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

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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, as well as atmospheric deposition samples. For details specific to stack gases, see Practice D7036, which covers administrative issues in full; several specifics in this guide regarding laboratory operations may yet be helpful and do not overlap with Practice D7036.

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 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

D3249 Practice for General Ambient Air Analyzer Procedures

D3670 Guide for Determination of Precision and Bias of Methods of Committee D22

D7036 Practice for Competence of Air Emission Testing Bodies

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, see Terminology D1356.

3.2 *Definitions of Terms Specific to This Standard:*

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

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.3 *generic criteria, n*—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.

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

3.2.5 *inspection, n*—the process of measuring, examining, testing, gauging, or otherwise evaluating materials, products, services, systems, or environments.

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

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

3.2.8 *quality assurance, n*—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.

3.2.9 *quality control, n*—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.

3.2.10 *testing, n*—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 or work space air, 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**).[3]

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 a minimum of 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 minimum 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 minimum of 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.

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

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.

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 14 to 28 m^2 (150 to 300 ft^2) per analyst or 3.6 to 7.3 m (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 subject to national, regional, or local regulations for the generation and disposal of generated wastes, for example, in the United States, the laboratory may be required to have an EPA waste generator number as required by the Resource Conservation and Recovery Act (**6**).

7.3.10 The physical conditions in the laboratory shall comply with applicable national, regional, or local regulations to ensure safe working conditions, for example, in the United States, requirements of the Occupational Safety and Health Act may be applicable (**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 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. Mass standards traceable to the National Institute of Standards and Technology (NIST) or other national standards laboratory provide the standard against which the laboratory balances may be checked. This also provides the basic method of volumetric calibration of glassware. A thermometer traceable to NIST or other national standards laboratory 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 traceable to NIST or other national standards laboratory, 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 electronic 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