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Standard Guide for Management Systems in Laboratories Engaged in Analysis of Water[1]

This standard is issued under the fixed designation D3856; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide provides information on consensus good laboratory practices for laboratories that provide services in the sampling and analysis of water. As consensus standards, these are the minimum criteria that all laboratories should consider in establishing their good laboratory practices. This guide may not be applicable to certain types of laboratories (e.g., microbiological).

1.2 This guide is designed to be used by those responsible for the selection, operation, or control of laboratory organizations engaged in sampling and analysis of water.

1.3 This guide presents features of organization, facilities, resources, and operations which affect the usefulness of the data generated.

1.4 This guide presents criteria for selection and control of the features described in 1.3 and also makes recommendations for the correction of unacceptable performance.

1.5 This guide describes methodology and practices intended to be completely consistent with the International Organization for Standardization (ISO) 9000 series of standards and Guide 25 – 1990 (**1**). [2]

1.6 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.7 *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:*[3]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3370 Practices for Sampling Water from Closed Conduits
D3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents
D4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data (Withdrawn 2002)[4]
D4375 Practice for Basic Statistics in Committee D19 on Water
D4447 Guide for Disposal of Laboratory Chemicals and Samples
D4840 Guide for Sample Chain-of-Custody Procedures
D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents
D5172 Guide for Documenting the Standard Operating Procedures Used for the Analysis of Water
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
E456 Terminology Relating to Quality and Statistics
E548 Guide for General Criteria Used for Evaluating Laboratory Competence (Withdrawn 2002)[4]

3. Terminology

3.1 For definitions of terms used in this guide, refer to Terminologies D1129, D4375, and E456, Guide E548, and *ASTM MNL 7* (**2**).

[1] This guide is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.02 on Quality Systems, Specification, and Statistics.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this guide.

[3] 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.

[4] The last approved version of this historical standard is referenced on www.astm.org.

4. Summary of Guide

4.1 This guide describes the criteria, guidelines, and recommendations for physical and human resources and data validation for the operation of a laboratory.

4.2 Although, philosophically, this guide is intended to apply to all analyses of water, there may be certain test methods to which parts of this guide are not applicable due to the nature of the samples, for example, microbiological analyses.

5. Significance and Use

5.1 Data on the composition and characteristics of water are frequently used to evaluate the health and safety to humans and the environment.

5.2 Moreover, such data are frequently used for process control or to ascertain compliance with regulatory statutes that place limits on acceptable compositions and characteristics of waters.

5.3 Laboratories that conduct water sampling and generate analytical data, and those persons who have the responsibility for selecting a laboratory to perform water quality studies, need to use criteria, guidelines, and recommendations that have been developed by consensus and are well accepted in making this selection.

5.4 Demonstration and documentation by a laboratory that there was judicious selection and control of organization, facilities, resources, and operations will enhance the credibility of the data produced and promote its acceptance.

6. Organization

6.1 *General*—The production of reliable data is effected through the effort of everyone involved with the service. It is paramount, therefore, that personnel have a clear understanding of their duties and responsibilities and their relationship to the product produced. Management has the responsibility for defining function and goals as applied to the individual. A formal document describing objectives, staff functions and responsibilities, should be distributed and explained to all staff members.

6.1.1 The personnel in a laboratory will vary with the specific functions that are to be served, but minimal qualifications and duties generally will be as described in 7.2 through 7.3.2.

6.2 *Laboratory Director*—Must have a BS or BA degree with a strong chemistry emphasis and with at least 5 years laboratory experience including supervisory roles or equivalent.

NOTE 1—The purpose of the —equivalent‖ requirement is to allow the assignment of persons who have comparable skills obtained through qualified training which did not result in the award of a baccalaureate degree. Interpretation of the term —equivalent‖ will necessarily require careful judgment by the user of these guidelines. Certification by professional boards is to be encouraged.

6.2.1 The laboratory director or manager should be a full-time employee who operates the laboratory with at least the responsibilities outlined below.

6.2.1.1 Establishment of long-term program plans and shorter term work plans and assignments to meet the program objectives.

6.2.1.2 Operation and maintenance of the physical plant (building, equipment, instrumentation, services, etc.).

6.2.1.3 Selection, training, and development of personnel.

6.2.1.4 Overview and approval of methods of sampling and analyses.

6.2.1.5 Oversee development and implementation of a Quality Assurance (QA) program to monitor and maintain the quality of laboratory performance. This includes ensuring staff participation in appropriate interlaboratory quality control activities, intercalibration checks, performance audit programs, etc. Such interlaboratory checks are the most effective measure of comparative performance and should demonstrate the worth of a good QA program to upper management or regulatory agencies. A QA program also provides each laboratory staff member with a copy of the QA plan for the laboratory, which documents responsibilities and kind and frequency of quality control checks. The plan should also specify the monitoring and overview responsibilities of management. This responsibility is implemented by the Quality Assuranace Manager or Coordinator.

6.2.1.6 Establishment of a development and operational performance appraisal system for the staff and an individual career development plan for each staff member. Performance standards should be developed and agreed to jointly by each staff member and their supervisor. The director should be responsible for assuring a periodic review of performance of all staff members by supervisors, for rewarding good quality performance, and for implementing and encouraging on-the-job or offsite training. This joint development of performance standards is key to obtaining an understanding between the worker and the supervisor, as to what is expected for satisfactory performance. It also paves the way for rewarding outstanding performance or identifying unsatisfactory performance. These standards should be used to evaluate performance frequently but informally, and formally on a less frequent (annual or semiannual) basis.

6.2.2 Quality Assurance Manager or Coordinator – Reports directly to the Laboratory Director.

6.2.2.1 Develops and implements the QA Plan as described above.

6.2.2.2 Investigates any quality issues and reviews on a regular basis the quality of all work performed by the laboratory.

6.2.2.3 Hosts third party laboratory assessments and responsible for seeing that all findings are addressed and corrective actions completed.

6.2.2.4 Implement intra- and inter-laboratory QA performance testing programs and evaluate results and taking corrective actions as necessary.

The laboratory shall have one or more of the following staff or persons responsible for multiple roles.

6.2.3 *Senior Staff*—The senior professional staff of the laboratory conduct the difficult and non-routine sampling and analyses, resolve analytical problems, and modify and develop analytical procedures.

6.2.3.1 Senior staff supervise and assist the technical staff in analyses, other laboratory operations and training.

6.2.3.2 Senior staff members should have earned a baccalaureate degree in science or engineering, with a strong chemistry emphasis, from an accredited college or the equivalent (see Note 1) and have at least two years experience at the bench level in a water laboratory.

6.2.4 *Technical Staff*—The technical staff are personnel who perform routine and specialized analyses.

6.2.4.1 Where appropriate, technical staff members should have formal training in the analytical methodology, and quality control, as applied to the specific sample types and concentration levels of analytes which are of interest to the laboratory.

6.2.4.2 Technical staff may be required to satisfactorily complete analytical tests to qualify initially and to periodically re-qualify throughout their work career. Qualification should be based on the generation of analytical results with precision and bias recovery within limits known to be possible for the particular method and which meet the data user's requirements.

6.2.5 *Laboratory Support Staff*—The support staff are nontechnical workers who perform routine field laboratory services in support of the professional and technical staff.

6.2.5.1 In the laboratory, they wash glassware, operate laboratory reagent water systems, autoclaves, drying ovens, and incubators. The support staff also receives, stores, and ships samples, materials, and laboratory equipment.

6.2.6 *Office Support Staff*—The office staff are nontechnical clerical or secretarial personnel who are trained either on the job or by formal schooling in computer programs, filing, recordkeeping, communications by telephone or personal visits, payroll, travel, or some combination thereof.

6.2.6.1 The laboratory or office support staff may be an integral part of the laboratory or may be provided as part of the administrative function in a larger organization.

6.3 *Physical Resources and Related Operating Procedures:*

6.3.1 The laboratory environment can significantly affect the results of water analyses; therefore, the laboratory facility should be carefully designed and periodically inspected and reevaluated. In general, the physical conditions in the laboratory should comply with the applicable U.S. OSHA requirements, and other regulatory safety and legal requirements.

6.3.2 *Equipment and Supplies*—The specific instrumentation, equipment, materials, and supplies needed for the performance of a standard test method are usually described in a written standard operating procedure (SOP). If the laboratory proposes to perform a new analytical procedure, it must be prepared to acquire the necessary instrumentation, supplies and space, and to conduct an appropriate training period prior to its routine use.

6.3.3 *Laboratory Environment*—The laboratory should be kept as free from environmental contamination as possible in order to protect the samples and instrumentation. Specific procedures should be established for assuring the quality of the laboratory reagent water per method specifications or Specification D1193. By doing so, the laboratory ensures the opportunity to produce quality data. The production of valid data not only depends on the collection of representative samples, but also on maintaining such samples as closely as possible to their original condition through careful handling and storage. If the sample cannot be analyzed at once, it should be preserved and stored as required for the analytes of interest. Recommended procedures for collecting, transporting and handling water and wastewater samples are described in this guide or in Practices D3370 and D3694. Recommended chain of custody procedures are described in Guide D4840. Whenever sample holding times must be determined, recommended procedures are described in Practice D4841.

6.3.4 *Ventilation System*—Laboratories should be well ventilated and free of dust, drafts, and extreme temperature changes. Central air conditioning is recommended because: 1) incoming air is filtered, reducing the likelihood of airborne laboratory contamination; 2) uniform temperature is conducive to stable operation of instrumentation and equipment; and 3) low humidity reduces moisture problems with hygroscopic chemicals, samples, and corrosion problems with analytical balances and other instrumentation.

6.3.4.1 In order for the hoods to be effective in removing fumes and aerosols from the laboratory environment, they must be operating at their designed capacity. Proper hood performance cannot be assumed. Hoods should be tested periodically for proper air flow by qualified support staff or a professional maintenance contractor. Hoods should not be located in areas of countervailing drafts, such as between two open doors. Under usual operating conditions, hoods require from 50 to 125 CFM/ft ^{2} (15 to 38 (m^3 /min)/m^2) of face area. For a more detailed treatment of ventilation consult *Industrial Ventilation—A Manual of Recommended Practice* (4) .

6.3.5 *Facilities*—Ideally, the areas provided for cleaning of glassware and portable equipment should be separated from the laboratory working area but located close enough for convenience.

6.3.5.1 Laboratories conducting trace organic analyses which use organic solvents in extraction and clean-up procedures must separate these activities from analytical instrumentation rooms to avoid contamination and reduce hazards.

6.3.5.2 Laboratories conducting analyses with a wide range of concentrations must take care to avoid cross contamination among samples in storage or analysis. Relatively clean samples, highly polluted samples and reagents should be stored separately from each other in vented cabinets and hoods to avoid cross-contamination.

6.3.5.3 Calibration standards should be stored separately from all samples.

6.3.5.4 *Laboratory Design*—Limited facilities and restricted work space may affect the quality and validity of results. Visitors and incidental traffic should be discouraged in work areas. Through traffic can be prevented by good laboratory design.

6.3.5.5 High standards of cleanliness should be maintained and monitored for contamination in work areas and the laboratory. If there is any doubt about the effects of the surrounding laboratory facility upon the analytical results, blanks that have been protected against the laboratory environment should be compared periodically against sample blanks that have been exposed to the laboratory environment.

6.3.5.6 A complete set of material safety data sheets (MSDSs), or equivalent safety information for all chemicals used in the laboratory, should be on file in a location accessible to all employees. Samples, reagents, and solvents that may contain harmful or interfering fumes shall be used in a properly operating hood or glove box. Smoking, eating, and drinking should not be allowed in the work area. Soiled hands should be washed before handling analytical materials. Sinks shall not be used for sample or reagent disposal. Laboratories shall dispose of waste in accordance with applicable environmental and safety regulations and standards. Standard operating procedures (SOPs) as described in Guide D5172 for handling, storage, and disposal of hazardous reagents and samples shall be defined. Additional information is available in Guide D4447, MSDSs and Refs (3-11), but this information is for reference purposes only and is not intended to be exhaustive or to supersede regulations. Short courses on handling hazardous and toxic chemicals are available from chemical companies and others.

6.3.5.7 *Electric Power Supply*—The reliability of the instruments is affected by the electrical power supply. Some instruments require separate circuits or a regulated power supply for stable operation. The line voltage and stability should be monitored periodically and not assumed as based on records. Surge suppressors should be installed for any sensitive instrumentation or computers. An isolated ground for individual instruments and antistatic pads are helpful in eliminating stray currents.

6.3.5.8 *Safety Considerations*—The laboratory should be supplied with fire extinguishers suitable for Class A, B, or C fires; spill control materials for acids, bases, and flammables; eye wash and safety shower facilities; and other safety devices that may be consistent with the particular laboratory operation. The facilities should provide for the safe disposal of reagents and samples with written instructions for the utilization of these procedures by all personnel. Wearing of safety glasses, goggles, or face shields should be required for everyone entering the laboratory. A senior staff member should be assigned the responsibility for monitoring laboratory safety, including periodic inspection of facilities and fire extinguishers. Staff should be trained and have the training documented in the following: handling and disposal of potential chemical or biological hazards, or both; use of appropriate safety and personal protection equipment; and general laboratory safety and hygiene. If a laboratory handles radiological samples, the laboratory must have a Radiological License and a Radiation Safety Officer responsible for proper safety and handling procedures.

7. Key Aspects of Management Systems

7.1 *General*—The function of a laboratory is to provide analytical results and related information which are adequate for the intended use. This function is achieved through effective use of a quality assurance program. Every laboratory should develop a written quality assurance program, plan, or manual that demonstrates the effectiveness of its procedures and practices in assuring this quality. In addition to addressing any applicable regulatory requirements, the program should consider the following:

7.2 *Organizational Structure*—A table of the organization should be available which shows the lines of authority, areas of responsibility, and job functions. The laboratory should also provide a description of its capabilities. Laboratory management should demonstrate and foster a positive quality assurance attitude and provide the analytical staff with a written policy to carry out a defined quality assurance program.

7.2.1 *Human Resources*— The key personnel of the organization should be described by means of personal résumés presenting the education and work experience appropriate to the table of organization and the qualifications of the position. For each employee, provision should be made for update of records to reflect additional education, work experience, and continuing training.

7.2.2 *Physical Resources*—The laboratory facilities should provide a working environment that is clean, comfortable, and safe. The instrumentation and equipment must be suitable for the operational needs of the laboratory.

7.3 *Quality Assurance Manager/ QC Coordinator:*

7.3.1 The laboratory regardless of size should have a designated experienced person to oversee quality . That person must be familiar with the methods performed by the laboratory and shall be responsible for maintaining and implementing the Quality Assurance Plan.

7.3.2 The QA designee must have formal training in QA and experience in QA/QC systems. This training and experience may vary with the size and complexity of the laboratory.

7.4 *Quality Assurance Plan:*

7.4.1 QA Plan must meet the requirements of an Accreditation Body or a defined oversite quality program. The QA Plan must include the the requirements defined in the following Sections 7.5-7.16.

7.5 *Methodology/SOPS*—Written Standard Operating Procedures must be readily available to personnel. These SOPs may be based on published procedures or laboratory developed methods. The SOP shall clearly define all steps required in the method.

7.5.1 Written sample receipt, handling and storage requirements should be followed.

7.5.2 Analytical procedures must be written

7.5.3 There should be a document control system to track the currency and completeness of procedures

7.5.4 All SOPs must be approved by QA and member of management. The QA Manager will maintain files of all current and historical SOPs.

7.5.5 There must be described in the SOP the Quality Control samples to be analyzed and criteria for acceptance of the results.

7.5.6 Strict adherence to the method SOP shall be maintained and checked using a system of method performance assessments. When deviations are necessary, the SOP should be rewritten to reflect the changes. If time does not permit a rewrite, the necessary deviations from the SOP shall be recorded and approved in writing by supervision before proceeding with the analysis. All SOPS must be reviewed on a defined periodic basis defined by the laboratory.

7.6 *Instrument Systems*—Instruments used for making measurements must have the following:

7.6.1 Written calibration procedures, including standards traceability and standard/reagent replacement schedules.

7.6.2 Written or referenced preventive maintenance procedures with scheduled intervals

7.6.3 Records available to document any repair or service of equipment, replacement or change of reagents, or modification of procedures.

7.7 *Sample Receipt and Handling*—the laboratory must have a written procedure for the receipt of samples to ensure the safety of laboratory personnel and the integrity of the samples.

7.7.1 A part of this procedure shall be a response to common issues that may occur in the sample receipt process. i.e. samples not preserved properly, broken samples, incomplete or incorrect paper work.

7.7.2 The procedure shall describe steps taken by the laboratory to log-in the samples into a database and store the samples after receipt.

7.7.3 It is important that samples are handled immediately upon receipt because of the short holding times of some analyses, and all storage and preservation steps must be applied as soon as possible or as soon as required.

7.8 *Instrument Calibration and Maintenance*—Instrument calibration will vary with methods and thus the procedure is best part of the method SOP.

7.8.1 This SOP shall also describe the routine maintenance of the instrument and may include common trouble shooting problems or refer to the instrument manual.

7.8.2 The calibration procedure shall outline the instrument setup procedures and initial settings of the instrument prior to analyzing standards.

7.8.3 The procedure shall define the number and concentration of the standards and the frequency of any check standards, method blanks, and quality control samples.

7.8.4 Proper procedures for system failures shall be defined including problems with the standard curve, and failed QC samples

7.8.5 Major repairs to the instrument to bring it back into operating condition must be documented and a recalibration performed to assure instrument ready for use.

7.9 *Quality Control Samples*—Quality control samples must be run with each batch or group of samples to ensure or understand the quality of the data.

7.9.1 Examples of QC samples are in Table.

Quality Control Sample	Brief Definition
Instrument Blanks	Solvent/reagent
Method Blanks	Solvent/Reagents processed as a sample
Laboratory Control Sample	Purchased or Lab prepared known standard in matrix
Duplicate Sample	A replicate of sample
Matrix Spike	Sample spiked with known standard value
Matrix Spike Duplicate	Replicate of matrix spike
Continuing Calibration Check Std	Standard to check calibration
Dilution blank	Solvent/Rgnt blank diluted same as sample (when applicable)

7.9.2 Trending of QC, through the use of control charts or tables, is necessary to make sure method is performing properly. These data are then used to develop acceptance criteria for a particular control sample for the method.. that is, the laboratory control sample must be within +/- 20% for approval of the sample data.

7.10 *Performance Evaluation(PE) or Testing(PT) Programs*—The laboratory shall participate in PE or PT programs covering key areas of the laboratory's analytical program. The results of these programs must be evaluated by the QAM, who will investigate any problem areas and define and oversee implementation of any corrective actions.

7.11 *Standards Traceability*—Standards must be traceable to a known documented source that certifies the standards contents. If the laboratory produces a standard from raw material then the purity of that material must be known and the preparation must be documented. The method SOP shall describe the standards and concentrations used for the analysis.

7.12 *Training*—All personal in the laboratory must be trained to perfom their job function. This training may be in various forms; on-the-job; third party training courses; or instrument vendor training. Also chemical hygiene, and proper safety training per laboratory function shall be given to the appropriate staff. All training shall be documented and kept current.

7.13 *Data Review, and Reporting*—The QA program shall have muti-level data review to assure data quality. The mimimum should be two reviews (1) the analyst review and (2) a second knowledgeable reviewer. The final review must be documented. The reports must be prepared to meet client or regulatory needs and must also be reviewed and signed by management. Copies of all project data and reports must be maintained for a period of time as designated by the laboratory or regulatory requirements.

7.14 *Laboratory Information Management Systems*—Computerized laboratory information management systems(LIMS) vary with laboratory size and sample load. The LIMS may vary from simple document forms where samples are logged into the laboratory and data entered for reporting per defined templates to systems that upload information directly from the instruments and generate data reports automatically. In all cases the LIMS must be tested to assure data is calculated correctly and access limited to a few personnel with a need to know and with the ability to change data.

7.15 Non- conformances – When laboratory processes or systems require a variance or where an anomaly has occurred in the laboratory with a particular sample, method or process this must be recorded to document a corrective action taken or initiate an investigation to determine cause and appropriate corrective action. All non-conformances must be reported to the QAM, who will determine the next step.

7.16 *Assessments*—– Assessments fall into two categories (1) Instrument or method assessments using performance evaluation or performance testing samples. These may be generated inhouse or where possible received from a third party as blind samples. The performance samples allow QA to

evaluate the management systems and the over quality of the procedure. (2 Internal assessments to be performed annually by the QAM to evaluate all quality related areas of the laboratory operation The internal audit items should be defined by QA in a document that may include checklists.

8. Metrology

8.1 A set of Class 1 weights or better must be available to make periodic checks on balances. A National Institute of Standards and Technology (NIST) certified thermometer should be used periodically to check temperature measurement devices. A set of color standards may be used to check the wavelength calibration and the stray light characteristics of a spectrophotometer or colorimeter. Systems such as balances and spectrometers can be maintained and certified under an annual service contract.

8.2 All metrology systems must have a record of calibration and maintenance schedules and should note configuration changes that may have occurred in such a system. Records of significant changes in calibration should be noted and reviewed periodically.

9. Data Recording

9.1 Laboratory data must be recorded either as a electronic or written document. The analyst should record information on the analyte, method of analysis, analytical conditions, date of analysis, analyst, and results, and remarks. There should be an example of the calculations. Written documents shall be in ink with no erasures or whiteout. Revisions should be indicated by a single line through the original entry with the correction alongside or referenced. Changes or corrections shall be dated and initialed.

9.2 When data are generated electronically, they must contain the information noted above in 9.1 and approval must be documented.

9.3 Electronic resultsResults are reviewed by the analyst usually on a monitor screen, and a hard copy is printed out only as desired. Results, evaluations, and summaries are archived off-line. Use of CD disks, floppy disks, DVD disks, “thumb” drives, network servers and memory stick back-ups provide the necessary redundancy to avoid loss from system crashes. A wealth of versatile software programs for personal computers permits statistical evaluations, spread sheets, scheduling, complete record-keeping for cost monitoring, supply management, quality control monitoring, report writing, and laboratory management. For further information and recommendations for ensuring data integrity in automated laboratory operations, consult the *Good Automated Laboratory Practices* (**12**).

9.4 The recording of the data and the analytical results should be in a format that is agreed to by the laboratory and the data user. The laboratory should have a written protocol regarding the number of significant figures, detection limits, reporting convention for nondetection, analytical range, etc.

10. Data Verification

10.1 *General*—The verification of data will require a variety of techniques due to the variety of ways in which data are produced. If the data are collected manually, the verification procedures should take into account the sample receipt, the sample handling/preparation, the calibration and performance of the analytical system, and the calculations. The sample preparation, the calibration, instrument performance and calculations should be taken into account if the data are generated by instrumental means.

10.2 *Sampling*—Because the sampling of water (Standard Practices D3370), whether performed manually or by instrumental means, involves operations upon a heterogeneous mass under uncontrolled conditions, reliable conclusions can seldom be drawn from one or a few samples. The sampling plan must provide an adequate number and volume of samples to permit statistical evaluation of the data produced. Information on the number of samples from which a final result is derived should be available to the data user but is beyond the control of the laboratory. The reasons for obtaining the information, the methods of obtaining it, and the desired levels of confidence in the information cannot be addressed within this guide. for all situations. For further information, see the U.S. EPA *Handbook for Sampling and Sample Preservation* **(13).**

10.3 *Sample Handling and Identification*—To ensure that proper procedures are observed, to track sample collection, transportation, storage, and analysis, and to protect against loss, misidentification, tampering, or other errors that may be introduced, the sampler is responsible for providing the following information for every sample collected:

10.3.1 Collection date, time, and location;

10.3.2 Weather conditions and other remarks considered appropriate;

10.3.3 Sample identification number and the name of sampler;

10.3.4 The analytes to be tested and the sample preservation techniques utilized, if applicable; and

10.3.5 Appropriate warnings whenever the samples are hazardous (identying the hazard), time, light, or temperature sensitive, coupled with an indication of the allowable holding time. If it is necessary to estimate appropriate holding times, refer to Practice D4841.

10.4 *Chain of Custody*— The laboratory should record the available history of every sample received, including its collection, preservation, transportation, transfers, analysis, and final disposal. This record will assist the laboratory in the investigation of any problems regarding the sample. If the sample is to meet regulatory or legal requirements, a formal chain of custody is essential. For details regarding chain of custody procedures see Guide D4840.

10.5 *Analytical Quality Control (AQC)*—Items stated in 10.6 through 10.12 are recommended as the basis for a routine within laboratory analytical quality control program. SOPs for each method should contain the QC specifications appropriate for that method. The appropriate QC samples will be defined by the QC section that appears in each ASTM Committee D-19 test method which are based on Practice D5847. If the method will be used to compare results between different laboratories, see Practice D2777. For further information, see the U.S. EPA

Handbook for Analytical Quality Control in Water and Wastewater Laboratories (**14**).

10.6 *Calibration*— For each analyte, prepare a calibration curve which covers the entire working range of the method. Construct the curve using at least three points, including one near the upper limit of the concentration range and one near the lower limit with a reasonably equitable distribution of the remaining points. The actual minimum number of calibration points depends upon the width of analytical range and the shape of the calibration curve. For example, a broad range or a curve not known to be linear might require calibration at five to seven points.

10.7 *Method Blank:*

10.7.1 A method blank should be run to identify sources of contamination arising from the reagents or handling procedures used in performing the analysis. Determine reagent water, reagents, and solvent blanks for each set of samples analyzed, when there is a change in the reagent water system, or whenever a new source (newly prepared reagent or solvent) is introduced into the analytical system. Reagents or solvents, or both, should also be checked for purity prior to use.

10.7.2 Carry each method blank through the entire procedure.

10.7.3 Response to a significant method blank contamination depends a great deal on the method, but the associated data shall certainly be evaluated, and every effort should be made to resolve or minimize system contaminants. For each method, establish a maximum limit for the method blank based on end user's requirements. The SOP shall describe the calibration points and if a new curve is not established each time then the SOP shall define the procdure for checking an existing curve and the criteria that the check procedure must meet. For example a continuing calibration check standard shall be run prior to and during analysis of samples (every 10 samples) and must be within 10% of true value.

10.8 *Field Blank:*

10.8.1 Different types of field blanks may be used during sampling to distinguish among potential sources of contamination that can affect the sampling process. Transport aliquots of analyte-free water or solvent to the field in sealed containers as field blanks for later return to the laboratory with the samples. Designate a specific number of field blanks as trip blanks, which are not opened in the field but are used to detect any contamination arising from handling, transport, or site storage. Designate a specific number of field blanks as equipment blanks, which are passed through the sampling equipment to detect any contamination from the equipment itself or the conditions during sampling.

10.8.2 Analyze appropriate field blanks with each set of samples from a given source. Carry each field blank through the entire procedure.

10.8.3 When interferences occur, it is best to discard the associated analytical results, investigate the cause so such losses may be avoided in the future, and resample. In situations where it is impossible to resample, however, it may be necessary to report the available results along with a note explaining the extent of the interference and its affect upon the data.

10.9 *Precision*—Precision is the closeness of agreement between the results of repeated analysis on the same sample.

10.9.1 *General*—Develop the necessary initial data by randomly selecting routine samples to be analyzed twice in order to provide duplicate analyses. Consider the steps in 10.9.1.1 – 10.9.8 .

10.9.1.1 Develop these data over a reasonable period of time to reflect day-to-day operations.

10.9.1.2 Choose the samples that are most representative of the interference potential of the sample type. If the laboratory handles multiple sample types with different precision characteristics, it will be necessary to establish and maintain separate background data and evaluation criteria for each sample type.

10.9.1.3 Ultimately, samples representing the entire concentration range should be included within each sample type if necessary.

10.9.2 From each pair of duplicate analytes (X_1 and X_2) calculate their relative range value (R):

$$R = \frac{|X_1 - X_2|}{(X_1 + X_2)/2}$$

where:

$|X_1 - X_2|$ = means the unsigned difference between X_1 and X_2.

10.9.3 After 50 to 100 R values are available for an analyte, order the R values by their related sample concentration estimates, organize the values into concentration ranges that seem to have a similar underlying R value, and calculate the average R value ($\bar{R}$) for each of these concentration ranges. Minimize the number of concentration ranges as much as practical.

10.9.4 Calculate the upper control limit (UCL) for each concentration range as follows:

$$UCL = 3.27\left(\bar{R}\right)$$

(See Note 2.)

NOTE 2—This factor may be found in Table 2, p. 83 of Ref. (**2**).

10.9.5 Review the initial data for R values greater than the UCL value for the appropriate concentration range. If such values are found, they should be discarded and the related UCL value should be recalculated from the remaining R values within that concentration range.

10.9.6 Within each set of 20 or fewer samples to be analyzed together, evaluate system precision by conducting duplicate analyses on one of the samples selected at random. If the relative range value (R) calculated from these duplicates is greater than the appropriate UCL value, system precision is judged to be out-of-control and analyses must stop until the problem has been resolved. Problems with these data may indicate the need for stricter adherence to accepted laboratory practices.

10.9.7 After obtaining 20 to 25 additional acceptable pairs of data within each concentration level for a sample type, periodically update the table of critical relative range values by repeating the step described in 10.9.4 using the new data. Review the criteria being maintained and combine any which are very similar for related concentrations or sample types. If

the criteria for adjacent concentration ranges are quite different, further subdivision by concentration may be necessary.

10.9.8 Table A1.1 gives an example of precision estimates from duplicate analyses within specific concentration ranges for three analytes.

10.10 *Bias Check Using Standard Solutions:*

10.10.1 Analyze at least one standard through the complete method for every subset of 20 or fewer routine samples to be analyzed together. This standard of known concentration can be purchased from an external source or prepared in house from materials or solutions of known purity. It should come from a source of material different from that used for the calibration.

10.10.2 To provide a complete record of the calibration and recovery for each analytical run, one of these standard samples should be the last sample analyzed.

10.10.3 Use concentrations that approximate those found in the related routine samples. Calculate percent recovery (P) as follows:

$$P = \frac{100\ (\text{observed value})}{(\ \text{true value})}$$

10.10.4 After 20 to 25 standards are analyzed over time, calculate average percent recovery ($\bar{P}$) and standard deviation (S_P) of the resulting P values.

10.10.5 If subsequent standards for percent recovery are not within the interval $\bar{P} \pm 3\ S_P$, the analytical system should be checked for problems. If problems exist, resolve them before continuing the analyses. Problems with these data often require greater care in sample processing prior to actual measurement.

10.10.6 Runs of seven or more successive points, all either above or below $\bar{P}$, also indicate the system is out-of-control. Use of a Shewhart $\bar{X}$-chart is recommended to facilitate evaluation of percent recovery results. An example of the calculation of percent recovery and development of a Shewhart $\bar{X}$-chart is given in Table A1.2 and Fig. A1.1.

10.10.7 Record recovery of all acceptable check standards and, after 20 to 25 additional results, revise the related control limits by recalculating P and S_P from the new data. As in 10.9.7, the criteria subdivisions by sample type and concentration range should be periodically reviewed to judge their appropriateness.

10.11 *Bias Check Using Recovery of Spikes:*

10.11.1 Do essentially the same thing for recovery as was done in 10.10, except that a concentrate spike is added to a randomly selected *routine environmental sample* from the current analytical run rather than to *reagent water*. Different types of routine environmental samples may have to be dealt with separately if the samples exhibit different spike recovery characteristics. From this point on, this discussion is in terms of a specific identified sample type. P values for the recovery data are calculated as follows:

$$P = \frac{100\left[A\left(V_s + V\right) - \left(BV_s\right)\right]}{CV}$$

where:

A = measured concentration of the component in the spiked sample,
B = measured background concentration of component in the sample,
C = concentration of component in spiking solution,
V_s = volume of sample before spiking, and
V = volume of spiking solution used.

10.11.2 In spiking samples, make sure that:

10.11.2.1 Sufficient spike is added to at least double the background concentration or to reach a concentration for which the calibration curve has been established. If the background concentration is higher than the midpoint of the standard curve, the background water should be diluted into the lower half of the calibration range and reanalyzed before spiking.

10.11.2.2 The volume of a spike should be kept to a minimum and not exceed 5 % of the sample volume. In organic analyses, the volume of spike should be no greater than 150 mL so that the solubility of the standard in the water will not be affected.

10.11.3 Resulting P values must fall within $\bar{P} \pm 3\ S_P$ calculated from previous related spike recovery data. If not, the system may be out-of-control, and the cause must be found and corrected before continuing the analyses. Problems with these data often indicate sample matrix interferences. Related spike recovery data are developed from a particular environmental matrix, that is, groundwater, wastewater, etc. These limits may differ from the limits calculated in 10.10.3 – 10.10.5.

10.11.4 As in 10.10.6, runs of seven or more results on the same side of X indicate the system is out-of-control, and the use of a Shewhart X-chart is recommended to facilitate evaluation of results.

10.11.5 By simply calculating P from P values calculated as specified in either 10.11.1 or 10.10.3, percent recoveries of a spike can be treated as shown in the example given in Table A1.2 and Fig. A1.1.

10.11.6 Periodically review and update the recovery criteria similarly to 10.10.7.

10.12 *Summary of the Analytical Quality Control*—The following recommended analytical quality control program should be the standard practice in any laboratory.

10.12.1 Three or more standards are needed to develop a calibration curve in concentrations covering the working range, as necessary, or measurement of two calibration standards to verify the existing calibration curve.

10.12.2 One method blank per run.

10.12.3 One field blank per set of samples.

10.12.4 One duplicate for precision check (at least one every 20 routine samples).

10.12.5 One standard sample for recovery and calibration check (at least one every 20 routine samples). A standard should be the last sample analyzed in each run.

10.12.6 One spiked sample for recovery check in the presence of a sample matrix (at least one every 20 routine samples).

10.12.7 *Total*—Depending on the end use of the data, seven to ten analytical quality control analyses may be required for

runs of up to 20 routine samples; 10 to 13 analytical quality control analyses may be required for runs of 21 to 40 routine samples, etc.

10.12.8 *Minimal Analytical Quality Control*—For very small operations or small sample loads, the described analytical quality control program may not be practical or necessary for all analytes. Whenever analytical quality control must be reduced below the level recommended, the following minimal analytical quality control program should be maintained.

10.12.9 Continue calibration or calibration checks as described in 10.6.

10.12.10 Analyze one field blank per set of samples to check for contamination. If an out-of-control situation is indicated, a method blank should be run to find out whether the contamination problem is in the laboratory or the field.

10.12.11 Analyze one spiked sample at the end of each analytical run to check for recovery or precision problems. If an out-of-control situation is indicated, analyze a standard to find out whether the problem is basic recovery or calibration, or both. Successful recovery of the standard would suggest either a matrix problem or a precision problem. A precision problem would produce random out-of-control indications, probably caused by poor or inconsistent analytical techniques or instrumentation.

10.13 *Performance Review*—Analysts should maintain a permanent record of the quality control checks which are performed. The laboratory supervisor should hold frequent meetings to review the quality control program with analysts to discuss the quality control checks performed and the resolution of any problems which are detected. Deficiencies which are detected should be documented in the record book indicating the analytes involved, the problem, the action taken, and the date of the action.

11. Trouble Shooting

11.1 Extreme, unexpected or questionable results are normally detected and reported by the analyst, or are noted by the supervisor in the daily reviews of results. When a deviation is noted, the train of sampling and analytical methods and quality control shall be investigated. The documented intralaboratory quality control checks provide the primary means for the investigation.

11.1.1 Review the records of the sample collection. Check the preservation technique used, the chain of custody record, the time in transit, and comments on the conditions of the samples upon arrival at the laboratory, for example, temperature upon arrival, etc.

11.1.2 *Analytical Procedure*—Check calculations for transposition of numbers and mathematical error. Any significant positive blank result indicates field or laboratory contamination of sample, sampling device, sample container, reagents, reagent water, etc. Check monitoring data on reagent water. Check reagents for changes in bottle and lot and expiration dates. Analyze or reanalyze samples to confirm source and resolution of problem. Confirm recoveries with analyses of known reference samples.

11.2 The investigation may indicate good field and laboratory practices were not followed, such as the following:

11.2.1 The field sampling team should keep a bound field logbook for recording field measurements, time, temperature, sampling location, weather conditions, and other pertinent information.

11.2.2 The analyst should keep records on incoming chemicals and reagents, and the preparation of reagents, with estimated shelf lives. Reagent containers should be properly labeled and dated. A mechanism should be established for reorder of chemicals within the estimated shelf lives.

11.2.3 Reagent blanks should be carried through all sampling and analytical procedures. In colorimetry, the reagent blank should be compared with reagent water to detect an unusual reagent blank response.

11.2.4 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 analytical method.

11.2.5 Use of reference materials of known quality from sources such as NIST, or others, should be used to confirm the adequacy of the technique and the analyst.

11.3 Senior analysts must maintain a permanent log of quality control checks performed. The laboratory supervisor shall hold frequent quality assurance review meetings with senior analysts to discuss the quality control checks performed and the resolution of problems detected. Deficiencies and corrective actions must be recorded in a log indicating the analytes involved, the problem, the action taken, and the date of the action. Only when the deficiencies have been discovered, corrected, and confirmed as corrected is the real benefit of a quality assurance plan realized, that is, improved data quality.

12. Keywords

12.1 analytical practices; analytical resources; good laboratory practice; quality assurance; quality control; trouble shooting

ANNEX

(Mandatory Information)

A1. QUALITY CONTROL EXAMPLES

A1.1 Table A1.1 presents the results of carrying out the steps described in 10.9.2 through 10.9.4 for three different analytes to illustrate use of the UCL values. If duplicate chromium results of 31.2 and 33.7 were obtained, the system precision would be checked as follows:

$$R = \frac{|\,31.2 - 33.7\,|}{(31.2+33.7)/2} = \frac{|-2.5|}{64.9/2} = \frac{2.5}{32.45} = 0.0770 \quad \text{(A1.1)}$$

Since the appropriate UCL from Table A1.1 is 0.109 and the current R values are not greater, precision of the analytical system is judged to be within control.

A1.2 The following calculations result from carrying out the step described in 10.11.3 using the data in Table A1.2:

$$\bar{P} = \frac{1}{n}\sum_{i=1}^{n} P_i = \frac{2105.27}{21} = 100.25 \quad \text{(A1.2)}$$

$$S_p = \sqrt{\frac{\sum\left(P_i - \bar{P}\right)^2}{n-1}} = \sqrt{\frac{719.839}{20}} = 5.999 \quad \text{(A1.3)}$$

$$\bar{P} \pm 3\ S_p = 100.25 \pm 18.00 = 82.25\ \textit{to}\ 118.25 \quad \text{(A1.4)}$$

A1.3 Therefore, as specified in 10.11.3, percent recovery values for total PO_4-P standards roughly within the concentration range from 0.34 to 4.9, that occurred below 82.2 % or above 118 %, would indicate that the accuracy of the analytical system is under control. The related Shewhart $\bar{X}$-Chart is illustrated in Fig. A1.1.

TABLE A1.1 Precision Estimates from Duplicate Analyses Within Specific Concentration Ranges for Three Analytes

Analytes	Concentration Range	No. of Sets of Duplicates	Average Concentration of Data	Average Relative Range (*R*)	*R* for Combined Concentration Ranges	Final UCL Results
BOD, 5-Day (mg/L) L	1 to <10	21	5.85	0.1776		
	10 to <25	30	17.6	0.1104	0.1381	0.452
	25 to <50	27	36.1	0.0924		
	50 to <150	29	102.0	0.0638		
	150 to <300	17	197.0	0.0564	0.0652	0.213
	300 to <1,000	12	520.0	0.0232		
	1,000 up	3	3341.0	0.0528		
Chromium (µg/L) L	5 to< 10	32	6.15	0.0612	0.0612	0.200
	10 to <25	15	16.7	0.0340		
	25 to <50	16	36.2	0.0310		
	50 to <150	15	85.1	0.0446	0.0334	0.109
	150 to <500	8	240.0	0.0218		
	500 up	5	3170.0	0.0240		
Copper (µg/L) L	5 to <15	16	11.1	0.1234		
	15 to <25	23	19.1	0.0736	0.0940	0.307
	25 to< 50	21	35.4	0.0338		
	50 to< 100	26	65.9	0.0354		
	100 to< 200	10	134.0	0.0210	0.0313	0.102
	200 up	3	351.0	0.0130		

TABLE A1.2 Analysis [A] of Total Phosphate-Phosphorus Standards, in mg/L Total PO_4-P

Point	Known	Obtained	% Recovery = P_i	$(P_i - \bar{P})^2$
1	0.34	0.33	97.06	10.176
2	0.34	0.34	100.00	0.062
3	0.40	0.40	100.00	0.062
4	0.49	0.49	100.00	0.062
5	0.49	0.49	100.00	0.062
6	0.50	0.47	94.00	39.063
7	0.50	0.53	106.00	33.062
8	0.50	0.56	112.00	138.063
9	0.52	0.59	113.46	174.504
10	0.66	0.70	106.06	33.756
11	0.66	0.60	90.91	87.236
12	0.67	0.65	97.01	10.498
13	0.68	0.65	95.59	21.716
14	0.83	0.80	96.39	14.900
15	1.30	1.20	92.31	63.044
16	1.30	1.30	100.00	0.062
17	1.60	1.70	106.25	36.000
18	2.30	2.30	100.00	0.062
19	2.30	2.40	104.35	16.810
20	3.30	3.30	100.00	0.062
21	4.90	4.60	93.88	40.577
		SUMS:	2105.27	719.839

[A] Using a colorimetric method with persulfate digestion.

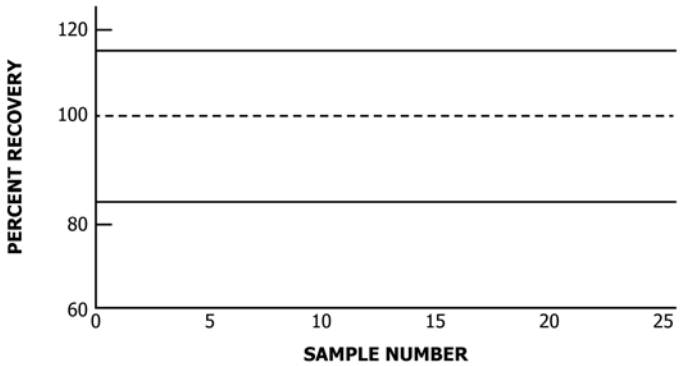

FIG. A1.1 Shewart Control Chart for Percent Recovery Data

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