calibration technique, the validation technique and the responses observed. This log should contain enough detail on the instrument operation to aid in the establishment of adequate maintenance schedules. All instruments should be recalibrated after service or repair.

13. Deficiency Correction

13.1 This guide has covered quality control procedures in the previous sections. When the product of the laboratory is found to be unsuitable for its purpose or when the quality or the reliability of its information is questioned, these procedures should be investigated. The documented quality control procedures provide the tools or the guidelines for this investigation. The structure in the organization that management should provide for this investigation is the quality assurance plan.

13.2 *Investigation or Trouble Shooting*—The investigation of the problem or trouble shooting should cover all phases of the information gathering and reporting systems such as:

13.2.1 *Sampling*—The records of the sampling should be reviewed.

13.2.1.1 *Sample Handling*—Check the record for the preservation technique, the chain of custody, the time in transit, and the condition of the samples upon arrival at the laboratory.

13.2.2 *Analytical Procedure*—Check the methodology that was used, calibration and maintenance log on the metrology systems used, and the raw data that were recorded. Also, check the reagents used for the date of expiration.

13.3 The investigation should reveal some practices that could be used to improve the laboratory operation. Refer to Section 10 of recommended practices of operation.

13.4 Deficiencies that have been discovered and corrected should be recorded in a log or filing system stating the parameter involved, the problem, the action taken, and the date of the action.

13.4.1 When the deficiencies have been discovered and corrected, a quality assurance plan is in operation. It is by this means that better laboratory practices may be instituted.

14. Keywords

14.1 laboratory organization; laboratory practices; laboratory quality assurance

APPENDIX

(Nonmandatory Information)

X1. SUGGESTED REPORT FORMAT

X1.1 The following items are suggested for inclusion in the report in the following format:

Date: _____

File Number: _____

Report Recipient: (To whom report is addressed.)

RE: (Description of project (user's P.O. No. or name)).

Purpose: (Objective of the project, examination, investigation, or assignment.)

Description of Sample(s): (Physical character, report used identification, and laboratory identification. Location of the sample collection point(s)).

Observations: (If a field examination or a laboratory examination is performed, this should be presented.)

Procedure: (The methods by which the objectives are to be achieved.)

Results: (The results of test, observations, and experiments. May be presented in a table or by the use of charts or diagrams, or combinations thereof. Example of calculations that were utilized should be presented.)

Discussion: (Meaning of results or observations, or both, and the limitations of procedures.)

Conclusion: (The result that can be reasonably extrapolated from the data and observations with a statement of reliability or confidence limit. Instead of the statement of reliability, a separate section may be included which describes quality control, quality assurance, or validation procedures, or a combination thereof.)

Recommendation: (If there is a precaution to be given or a change that should be made to remedy a problem or an eminent danger or violation of which the report user should be apprised or if additional testing or examination should be performed, this section may be used. A disclaimer may be needed to limit the liability of the laboratory in the use of this information, due to limitations of the sampling, or analytical procedures, or age, or condition of the samples, or whether the samples were submitted *blind*, or were collected by laboratory personnel, or combination thereof.)

Submitted by (Name of laboratory) _____

(signed) _____

Laboratory Director

(signed) _____

Chemist/Analyst/Investigator

REFERENCES

(**1**) Armentrout, D. W., Johnson, T. R., and Nelson, A. C., *Validation of Air Monitoring Data*, EPA 600/4–80–030, U.S. Environmental Protection Agency, Research Triangle Park, NC, 1990.

(**2**) *Quality Control System Requirements for a Testing and Inspection Laboratory*, America Council of Independent Laboratories, Inc., 1725 I St., N. W., Washington, D. C. 20006. 1976.

(**3**) *Industrial Ventilation–A Manual of Recommended Practice*, American Conference of Governmental Industrial Hygienists, P. O. Box 1937, Cincinnati, OH, 45201, 1987.

(**4**) Baker, C. J., *The Firefighters Handbook of Hazardous Materials*, edited by Baker, C. J., Maltese Enterprises, Inc., 8309 W. Morris St., Indianapolis, IN 46231, 1984.

(**5**) Lewis, H. F., *Laboratory Planning for Chemistry and Chemical Engineering*, edited by Harry F. Lewis, Reinhold Publishing Corp., 1962.

(**6**) The Resource Conservation and Recovery Act of 1976 (RCRA), Public Law 94 580.

(**7**) The Occupational Safety and Health Act of 1970, Public Law 91-596, CFR 1910.

(**8**) *Quality Assurance Handbook for Air Pollution Measurement Systems, Vol I, Principles*, EPA–600/9–76–005 *Vol II, Ambient Air Specific Methods*, EPA–600/4–77–027a *Vol III, Stationary Source Specific Methods*, EPA–600/4–77–027b.

(**9**) Riggin, R. M., *Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air*, EPA 600/4–83–027, U.S. Environmental Protection Agency, Research Triangle Park, NC, June 1983.

(**10**) Estes, E. D., and Mitchell, W. J., *Techniques to Determine a Company's Ability to Conduct a Quality Stack Test*, EPA 600/S4–82–018, U.S. Environmental Protection Agency, Research Triangle Park, NC, August 1982.

(**11**) Youden, W. J., *Statistical Techniques for Collaborative Tests*, The Association of Official Analytical Chemists, Box 540, Benjamin Franklin Station, Washington, D. C. 20044, 1969.

(**12**) *Symposium on Calibration in Air Monitoring*, ASTM STP 598, ASTM, 1975.