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Standard Guide for Quality Management in an Organization That Samples or Tests Coal and Coke[1]

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1. Scope

1.1 This guide describes the essential activities that are required to establish and operate a quality management system in a laboratory or organization that provides services in sample collection, sample preparation, or testing of coal, coke, or combustion residues from coal and coke.

NOTE 1—The word "laboratory" is used throughout this guide when referring to an organization that provides services in coal sampling or testing, or both. It is recognized, however, that the word may not be appropriate to an organization that does not perform actual laboratory sample testing.

1.2 The principles of this guide are suggested as being the minimum acceptable requirements for the management of quality in a coal or coke sampling or testing organization, although some elements may not be necessary or appropriate for organizations that provide limited services.

1.3 Laboratories meeting the requirements of this guide may or may not meet the requirements of the ISO 9000 series of standards.

NOTE 2—An accrediting body may also impose other nontechnical requirements such as payment of fees or submittal of quality documentation for review.

1.4 This guide is based upon many of the issues presented in Guide E 548. The user may also consult Guide E 882 for other items that may be pertinent. ISO Guide 25 has also been extensively consulted.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 2013 Method for Preparing Coal Samples for Analysis[2]

D 2234 Practice for Collection of a Gross Sample of Coal[2]

D 3180 Practice for Calculating Coal and Coke Analyses from As-determined to Different Bases[2]

D 3302 Test Method for Total Moisture in Coal[2]

D 4182 Practice for Evaluation of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke[2]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Methods[3]

E 456 Terminology Relating to Quality and Statistics[3]

E 548 Guide for General Criteria Used for Evaluating Laboratory Competence[3]

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[4]

E 1187 Terminology Relating to Laboratory Accreditation[3]

E 1323 Guide for Evaluating Laboratory Measurement Practices and Statistical Analysis of the Resulting Data[3]

E 1579 Guide for Ensuring Data Integrity in Highly Computerized Laboratory Operations[3]

2.2 *Other ASTM Document:*

Manual on Presentation of Data and Control Chart Analyses, *ASTM Manual* 7, 1996[5]

2.3 *ISO Document:*

Guide 25 General Requirements for the Competence of Calibration and Testing Laboratories[6]

3. Terminology

3.1 *control limits, n*—limits on a control chart which are used as criteria for signaling the need for action, or for judging whether a set of data does or does not indicate a state of statistical control

3.1.1 *Discussion*—Action may be in the form of investigation of the source(s) of an "assignable cause," making a process adjustment, or terminating the use of a process. (**E 456**)

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[2] *Annual Book of ASTM Standards*, Vol 05.06.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

[5] The procedure described herein was adapted from *Basic Statistics—Tools for Continuous Improvement*, M. J. Kiemele and S. R. Schmidt, Air Academy Press, Colorado Springs, CO, 1991.

[6] *Manual on Presentation of Data and Control Chart Analyses, ASTM Manual 7*, American Society for Testing and Materials, 1996.

3.2 *calibration*, *n*—the set of operations which establishes, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system, or values represented by a material measure, and the corresponding known values of a measurand. (**E 548**)

3.2.1 *Discussion*—The act of calibration is also sometimes referred to as standardization. The result of a calibration is sometimes expressed as a calibration factor, or as a series of calibration factors in the form of a calibration curve.

3.3 *certified reference material, CRM*, *n*—a reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation that is issued by a certifying body.

3.4 *certifying agency*, *n*—an agency that, by virtue of its technical capability, reliability, and leadership, is recognized as capable of producing one or more reference materials, and certifying the magnitude of selected properties of the materials after testing or analyzing them using a reference method.

3.5 *control, statistical*, *n*—a process is in statistical control if the variations between the observed test results from it can be attributed to a constant system of chance causes. (**E 177**)

3.6 *control material, CM*, *n*—a material of known property values, that is analyzed or tested concurrently with test samples or at specified intervals to evaluate the accuracy of a measurement process.

3.7 *quality assurance, for laboratories*, *n*—the activity of providing the evidence needed to establish confidence that laboratory data are of the requisite accuracy. (Precision and Bias) (**E 1187**)

3.8 *quality control*, *n*—the process through which a laboratory measures its performance (of analyses and tests), compares its performance with standards, and acts on any differences. (**E 1187**)

3.9 *quality manual*, *n*—a document stating the quality policy, and describing the quality management system and quality practices of an organization.

3.9.1 *Discussion*—The quality manual may incorporate by reference other documentation relating to the laboratory's quality arrangements.

3.10 *reference material, RM*, *n*—a material or substance, one or more properties of which are sufficiently well-established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (**E 1187**)

3.10.1 *Discussion*—Reference materials should bear sufficient resemblance to the material to be analyzed or tested so that no significant differences in procedures or corrections (for example, for interferences or interelement effects) are required.

3.11 *reference method*, *n*—a method that has been specified as capable, by virtue of recognized accuracy and authority, of providing reference data, which when verified by existing reference materials of the same or similar matrix, can be used to characterize reference materials.

3.12 *standard*, *n*—a means established by authority, of determining the measure of quantity, quality, extent, or value.

3.13 *standard operating procedure (SOP)*, *n*—any written procedure describing a specific test method or any other aspect of the laboratory operation, including quality practices.

3.14 *standard reference material*, *n*—Because this term has been used in a variety of ways in this guide, it is not defined or used herein, although it is understood by some persons to be equivalent to certified reference materials.

4. Significance and Use

4.1 This guide describes the elements of a quality management system for an organization that samples, analyzes, or tests coal, coke, or their products.

4.2 The quality management system stresses the use of documented accountability and quality control procedures that serve to assure the laboratory personnel and its clients that adequate accuracy and precision are achieved in the routine performance of its sampling and testing activities, and that reported data were obtained from the samples submitted.

4.3 A laboratory may use this guide to develop its quality management system.

4.4 Laboratory clients, regulatory authorities, and organizations or individuals that evaluate the competency of testing laboratories may find this guide useful. Specific guidelines for evaluating a laboratory can be found in Practice D 4182.

4.5 Other accountability and quality control procedures can be developed and may be considered equivalent if they provide all of the benefits previously outlined.

5. Components of a Quality Management System

5.1 A laboratory quality management system consists of the following principal components, which are described in detail in subsequent sections of this guide:

5.1.1 An organizational structure and management commitment designed to ensure that services are performed in a timely and accurate fashion.

5.1.2 A quality manual that documents all aspects of the quality management system;

5.1.3 A staff that has well-defined responsibilities and is trained not only in the technical procedures of sample collection, sample preparation, or sample testing, but also in the philosophy and methods of the quality management system;

5.1.4 Physical facilities and environments that are suitable for the performance of the tasks required;

5.1.5 Equipment and supplies that provide the capability to perform the services;

5.1.6 A quality assurance program that ensures that the laboratory constantly monitors and improves its performance;

5.1.7 Well-defined (that is, documented) procedures for (as appropriate) sample intake or collection, sample management, sample preparation, sample testing, and the calculation and reporting of test results;

5.1.8 A quality control program that constantly ensures that the testing procedures do provide accurate data;

5.1.9 Procedures to ensure that reports accurately reflect the test data, and attribute them to the appropriate sample;

5.1.10 A document control system to ensure that only the latest approved version of procedures are in use;

5.1.11 A record-keeping system that defines what records are required and the length of time that they must be maintained;

5.1.12 A program that provides for audits and reviews of the activities of the organization to ensure that the quality management system is performing as designed.

6. Organization and Management

6.1 Proper organization and management of the laboratory requires the following:

6.1.1 The laboratory is a legally identifiable entity organized and operated in such a way that its permanent, temporary, and mobile facilities meet the requirements of this guide;

6.1.2 The laboratory is organized in such a way that there is confidence in its independence of judgment and integrity at all times;

6.1.2.1 The laboratory ensures that its personnel are free from any commercial, financial, or other pressures that might adversely affect the quality of their work;

6.1.2.2 The laboratory has documented policies and procedures to protect clients' confidential information and proprietary rights;

6.1.3 The laboratory has the staff needed to carry out the laboratory's functions, and specifies and documents the responsibility, authority, and interrelations of all personnel who manage, perform, or verify work affecting the quality of sample collection, preparation, and testing;

6.1.4 The laboratory has a technical manager (however named) who has overall responsibility for the technical operations;

6.1.5 The laboratory has a quality manager (however named) who has responsibility for the quality management system and its implementation.

NOTE 3—The quality manager has direct access to the highest level of management at which decisions are taken on laboratory policy or resources, and has direct access to the technical manager. In some laboratories, the quality manager may also be the technical manager or a deputy technical manager.

6.1.5.1 The laboratory has a well-defined procedure to nominate deputies in case of absence of the technical or quality manager;

6.1.6 The laboratory provides supervision by persons familiar with the sample collection or test methods and procedures, the objectives of the tests and the assessment of the results;

6.1.7 The laboratory has written policies for continuous improvement;

6.1.8 The laboratory has well-defined procedures for soliciting customer feedback in a systematic way and for dealing with customer complaints;

6.1.9 The laboratory, if appropriate, has documents readily available attesting that the laboratory is certified or registered by a recognized agency.

7. Quality Manual

7.1 The quality manual documents the elements of the quality policy, the quality management system, and quality practices.

7.2 Contents of the quality manual are communicated to, understood by, and implemented by all of the staff.

7.3 The quality manual is readily available for use by the staff.

7.4 There is a well-defined procedure for keeping the manual current by modifying the manual whenever any quality system provision is changed. The individual(s) who has/have the authority to make changes is/are identified.

7.5 The quality manual or formal quality documents referenced by the manual contain:

7.5.1 A quality policy statement, including the purpose of the quality management system and the commitments to it by laboratory or institutional management;

7.5.2 A description of the organization and management structure of the laboratory, its place in any parent organization, and all relevant organizational charts;

7.5.3 Documentation of the responsibility, authority, and interrelation of all personnel who manage, perform, or verify work affecting the quality of calibrations and tests;

7.5.4 Identification of the laboratory's authorized signatories (where this concept is appropriate);

7.5.5 An outline of the documentation used in the quality management system;

7.5.6 A description of procedures for control and maintenance of documentation (Section 18);

7.5.7 Procedures to ensure that the laboratory has the appropriate facilities and resources before commencing any new test or contract;

7.5.8 Descriptions of or reference to a document that describes the major equipment and equipment maintenance schedules and procedures;

7.5.9 A list of procedures and tests that the laboratory performs routinely (Section 15);

7.5.10 Documented, standard operating procedures (SOPs), or references to same, for each procedure and test that is routinely conducted by the laboratory. Appropriate ASTM standards are referenced and, if the laboratory deals with international customers, international standards are referenced;

NOTE 4—The terms "standard operating procedure" and "SOP" are used in this guide to designate any written procedure whether it applies to a specific test method or to a procedure that is used in any other aspect of the laboratory operation, including quality elements. The laboratory may refer to such documents by a name other than standard operating procedure. Reference simply to published standards is generally inadequate because they may need to be supplemented by additional information pertaining to quality control activities that are not commonly covered in standard test methods. If an operating procedure deviates from an established standard, data should be available to demonstrate that the modified procedure provides results that are equivalent to those obtained by using the standard procedure.

7.5.11 Calibration procedures and the proper use of reference materials in calibration;

7.5.12 Verification practices, including interlaboratory comparisons or proficiency testing programs, use of reference materials to verify calibrations, and internal quality control procedures;

7.5.13 The laboratory's procedures for achieving traceability of measurements to specific samples (Section 12);

7.5.14 Procedures for recognizing testing discrepancies or nonconforming work or departures from documented policies and procedures, for taking corrective action in such instances,

and for communicating the information to key personnel and (where appropriate) to clients;

7.5.15 Procedures to prevent unauthorized tampering with equipment or modification of control or test procedures, a current list of personnel who are authorized to make any required changes, and criteria to be used to determine when changes are required;

7.5.16 A list of persons who are authorized to permit exceptional departures from documented policies and procedures or from standard specifications, and descriptions of the extent of their responsibilities and authority;

7.5.17 Procedures for soliciting customer feedback and for dealing with customer complaints;

7.5.18 Procedures for protecting confidentiality and proprietary rights.

8. Staff

8.1 The laboratory has personnel with appropriate education, training, technical knowledge, and experience to conduct the operations of the laboratory.

8.2 Where appropriate, technical personnel are able to demonstrate a satisfactory knowledge of sample collection and sample preparation procedures, and are capable of collecting and preparing samples in accordance with appropriate methods or practices.

8.3 Where appropriate, technical personnel are able to demonstrate a satisfactory knowledge of test methods and of sample procurement and processing procedures for which they are responsible. Any procedure for which the laboratory claims competence can be performed by at least one capable individual.

8.4 A formal program is used to identify training needs and to train the technical staff to carry out the procedures for which the laboratory claims competence.

8.4.1 There is an ongoing procedure for evaluating the competence of laboratory personnel.

8.4.2 Records are kept of the relevant qualifications, training, skill, and experience of the technical personnel.

8.5 There is a formal program to train the staff in and to maintain its awareness of the quality management system including quality assurance and quality control methods.

9. Facilities and Environment

9.1 Laboratory facilities, including benches, desks, calculators, computers, supplies, energy sources, lighting, heating, ventilation, and cooling, are appropriate for proper performance of laboratory activities.

9.2 The environment in work areas supports the achievement of required accuracy of sampling or testing.

9.2.1 The laboratory monitors and controls environmental conditions. Appropriate attention is paid, for example, to dust, electromagnetic interference, electrical power voltage level and stability, room temperature and humidity, and sound and vibration levels.

9.2.2 There is in place a documented procedure to stop technical activities when environmental conditions might jeopardize results of tests, and a record is maintained of such occurrences.

9.2.3 Particular care is taken when activities are undertaken at sites other than the permanent laboratory premises.

9.3 Adequate measures are taken to ensure good housekeeping in the laboratory.

9.4 Work areas are separated effectively to prevent inadvertent interference or contamination.

9.5 Access to and use of all areas affecting the quality of results is controlled.

NOTE 5—It is the laboratory's responsibility to comply with relevant health and safety requirements. This aspect, however, is outside the scope of this guide.

10. Equipment and Supplies

10.1 All items of equipment and all supplies required for the proper performance of sample collection, sample preparation, and tests are available in the laboratory. If the laboratory needs to use equipment outside its permanent control, it ensures that the equipment meets the requirements of the appropriate procedure.

10.2 A copy of the manufacturer's instructions (where available) or other instructions are kept in reasonable proximity to the equipment.

10.3 For each major item of equipment, a log is maintained that includes: the name of the item of equipment and an unique identification number; the manufacturer's name; model identification; date received and date placed in service; condition when received (for example, new, used, reconditioned); current location; dates and results of calibrations or verifications and date of the next calibration or verification; details of maintenance carried out to date and planned for the future; and history of any damage, malfunction, modification, or repair.

10.4 All measuring and testing equipment is calibrated or verified for accuracy before being put into service.

10.4.1 Balances are sufficiently sensitive and accurate for the activities in which they are used.

10.4.2 The laboratory has an established program for periodically verifying the calibration status of its measuring and test equipment (Section 14). A record of calibration status is maintained in near proximity to all equipment in use and also, preferably, in the equipment log.

10.5 All equipment is properly maintained according to written procedures and according to a written schedule.

10.6 An updated list of critical parts is maintained and provisions are made to ensure that the list is used to immediately reorder needed spare parts in a timely fashion in order to keep the operation functional.

10.7 Any item of equipment that has produced results suspected to be in error, or that has been discovered to be defective, is immediately taken out of service, clearly marked, and, wherever possible, stored at a specified place until it has been tested, calibrated, or, if necessary, repaired.

10.7.1 The laboratory has well-defined procedures to examine the effect of any defect on previous tests, and is prepared to retest samples that may have been improperly tested because of such defect.

10.8 Unauthorized tampering with or modification of equipment is prohibited. Modifications are made only after a specific problem has been identified and only after approval by an

authorized person, for instance, the technical manager. All corrective actions are documented and the equipment is verified for fitness before being placed in service.

10.9 Sufficient supplies (including reference materials) are kept in stock to ensure that all operations can function. A list of the quantities of critical consumable materials and supplies is maintained and routinely updated, and is used to reorder them in a timely manner.

10.9.1 Records are maintained for every reference material used in the laboratory, and include source, date of acquisition, and other information pertinent to their proper use.

10.10 Wherever appropriate, purchased equipment and consumable materials are not used until they have been inspected, calibrated, or otherwise verified as complying with any standard specifications relevant to the tests concerned.

11. Quality Assurance Program

11.1 The laboratory maintains a quality assurance program that continuously controls and evaluates its performance to ensure that test results meet the needs of its users and clients. The critical elements of the quality assurance system are listed as follows, and are discussed in detail in subsequent sections of this guide.

11.2 *Sample Identification and Data Control*—Documented procedures exist for sample intake or procurement, identification, preparation, and maintenance. Samples and test data are handled in a well-defined and controlled manner (see Sections 12 and 13 for details).

11.3 *Calibration of Equipment*—Documented procedures ensure the appropriate use of reference materials or standardized test equipment to calibrate equipment and instruments so that they provide accurate results (Section 14).

11.4 *Conducting Procedures and Test Methods*—Every technical procedure for sample procurement, sample preparation, equipment calibration, or sample testing is performed only in accordance with the latest, approved version of a written standard operating procedure (SOP) (Section 15).

11.5 *Quality Control Testing*—The on-going quality of results that are provided to clients is ensured by vigorously pursuing a quality control test program. Results of quality control testing are always documented and are reviewed regularly (Section 16 and Appendix X2 and Appendix X3).

11.6 *Data Review, Control, and Reporting*—There is a documented procedure for data transfer and review to ensure that the result of each test or activity is attributed to the proper sample and is reported accurately, clearly, unambiguously, and objectively (Section 17).

11.7 *Documentation*—Documentation and record keeping are performed in a timely manner and are adequate to ensure auditors, staff, and customers that key elements of the quality management program are practiced and are accomplishing their objectives (Section 18).

11.8 *Audits and Reviews*—The laboratory conducts audits of operations (including its quality management program) on a routine basis to ensure that they are operating effectively (Section 19).

12. Collection, Intake, and Management of Test Samples

12.1 If the laboratory collects gross samples, it can demonstrate that personnel engaged in sample collection are adequately trained and able to accomplish the tasks in accordance with ASTM prescribed methods (for example, procedures in Practice D 2234).

12.2 There is a procedure to ensure that the laboratory and customer agree on the method of sampling for a particular job, and that a description of or reference to the test method used becomes a part of the sample log and final report.

12.3 Any mechanical sampling system for which the laboratory has responsibility is:

12.3.1 Tested for bias when it is put into service or when substantial modifications have been made;

12.3.2 Tested for bias on a relatively short-term, routine schedule, generally employing a simplified procedure;

12.3.3 Maintained in accordance with written procedures on a specified, routine basis;

12.3.4 Checked before and during the collection of the gross sample, including determination that sample mass is consistent with product throughput.

12.3.5 The laboratory maintains records of the condition of, maintenance and modifications performed on, and bias test results from mechanical sampling systems for which it has responsibility.

12.4 The laboratory has documented procedures for the receipt, retention, and safe disposal of test samples.

12.5 To ensure that there can be no confusion regarding the identity of samples at any time, the laboratory has a documented system for uniquely identifying samples to be tested. The laboratory assigns to each sample a unique laboratory identifier (ID), for instance, a unique number of letter/number combination. Each sample or subsample is labeled with its ID in a way that it will not be separated from the sample or rendered unreadable during its residence in the laboratory. All laboratory work records, intermediate sample containers, data sheets, and reports clearly show the laboratory ID. The ID number is included on any invoice for work on the sample.

12.6 For each sample, a sample log is maintained that contains information required for historical reference, such as account or job number, mass or volume of sample, condition of sample, type of sample, top size of sample, person or organization submitting sample, location from which the sample was collected (if known), date and time of collection (if known), date and time samples were received in the laboratory, and type of container. The sample log is also used to maintain a record of chain-of-custody of a sample.

12.7 Instructions provided by the customer about the sample are communicated to appropriate personnel and are followed by them.

12.8 Where there is any doubt as to the sample's suitability for testing, where the sample does not conform to the description provided, or where the test required is not fully specified, a specified, responsible individual consults the client for further instruction before proceeding. Laboratory personnel have specific responsibilities to establish whether the sample has been adequately prepared, or whether the client requires preparation to be undertaken in or arranged by the laboratory.

12.9 Because oxidation and moisture changes occur readily to many coal samples, documented practices are designed to minimize deterioration or damage to the test sample during storage, handling, preparation, and testing (Note 6). Where a test sample or portion of a sample is to be held after testing (for example, for reasons of record or to enable check tests to be performed later), the laboratory has storage and security arrangements that protect sample condition and integrity.

NOTE 6—Well-sealed solid containers, Mylar® or foil-lined bags preserve sample condition best. However, the condition of even a well-stored sample of coal (especially low-rank coal) almost always changes with time. Therefore, the laboratory should normally test samples within a few days of receipt. Data from analyses of samples that have been stored for more than a few days should be used cautiously.

13. Sample Preparation

13.1 Appropriate personnel can demonstrate capability to prepare samples in accordance with Method D 2013 (for coal) or Practice D 346 (for coke), or both.

13.2 Equipment for size reduction and sample division is appropriate for the task and is used appropriately.

13.2.1 Riffles or other dividers are tightly enclosed to prevent dust loss, have correct opening or chute sizes, and are used in an appropriate manner (Method D 2013).

13.2.2 Essentially all sample material is recovered from preparation equipment such as riffles, crushers, and pulverizers to prevent creation of a biased sample.

13.2.3 To prevent sample contamination, size-reduction and sample division equipment are thoroughly cleaned after each use.

NOTE 7—Vacuuming is preferred for cleaning. The use of an air blast to clean equipment is not advisable if it disperses dust into the atmosphere. Although thorough cleaning is always appropriate, it is especially important before small quantities of sample are to be processed.

13.3 Sample integrity is preserved while samples are being processed or while they are awaiting processing. In particular, samples are protected from temperature extremes and are kept in sealed containers to minimize moisture changes, oxidation, and contamination.

NOTE 8—Although not mandatory, it is desirable to conduct some tests to assess the magnitude of any incidental moisture changes that might occur during routine storage, reduction, and division of gross samples. The protocol for such tests is left to the individual laboratory. If tests are conducted, results should be documented and made accessible to customers or clients upon request. If significant changes are detected, the procedure should be modified to further minimize such inadvertent changes.

13.4 Air-drying is carried out according to procedures specified in Method 2013 or Test Method D 3302.

13.5 The magnitude of the variance attributable to routine sample preparation procedures has been measured and documented (see Annexes in Method D 2013), and is made available to customers or clients when requested. This may not be practical where customers provide only small quantities of sample. However, where coal from a single source or a limited number of sources is tested, the assessment of variance is recommended.

13.6 Sample mass at each stage of division conforms to Method D 2013.

13.7 Particle sizes of crushed or pulverized samples are checked routinely to ensure that samples meet the appropriate size criteria. Screens in size reduction equipment are frequently examined visually, preferably before each use.

13.8 Pulverized samples are mixed on a mixing wheel (a child's toy jack or "jack rock" may be placed in the container to improve mixing). To minimize size segregation (commonly sifting of finest particles to the bottom of a container), samples are not unduly shaken or tapped after mixing, and the aliquot used in a test is recovered from the container in a manner that minimizes selecting a biased size fraction. Samples removed from storage or that have been standing for extended periods are remixed before testing.

13.8.1 Test samples are maintained in well-sealed bottles or other containers until analyzed or tested and are exposed for as short a period as possible to the atmosphere.

NOTE 9—It is desirable to perform some tests to demonstrate that the container lids do seal well, thereby preventing moisture changes and oxidation. A convenient test is to determine, by weighing, if water evaporates when sealed in typical containers.

14. Calibration of Equipment

14.1 A program of calibration, verification, or validation of equipment is carried out when equipment is first placed in service or when it has been placed in service after repair or modification.

14.2 Wherever possible, calibration is performed using certified reference materials (CRMs) or certified measuring devices (for example, temperature-measuring devices).

NOTE 10—It is good practice to verify that the certified value for a reference material is reliable, for example, by comparing results from different batches of the reference material, that is, "old" bottle versus "new" bottle.

14.3 There is a documented program for frequently verifying the calibration status of test equipment between routine, scheduled recalibrations.

NOTE 11—For most test methods, it is appropriate to verify calibration with a device, reference material, or control material daily or at the beginning of each shift. For some tests, more frequent verification is required; for some, less frequent verification is appropriate. The quality manager uses his/her best judgment to define the frequency required for each test.

14.4 Calibrations and calibration verifications are conducted at different times of day, on different days of the week, and by different personnel (preferably those who test or analyze samples).

14.5 An appropriate number of determinations are used to establish or verify calibrations.

14.6 Data from calibrations and calibration verifications are maintained in statistical or control chart format (Appendix X3), and are kept within reasonable proximity to the equipment to which they apply.

14.7 Written policies define the level of nonconformance by calibration verification where action needs to be taken to correct nonconforming equipment or procedures (see "control limits" in Appendix X3). Upon detection of nonconformance, the piece of equipment is immediately removed from service

and is recalibrated or repaired prior to further use for measuring or testing. Permanent records are maintained of actions taken when nonconforming incidents are detected and remedied. Following repairs or modifications, the equipment is always calibrated.

14.8 Certified reference materials are used for calibration only and for no other purpose, unless the laboratory can demonstrate that their performance as reference materials is not compromised by such other use. If CRMs are unavailable or their expense is not warranted, then other appropriate reference materials are used. The values of the other reference materials are verified and validated in some appropriate fashion, for example, by comparison with CRMs or by having them tested by another competent laboratory or by another standardized procedure.

14.8.1 Special care is taken to minimize oxidation and changes in moisture content of any coal samples used as reference materials.

NOTE 12—When certified values of coal-based reference materials are reported on a dry basis, it is generally necessary to determine residual moisture values and make appropriate calculations to adjust the certified values from the dry to the as-determined basis. It is poor practice to use dried coal samples for calibration, as they may oxidize while drying and absorb moisture from the atmosphere after being dried. If a dried reference material is used, it is good practice to dry and weigh it in the crucible or boat to be used in the test, and to then keep them in a desiccator.

14.8.2 Reference materials are discarded when less than 5 % of the original mass remains in the container because this fraction is often size-biased.

14.9 Where traceability of test results to certified reference materials is not possible, the laboratory provides satisfactory evidence of accuracy of test results by participating in a suitable program of interlaboratory comparisons or proficiency testing (16.3).

15. Conducting Procedures and Tests

15.1 The laboratory uses appropriate, documented standard test methods and procedures for all tests and related activities, such as, sample collection, preparation, handling, transport, and storage.

15.2 All instructions, SOPs, manuals, and reference data relevant to the technical work of the laboratory are up to date and are readily available to the staff.

15.3 If the client does not specify test methods, the laboratory uses test methods, wherever possible, that national or international bodies (for example; ASTM, ISO) have standardized. The laboratory has in place a policy for advising a client when the test method requested is considered inappropriate or out of date.

15.4 Where it is necessary to use test methods that have not been established as standard, the laboratory attempts to use test methods that have been published by reputable technical organizations or that have been published in reputable scientific texts or journals. Their use is subject to agreement with the client. The test methods are fully documented and validated, and the documentation is available to the client and other recipients of the relevant reports.

15.5 Procedures are in place to ensure that only personnel who are fully trained and capable of performing a procedure or test actually perform that procedure or test.

15.6 Personnel who are responsible for performing a procedure or test method are also responsible for verifying calibration, for using control samples, and for interpreting and applying the results from control samples.

15.7 A specific form is used to record intermediate and final data for each test method (unless data are entered directly into or are captured by computer). A documented procedure is used to ensure accurate data input to the worksheet or computer.

15.8 Individuals not responsible for their initial execution, check hand calculations and data transfers.

15.8.1 For ease of reference and to facilitate auditing, a single document that shows all calculation procedures employed for all test methods is included in or referenced by the quality manual.

15.9 Where computers or automated equipment are used to capture, process, manipulate, store, retrieve, or report test data, the laboratory ensures that:

15.9.1 All appropriate requirements of this guide are complied with;

15.9.2 Computer software is documented and adequate for use, and has been verified to provide correctly calculated results and to apply them to the correct sample;

15.9.3 Procedures are followed to maintain the integrity of data entry or capture, storage (including appropriate backup), processing and transmission; and

15.9.4 Computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental conditions and operating procedures necessary to ensure the integrity and validity of test data.

15.10 For additional information on maintaining integrity in the highly computerized laboratory, see Guide E 1579.

15.11 Appropriate procedures ensure the security of data including the prevention of unauthorized access to, and the unauthorized amendment of written and computer records.

16. Quality Control Practices

16.1 To the greatest extent possible, personnel who actually perform the work are involved in the quality control process.

16.2 The laboratory generates current estimates of process capabilities for test methods. Such estimates include results from interlaboratory test programs, from process capability programs (Appendix X1) and from measurement of variability attributable to sample preparation and testing (13.5 and Method D 2013). These uncertainties are consistent with the accuracy required by the user(s), and are consistent with any standard test methods relevant to the tests. Measurement uncertainties are communicated to clients upon request.

16.2.1 Repeatability capability is established for each test method, using appropriate statistical procedures on data from replicate analyses of control material or test samples (see Appendix X1).

16.2.2 Reproducibility capability is determined through participation in one or more interlaboratory proficiency test programs (16.3).

16.2.3 Repeatability and reproducibility statistics are routinely updated and readily available (see Practice E 177 and Guide E 1323 for guidance).

16.3 *Interlaboratory Proficiency Tests*—The laboratory participates in at least one interlaboratory comparison or proficiency testing program (sometimes referred to as a "round-robin" program) that is relevant to the test methods performed (Practice E 177).

16.3.1 An SOP exists that defines who is responsible for managing participation, and how the participation is conducted and administered.

16.3.2 Data from proficiency testing programs are preserved and are summarized in statistical form or on control charts, or both. The SOP defines a procedure for routinely examining data for evidence of any persistent deviations from proficiency test averages which could indicate a bias (Note 13).

NOTE 13—Ascertaining that the laboratory's results are within some arbitrary range of the proficiency test mean value on a month-to-month basis is not a satisfactory use of proficiency test data because it does not lead to the detection of any persistent biases.

16.3.3 Written policies define statistically based levels of nonconformance ("control limits") at which action needs to be taken to modify or recalibrate a procedure based on performance in the proficiency test program. Responsibilities and procedures for taking action are explicitly defined, and a record is maintained of actions that are taken.

16.4 *Control of Quality*—The laboratory routinely ensures that every test method is in control and applies statistical techniques or control charts to interpret and communicate results from such control testing in a timely fashion.

16.4.1 Calibration verification is one part of the quality control process (Section 14).

16.4.2 Test methods are verified by routinely analyzing or testing one or more quality control materials (Appendix X2).

16.4.3 The procedures for using samples of control materials are specified in the SOP for the test method or in a more general "quality control SOP," or both.

16.4.4 Quality control measurements are performed on a timely basis to detect out-of-control situations before considerable effort is wasted in improper sample testing. When a prolonged series of measurements (sequential analyses) is made, for example specific energy or instrumental sulfur analyses, the laboratory verifies that the test method is in-control at intervals throughout the series of tests. If a number of samples are processed simultaneously (for example, a batch of crucibles are placed in a furnace at the same time for determining ash contents), the laboratory verifies that the process is in control by including a sample of a control material within the batch.

16.4.5 Data from control materials are preserved in a log or file and are summarized in statistical or control chart form using the simplest, most direct presentation for easy comprehension. Generally, control chart procedures are preferred (Appendix X3), because they provide, at a glance, information about the control history of a process. Statistical or control chart summaries are updated as soon as possible to provide timely information about the state of control.

16.5 *Out-of-Control Conditions*—Specific, statistically based criteria for recognizing out-of-control conditions, including numerical control limits that require action when exceeded are established for each test method. See Appendix X3 and Guide E 1323 for guidance on recognizing out-of-control situations.

16.5.1 Explicit instructions define what actions are to be taken when control sample data exceed control limits (Appendix X3).

16.5.2 Analysts or other technical personnel are required to notify a designated individual (for example, a supervisor or manager) when an out-of-control excursion is detected. The authorized individual decides whether the analyst can analyze and correct the problem or if additional persons need to become involved.

16.5.3 Only specified individuals have authority to modify equipment or procedures to achieve an acceptable level of control. This policy is strictly enforced to prevent casual tampering with or modification of equipment and procedures. Whenever a procedure or equipment is modified, the appropriate SOP or equipment log is immediately changed.

16.5.4 Written procedures are followed to verify that any modification does accomplish the objective, and that the process is under control.

16.5.5 After a process has been brought under control, specific steps are taken to prevent recurrence of the nonconformance.

16.5.6 A quality discrepancy record documents each excursion beyond control limits and the corrective actions taken to bring it under control. Responsibility for generating a quality discrepancy record is well defined.

16.6 The quality manager or a designee who is familiar with the quality assurance process but who does not participate in the quality control activities periodically monitors the status of the quality control system and data. The quality manager prepares a report documenting the performance to assure the staff and clients that the testing procedures are performing within expected limits.

16.7 Whenever a new test method is adopted, or new piece of equipment is placed in service or a change in the character of samples is observed, the quality control procedures are reviewed to determine if they are still appropriate.

16.8 Any changes to the quality control system are documented in appropriate manuals and SOPs.

17. Reviewing and Reporting Results

17.1 All results are reviewed to be certain that all information requested has been determined and that the work has been performed in accordance with specified test methods.

17.1.1 Only one individual or a limited number of individuals is responsible for these validation activities.

17.1.2 The responsible individual(s) has the authority, if incorrect data are suspected, to institute retesting or to check calibrations.

NOTE 14—Often, the validity of data can be checked by assessing whether different parameters are consistent with each other. For instance, laboratories that analyze coal from one mine or source can check whether specific energy (calorific value), volatile matter, and other compositional values are consistent with averages from earlier analyses when calculated

to a dry, ash-free basis. Determined specific energy should agree with specific energy calculated from elemental analyses (where available). Data for certification of sample properties can often be checked against data from samples that have been collected and tested for on-line control of the quality of coal being produced or out-loaded. Data from a lot out of a consignment can also be checked for consistency with other lots, especially, where appropriate, when calculated to a dry, ash-free basis.

17.2 The results of each test, or series of tests carried out by the laboratory are reported accurately, clearly, unambiguously, and objectively in a test report or test certificate that includes information necessary for the interpretation of the test results, including (unless it would be inappropriate):

17.2.1 A title, for example, "Test Report," or "Test Certificate";

17.2.2 The name and address of the laboratory, and the location where any test was carried out if different from the address of the laboratory;

17.2.3 Unique identification of the certificate or report (such as a serial number) and page numbers and reference to the total number of pages;

17.2.4 The name and address of the client;

17.2.5 An unambiguous identification of the sample(s) tested and any information about the sample condition that could result in questionable results;

17.2.6 Date of report, date of receipt of the test sample, and date(s) of performance of test(s);

17.2.7 Identification of the test method(s) used, or unambiguous description (as an attachment if necessary) of any agreed-upon, nonstandard method used;

17.2.8 Reference to the sample collection procedure;

17.2.9 Any deviations from, additions to, or excursions from the test method, and any other information important and relevant to a specific procedure, such as environmental conditions during sample collection;

17.2.10 An unambiguous indication of the basis (as-received, dry, or dry and ash-free) on which the data are reported (Practice D 3180);

17.2.11 A signature and title, or an equivalent identification of the person accepting responsibility for the content of the certificate or report, however produced, and an indication of how the responsible person can be contacted by the customer;

17.2.12 A statement to the effect that the results relate only to the sample(s) tested;

17.2.13 A statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory; and

17.2.14 Clear identification of results of any test methods performed by subcontractors (Section 20).

17.3 The certificate or report is arranged so that the user can easily assimilate important points. The format is carefully and specifically designed for each type of test carried out, but the headings are standardized as far as possible.

17.4 If material amendments to a test report or test certificate are required after issue, they are made only in the form of a follow-up document (or data transfer). The statement "Supplement to Test Report or Test Certificate, serial number... (or as otherwise identified)," or equivalent wording, is used. Amendments meet all the relevant requirements of this guide.

17.5 The laboratory has in place a procedure to notify clients promptly, with written confirmation, of any event that casts doubt on the validity of results given in any test report or test certificate or amendment to a report or certificate (such as the discovery of defective measuring or test equipment).

17.6 Where clients require transmission of test results by telephone, telex, facsimile, or other electronic or electromagnetic means, the laboratory follows documented procedures that adhere to the requirements of this guide and that preserve confidentiality.

18. Document Control and Record Maintenance

18.1 The laboratory maintains a readily available master document control list that defines the current revision status of all reference documents, practices, procedures, and standards in use.

18.1.1 All documents generated by the laboratory are uniquely identified, including the date of issue and revision, the revision number, the total number of pages, and the authority for issue.

18.1.2 The laboratory reviews its documented practices and procedures on a routine schedule to evaluate their suitability for the tasks undertaken.

18.1.3 Obsolete documents are promptly removed from all points of issue or use or are otherwise precluded from unintended use.

18.2 The following documentation and records are maintained and readily accessible:

18.2.1 Standard operating procedures for sample collection, sample preparation, sample testing, safety, administration, quality assurance, and quality control;

18.2.2 Records of quality control activities, especially proficiency test results, control charts (calibration and control material tests), and discrepancy reports (Appendix X3);

18.2.3 Equipment maintenance logs;

18.2.4 Personnel training and competency records;

18.2.5 Records of client complaints and their disposition; and

18.2.6 Records of all instances of nonconformance and their disposition;

18.2.7 Audit and review documents.

18.3 The laboratory retains records of test results and sample logs to suit its particular circumstances and to comply with any applicable regulations (Note 15).

NOTE 15—It is good practice to retain each analytical test report for a minimum of one year, and all original observations, calculations, and derived data until the customer has had a reasonable opportunity to submit questions concerning the results and to request return of the samples. Ninety days after the analyses have been completed and reported to the customer is normally adequate time for the analytical results to be reviewed and rechecked if necessary.

18.4 Client-related records, including data held on computers, are held secure and in confidence to the client.

19. Audits and Reviews

19.1 *Audits*—At appropriate intervals, the activities of the laboratory are formally audited to verify that its operations comply with the requirements of the quality management system.

19.1.1 A document in the quality manual describes the audit policy(s).

19.1.2 Knowledgeable personnel who are not directly involved with the daily activities conduct internal audits on a predetermined, fairly frequent schedule (for example, quarterly). Internal audits are scheduled so that different parts of the system are audited at different times. Internal audits are carried out with the aid of a checklist (see Practice D 4182 for an example).

19.1.3 Audits by external examiners are conducted on a more long-term basis (for example, yearly), and may also use a checklist.

19.1.4 The laboratory has in-place procedures to take corrective action immediately when audit findings cast doubt on the correctness of practices or the validity of any test results, and immediately notifies, requesting written confirmation, any client whose samples or results may have been affected.

19.1.5 A written procedure defines who is responsible for taking action when an audit identifies a problem. This procedure also defines who is responsible for ensuring that the action is taken; that is, there is a specific "sign-off" process.

19.1.6 Records are retained of the results of audits, including specific actions taken to correct deficiencies.

19.2 *Reviews*—A written policy provides for an annual review of the quality management program by the laboratory management. This is commonly done in conjunction with an audit, and uses audit results. The policy defines specifically the procedures that management should follow to maximize the effectiveness of the review.

19.2.1 The review takes into account reports from managerial and supervisory personnel, the outcome of audits performed since the last review, calibration and quality control results (especially the results of interlaboratory comparisons or proficiency tests), any changes in volume and type of work performed, feedback from clients, and other factors.

19.2.2 Results are used in corporate planning programs, including setting goals, objectives, and action plans for the following year.

19.2.3 Review results and any corrective actions that arise from them are documented. The person responsible for quality shall ensure that these actions are discharged within an agreed upon time frame.

20. Subcontracting Tests to Outside Agencies

20.1 The laboratory advises its clients in writing of its intention to subcontract any portion of the testing to another laboratory.

20.2 The laboratory assures itself that a subcontractor is competent to perform the activities in question and that it complies with the same criteria of competence as the laboratory in respect to the work being subcontracted.

20.3 Wherever possible, subcontracts are placed with organizations that use good quality management practices.

20.4 The laboratory documents details of its investigation of the competence and compliance of its subcontractors and maintains a register of all capable subcontractors.

21. Outside Support

21.1 Where the laboratory procures outside services in support of tests, the laboratory uses only those services that are of adequate quality to sustain confidence in the laboratory's results.

21.2 Where no independent assurance of the quality of outside support services is available, the laboratory has or develops procedures to ensure that the outside organization complies with requirements specified by the laboratory.

21.3 The laboratory maintains records of the performance of all organizations from whom it obtains support services required for tests.

22. Complaints

22.1 The laboratory has documented policies and procedures for the resolution of complaints received from clients or other parties about the laboratory's activities.

22.2 A record is maintained of all complaints and of the actions taken by the laboratory to resolve them.

22.3 Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with its policies or procedures, or with the requirements of this guide or otherwise concerning the quality of the laboratory's tests, the laboratory promptly audits those areas of activity and responsibility.

23. Keywords

23.1 quality assurance; quality control; quality management

APPENDIXES

(Nonmandatory Information)

X1. ESTABLISHING PROCESS CAPABILITY

X1.1 *Introduction*—For each test method in the laboratory, data should be available to demonstrate that the laboratory can produce test results consistent with some specified minimum levels of accuracy and precision. Practice E 177 provides general guidelines for understanding and determining process capability.

X1.2 *Selecting Specifications for a Test Method*—Management should decide what level of accuracy and precision the laboratory should achieve routinely, that is, what the process capability is when the process is in a state of statistical control.

X1.2.1 At a minimum, the process capability specification should be based on ASTM-established repeatability and reproducibility limits. These limits, based on tests performed under the auspices of ASTM Committee D-5, indicate the precision that the test method is capable of achieving in other laboratories. ASTM precision limits are defined by data collected under repeatability conditions (same sample, same operator, same equipment, in as short a period of time as practical). Actual capability may be less when different operators test different samples in different equipment on different days.

X1.2.1.1 For special situations as required by a customer, the laboratory may have to achieve a process capability better than ASTM repeatability and reproducibility. This will generally require that sources of variability will need to be investigated and eventually controlled to tolerances better than those defined in the ASTM standard test method.

X1.3 *Measuring Laboratory Process Capability*—Process capability can be determined by testing one or more samples over an extended period of time, with different equipment, if available, and different operators, if more than one routinely performs tests with a given test method.

X1.3.1 Use the following guidelines to design a program to develop capability data.[5] Data from an example program are shown in Tables X1.1 and X1.2. Control charts from the data are shown in Figs. X1.1 and X1.2.

TABLE X1.1 Example Data from a Process Capability Study

Subgroup	Date	Operator	X	For Subgroups $\bar{X}$	For Subgroups R	For Subgroups s
1	4-March	1	7.23			
1	4-March	1	7.21	7.22	0.02	0.0141
2	4-March	2	7.35			
2	4-March	2	7.33	7.34	0.02	0.0141
3	4-March	3	7.38			
3	4-March	3	7.23	7.31	0.15	0.1061
4	5-March	1	7.37			
4	5-March	1	7.16	7.27	0.21	0.1485
5	5-March	2	7.28			
5	5-March	2	7.39	7.34	0.11	0.0778
6	5-March	3	7.25			
6	5-March	3	7.27	7.26	0.02	0.0141
7	6-March	1	7.35			
7	6-March	1	7.36	7.36	0.01	0.0071
8	6-March	2	7.31			
8	6-March	2	7.29	7.30	0.02	0.0141
9	6-March	3	7.28			
9	6-March	3	7.38	7.33	0.10	0.0707
10	7-March	1	7.32			
10	7-March	1	7.34	7.33	0.02	0.0141
11	7-March	2	7.41			
11	7-March	2	7.18	7.30	0.23	0.1626
12	7-March	3	7.17			
12	7-March	3	7.26	7.22	0.09	0.0636
13	8-March	1	7.52			
13	8-March	1	7.29	7.41	0.23	0.1626
14	8-March	2	7.28			
14	8-March	2	7.36	7.32	0.08	0.0566
15	8-March	3	7.25			

TABLE X1.2 Summaries of Data and Control Chart Lines from Table X1.1

Subgroup Number	R	UCL for R	$\bar{R}$	$\bar{X}$	$\bar{\bar{X}}$	UCL for X	LCL for X
1	0.02	0.30	0.091	7.22	7.30	7.47	7.13
2	0.02	0.30	0.091	7.34	7.30	7.47	7.13
3	0.15	0.30	0.091	7.31	7.30	7.47	7.13
4	0.21	0.30	0.091	7.27	7.30	7.47	7.13
5	0.11	0.30	0.091	7.34	7.30	7.47	7.13
6	0.02	0.30	0.091	7.26	7.30	7.47	7.13
7	0.01	0.30	0.091	7.36	7.30	7.47	7.13
8	0.02	0.30	0.091	7.30	7.30	7.47	7.13
9	0.10	0.30	0.091	7.33	7.30	7.47	7.13
10	0.02	0.30	0.091	7.33	7.30	7.47	7.13
11	0.23	0.30	0.091	7.30	7.30	7.47	7.13
12	0.09	0.30	0.091	7.22	7.30	7.47	7.13
13	0.23	0.30	0.091	7.41	7.30	7.47	7.13
14	0.08	0.30	0.091	7.32	7.30	7.47	7.13
15	0.10	0.30	0.091	7.28	7.30	7.47	7.13

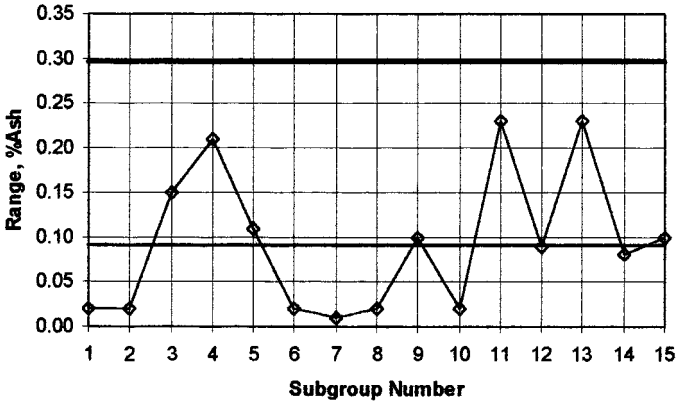

FIG. X1.1 Control Chart for Ranges (Data from Tables X1.1 and X1.2)

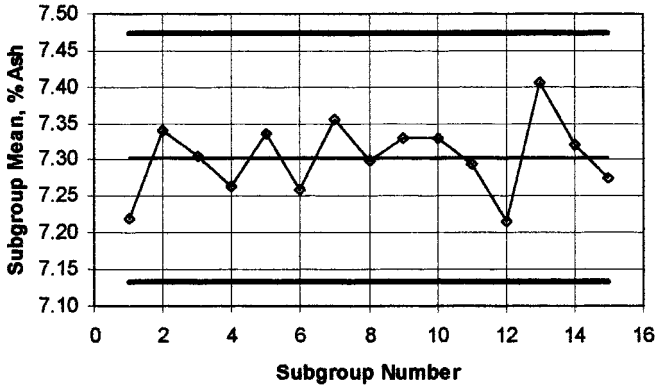

FIG. X1.2 Control Chart for Means (Data from Tables X1.1 and X1.2)

X1.3.2 Select a sample that represents the quality of coal that is normally tested by the laboratory. If the laboratory routinely tests a variety of samples, then measurement of process capability might be performed on a number of samples that span the range found in samples that are routinely tested. Be certain that the mass of the sample will be adequate for the many tests to be performed.

X1.3.3 Test the sample over a period of days using all operators that normally perform the test and each piece of

equipment that is routinely used. Each operator should perform the test on each piece of equipment at least twice in as short a period of time as is practical (the replicated, short-term test data from a "subgroup"). Each operator should perform subgroup tests on different days and at different times of the day. There should be at least twelve subgroup tests. Number each subgroup and maintain information on date, time of day, equipment, and operator.

X1.3.4 Calculate the mean ($\bar{X}$) and range (R) for each subgroup using the following equations:

$$\bar{X} = (X_1 + X_2 + ... + X_n)/n \quad \text{(X1.1)}$$

$$R = X_{\text{highest}} - X_{\text{lowest}} \quad \text{(X1.2)}$$

where:
X_i = individual measurement responses and
n = number of tests in a subgroup.

X1.3.5 To establish a meaningful measure of process capability, the test method must be in a state of statistical control when the data are collected. Control charts are used to identify any evidence that points to process variability or the process average not operating at a constant level. After the data are collected, construct one control chart for the mean values, and one for the ranges. Plot $\bar{X}$ and R values horizontally against the subgroup number on their respective charts, and connect points with lines to help visualize patterns.

X1.3.6 Calculate, from all subgroup ranges, the average range ($\bar{R}$). Also, calculate the overall average of the test method subgroup averages ($\bar{\bar{X}}$) using the following equations:

$$\bar{R} = (\bar{R}_1 + \bar{R}_2 + ... \bar{R}_k)/k = 0.091 \quad \text{(X1.3)}$$

$$\bar{\bar{X}} = (\bar{X}_1 + \bar{X}_2 + ... \bar{X}_k)/k = 7.303 \quad \text{(X1.4)}$$

where:
R_i = individual range values and
k = number of subgroups.

NOTE X1.1—Numerical values are from example data in Table X1.1.

X1.3.7 Calculate upper and lower control limits (*UCL*, *LCL*) using the following equations:

$$UCL\ (\bar{R}) = D_4\bar{R} = 0.296 \qquad UCL\ (\bar{\bar{X}}) = \bar{\bar{X}} + A_2\bar{R} = 7.474 \quad \text{(X1.5)}$$

$$LCL\ (\bar{R}) = D_4\bar{R} \qquad LCL\ (\bar{\bar{X}}) = \bar{\bar{X}} - A_2\bar{R} = 7.133 \quad \text{(X1.6)}$$

where:

N	2	3	4	5
D_4	3.267	2.575	2.282	2.115
D_3	[A]	[A]	[A]	[A]
A_2	1.880	1.023	0.729	0.577

[A]For sample sizes below 7, the LCL_R would technically be a negative number; in those cases, there is no lower control limit. Where $n > 5$, consult Manual 7, Table 2.

X1.3.8 Draw lines for $\bar{\bar{X}}$, $\bar{R}$, and the control limits on their respective charts.

X1.4 Analyze each chart separately.

X1.4.1 The R chart is analyzed first. Data points are compared to control limits to detect points out of control or unusual patterns or trends. Any point beyond a control limit indicates that the process was out of control at that point, and is a signal for immediate analysis of the operation. Any unusual patterns indicating a progressive change should be considered a sign that the process conditions changed during the test program.

X1.4.2 For any indication of special causes (out-of-control situations) conduct an analysis of the process and attempt to discover the cause. Correct the condition and prevent it from happening again.

X1.4.3 Delete data from any subgroups affected by special causes. Recalculate the control limits and drop any data points from the $\bar{X}$ chart for subgroups that were removed from the R chart.

X1.4.4 Analyze the $\bar{X}$ chart, using the same criteria that were applied to the R chart. Find and correct special causes and remove offending subgroups. If necessary, make additional analyses of subgroups to increase the number of subgroups, recalculate the control limits, and check that the new data are in control. Continue only after it is certain that all data were collected while the process was in a state of statistical control.

X1.5 Calculate the process standard deviation as follows:

$$\hat{\sigma} = \frac{\bar{R}}{d_2} = 0.079 \quad \text{(X1.7)}$$

where:

n	2	3	4	5
D_2	1.13	1.69	2.06	2.33

X1.6 Calculate the process capability in terms of the number (Z) of process standard deviation units ($\hat{\sigma}$) in the table of standardized normal distribution to estimate the proportion of output that will fall within any specification limit (SL).

X1.6.1 Approximately 95 % of the results from repeated tests on a material should be within $\pm 1.96\ \hat{\sigma}$ of the mean.

X1.6.2 Essentially 100 % of repeated test results should be within $\pm 3\ \hat{\sigma}$ of the mean.

X1.7 Calculate the repeatability for the test method as performed in the laboratory.

X1.7.1 Calculate the subgroup standard deviation, s, for each subgroup as follows:

$$s = \sqrt{\{[\sum_1^n (X_i - \bar{X})^2] / (n-1)\}} \quad \text{(X1.8)}$$

where:
X_i = individual measurements in a subgroup,
$\bar{X}$ = grand average for the subgroup, and
n = number of test results in a subgroup.

X1.7.2 Calculate the repeatability standard deviation, s_r, as follows:

$$s_r = \sqrt{[\sum_1^k s^2/k]} = 0.084 \quad \text{(X1.9)}$$

where:
s = subgroup standard deviations (k of them).

X1.7.3 Calculate the laboratory repeatability, r, for the process as $r = 2.77\ s_r$ (example, $r = 0.236$). (See Section 27 of Practice E 177 for derivation of constant 2.77.)

X2. CONTROL MATERIAL (CM)

X2.1 *Desirable Properties*:

X2.1.1 A control material should have property values similar to samples routinely tested in the laboratory.

X2.1.2 It should be homogeneous, lest variance of the results reduces the sensitivity of the quality control procedure designed to detect changes in the measurement process.

X2.1.3 It should be stable through time. With coal, moisture changes and oxidation may result in long-term changes. Minimize the opportunities for this to happen as much as possible.

X2.2 *Choosing a Control Material*:

X2.2.1 The choice of a CM is the responsibility of laboratory management.

X2.2.2 For laboratories that test coal or coke with consistent properties, a single CM is acceptable; for laboratories monitoring a wide variety of coals, a set of CMs covering the range of expected values is more appropriate.

X2.2.3 For laboratories engaged in the analysis of coal or coke, the most suitable and economic CM is often a sample that has been submitted for testing. It should have sufficient mass to last for a reasonable period (for example, 30 days; see 2.3), and it should exhibit the desirable characteristics previously listed.

X2.2.4 Reference materials can also be used for control purposes if their use does not compromise their integrity as reference materials. Proficiency test samples also make excellent CMs if the sample is large enough.

X2.2.5 A sample that was tested on a previous day or previous shift may be retested as a form of CM. In that case, the construction of a control will differ slightly from that shown in Appendix X3.

X2.3 *Preparing a Control Material*:

X2.3.1 Coal samples that are to be used as CMs should be equilibrated with the laboratory atmosphere so that the moisture content varies as little as possible during its use.

X2.3.2 The particle size should be appropriate for the test method(s) where it is to be used.

X2.3.3 If the property of interest and the moisture content remain stable with time, the laboratory should prepare as much CM as can be prepared in a homogeneous state. If the property or moisture content may change with time, the laboratory should prepare as large a quantity of the material as will be needed before the property significantly changes.

X2.3.4 A new quantity of CM should be obtained or prepared in advance of exhausting the old one, so that it can be used simultaneously with the old one and a new control chart started before the old material is exhausted (Appendix X3, section X3.2.2).

X2.4 *Using the Control Material*:

X2.4.1 The CM may be tested in the same batch as test samples, or at intervals during sequential analyses/tests of test samples (see Section 16 of this guide for amplification of these concepts).

X2.4.2 Data from CM test methods should be recorded and maintained in a document or computer file, and the record should indicate with which test samples the CM was tested.

X2.4.3 Data should be placed into a control chart in order to see any progressive changes in properties or procedures (Appendix X3).

X2.4.4 The CMs should be discarded when about 5 % remains in the container or when properties begin to change significantly.

X3. USING CONTROL CHARTS TO MONITOR PROCESS CONTROL

X3.1 *Introduction*:

X3.1.1 Commonly, a sample of a control material is tested with a batch of client test samples or at intervals between *'X'* numbers of client samples (16.4.4). A control chart is a method for displaying data from these tests of a CM (or a reference material). A control chart provides good visual feedback to the analyst about how well the system is being controlled.

X3.1.2 Control charts generally plot a horizontal line representing a mean from a number of tests of the CM or a compositional value established by some other standard test method (for example, if the CM is a sample from an interlaboratory study). Above and below the central value are lines representing control limits. These are established from statistical calculations representing the variability inherent in the testing procedure when it is in control, that is, when variations are due to relatively small chance causes. The values from tests of the control material are plotted consecutively from left to right on this graph.

X3.1.3 The null hypothesis being tested by each CM data point is: “The testing process was in control while the sample batch was tested, or was in control during the interval since the last test of the CM.” As long as the points remain within the control limits, the null hypothesis is accepted as true. If a test value (or in some charts, a mean of two or more test values) falls outside of one of the control limits, the null hypothesis is rejected and it is presumed that something has happened to the system to cause it to go out of control. Certain corrective actions are then required.

X3.1.4 There are many types of control charts of varying degrees of complexity. For coal and coke control materials, a

simple chart that requires only one test of the CM in a batch of samples or between *'X'* number of client sample tests is generally adequate.

X3.2 *Creating the Control Chart*:

X3.2.1 The following control chart procedure uses a chart for individuals, *X*, and one for a moving range, *R*, of two consecutive observations, with a standard given for the control limits.

X3.2.2 When a new CM is first begun, test it five times for the property of interest while the test method is known to be in control, for example, through the concurrent or simultaneous testing of a reference material or another CM (which could be one that is to be retired).

X3.2.3 Calculate the mean of the first and second data points, then the mean for the first, second, and third points. Continue this process as new data are acquired. For instance, the mean for the fifth point is calculated as the sum of all five points divided by five.

X3.2.4 For the second data point, calculate the range as the value from the first data point (X_1) minus the second (X_2). Progressively calculate the ranges of each successive pair, for example $X_2 - X_3$ for Point 3, $X_3 - X_4$ for Point 4, and so forth.

X3.2.5 Table X3.1 is an example showing data derived from 40 tests for ash content in a CM.

X3.2.6 On graph paper or by using a computer spreadsheet or graphing program, construct a chart for the ranges on which the following statistics are plotted horizontally for each CM test beginning with Test 6 (see Fig. X3.1):

X3.2.6.1 Individual range values;

X3.2.6.2 Upper control limit equal to 3.868 times $\hat{\sigma}$ obtained from the process capability study; and

X3.2.6.3 Central line equal to 1.128 times $\hat{\sigma}$ obtained from the process capability study.

X3.2.7 Construct a chart for the individuals, that is, the measured value of the property of interest. Beginning with Point 6, plot the following (see Fig. X3.2):

X3.2.7.1 An upper control limit equal to $\bar{X} \times 3\ \hat{\sigma}$ obtained from the process capability study;

X3.2.7.2 A lower control limit equal to $\bar{X} - 3\ \hat{\sigma}$ obtained from the process capability study;

X3.2.7.3 A central line represented by the cumulative mean.

X3.2.8 To catch any problems quickly, plot the individual and range values as soon as practical after making the analyses.

X3.3 *Excursions*—Look for excursions immediately when the data are plotted. An excursion is a situation where control or calibration verification data indicate that there may be a problem with the control of a procedure. Criteria for recognizing an excursion vary depending upon the type of control chart. For the range and individual types described previously the following rules apply (see Figs. X3.1 and X3.2):

X3.3.1 One CM data point (range or individual) falls outside a control limit;

X3.3.2 A run of 7 consecutive values from a CM is on one side of the central line of the control chart;

X3.3.3 Ten out of eleven values are on one side of the central line;

TABLE X3.1 Data from Tests of Ash Content in a Control Material

Property: Ash		CM #: 3146		Standard Deviation of Process Capability: 0.08			
Test Number	Test Result	Cumulative Mean	Abs. Differ. (Range)	Center Line for *R*	*LCL* for *X*	*UCL* for *X*	*UCL* for *R*
1	8.23						
2	8.21	8.22	0.02	0.10	7.98	8.46	0.30
3	8.27	8.24	0.06	0.10	8.00	8.48	0.30
4	8.42	8.28	0.15	0.10	8.04	8.52	0.30
5	8.22	8.27	0.20	0.10	8.03	8.51	0.30
6	8.17	8.25	0.05	0.10	8.01	8.49	0.30
7	8.11	8.23	0.06	0.10	7.99	8.47	0.30
8	8.28	8.24	0.17	0.10	8.00	8.48	0.30
9	8.10	8.22	0.18	0.10	7.98	8.46	0.30
10	8.19	8.22	0.09	0.10	7.98	8.46	0.30
11	8.29	8.23	0.10	0.10	7.99	8.47	0.30
12	8.17	8.22	0.12	0.10	7.98	8.46	0.30
13	8.32	8.23	0.15	0.10	7.99	8.47	0.30
14	8.28	8.23	0.04	0.10	7.99	8.47	0.30
15	8.29	8.24	0.01	0.10	8.00	8.48	0.30
16	8.35	8.24	0.06	0.10	8.00	8.48	0.30
17	8.24	8.24	0.11	0.10	8.00	8.48	0.30
18	8.27	8.25	0.03	0.10	8.00	8.49	0.30
19	8.12	8.24	0.15	0.10	8.00	8.48	0.30
20	8.15	8.23	0.03	0.10	7.99	8.47	0.30
21	8.24	8.23	0.09	0.10	7.99	8.47	0.30
22	8.28	8.24	0.04	0.10	8.00	8.48	0.30
23	8.23	8.24	0.05	0.10	8.00	8.48	0.30
24	8.23	8.24	0.00	0.10	8.00	8.48	0.30
25	8.19	8.23	0.04	0.10	7.99	8.47	0.30
26	8.17	8.23	0.02	0.10	7.99	8.47	0.30
27	8.26	8.23	0.09	0.10	7.99	8.47	0.30
28	8.18	8.23	0.08	0.10	7.99	8.47	0.30
29	7.97	8.22	0.21	0.10	7.98	8.46	0.30
30	8.27	8.22	0.30	0.10	7.98	8.46	0.30
31	8.10	8.22	0.17	0.10	7.98	8.46	0.30
32	8.08	8.22	0.02	0.10	7.97	8.46	0.30
33	8.25	8.22	0.17	0.10	7.98	8.46	0.30
34	8.30	8.22	0.05	0.10	7.98	8.46	0.30
35	8.15	8.22	0.15	0.10	7.98	8.46	0.30
36	8.26	8.22	0.11	0.10	7.98	8.46	0.30
37	8.30	8.22	0.04	0.10	7.98	8.46	0.30
38	8.31	8.22	0.01	0.10	7.98	8.46	0.30
39	8.23	8.22	0.08	0.10	7.98	8.46	0.30
40	8.27	8.22	0.04	0.10	7.98	8.46	0.30

X3.3.4 Seven consecutive points form a continuous trend upward or downward in a control chart;

X3.4 *Dealing with Single-Point Excursions*—When a single value exceeds a control limit, the actions defined in this section should be taken. If these do not discover a reason, then the analyst should assume that the system is out-of-control, and the actions in X3.6 should be taken:

X3.4.1 Ensure that CM test values have been attributed to the proper CM, and that the proper material was used;

X3.4.2 Where possible, ensure that numbers (weights, instrument readings, and so forth) were copied correctly from working data sheets to computer files or calculation sheets;

X3.4.3 Check that test conditions are nominal; for example, ensure that equipment temperature is correct (where applicable), that appropriate residence time was used, that atmospheres are correct, and so forth;

X3.4.4 Review notes on any worksheets that might indicate a problem source;

X3.4.5 Ensure that any hand calculations were correctly done;

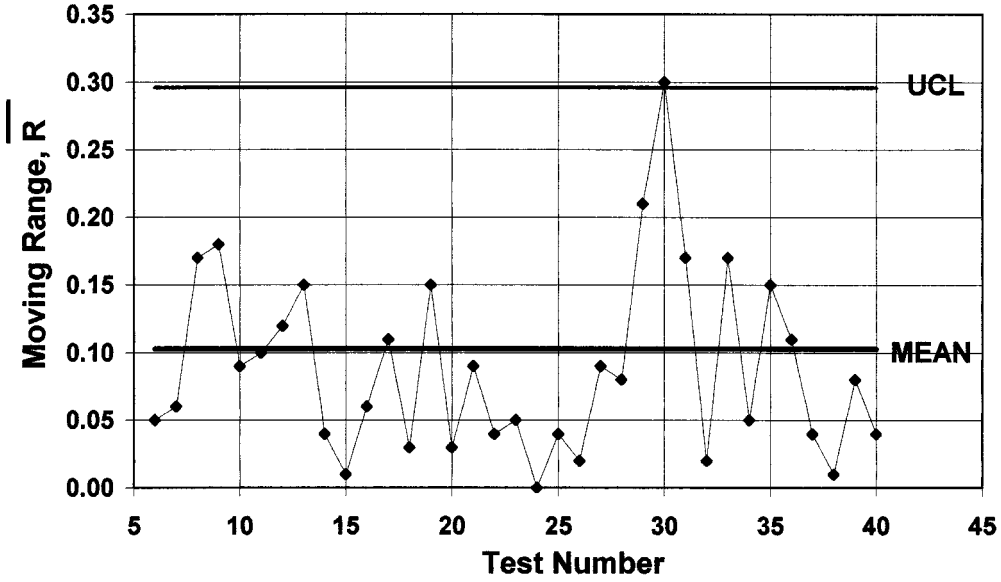

FIG. X3.1 Example of Control Chart for Ranges (Data from Table X3.1)

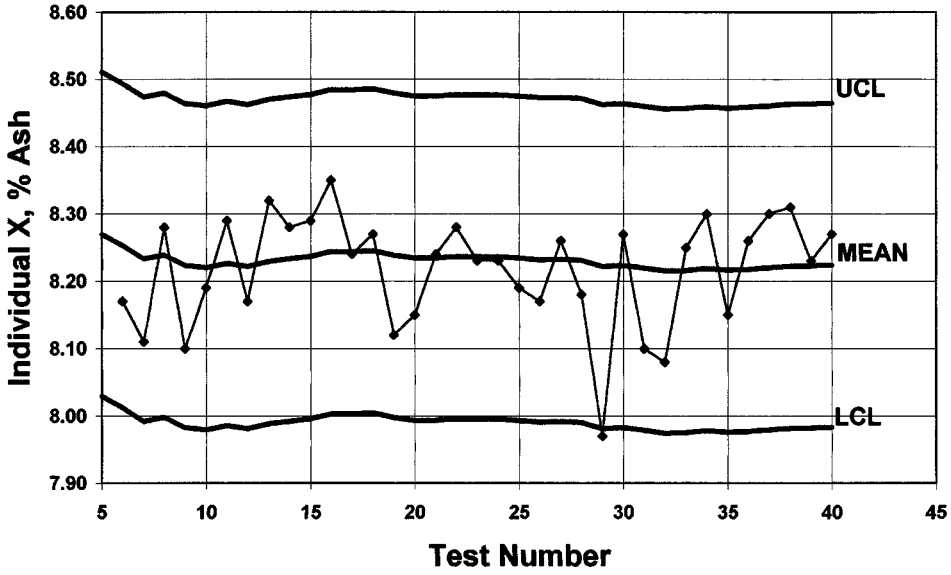

FIG. X3.2 Example of Control Chart for Individuals (Data from Table X3.1)

X3.4.6 If the CM was tested within a batch of test samples (for instance, a batch of samples placed in a moisture oven), retest the CM and all test samples in the batch. If the excursion persists, then technical evaluations specific to the test procedure must be made (X3.6) to determine the cause of the excursion before any further client samples are analyzed, and all test results from both batches should be discarded;

X3.4.7 If the CM was tested at intervals between test samples, retest the CM. If the problem was a single point out of control, and the retest indicates no problem, substitute the value for the erroneous one in the control chart and accept test results obtained in the interim since the CM last indicated no problem. If the retest of the CM continues to indicate an excursion beyond control limits, conduct technical evaluations specific to the equipment or procedure to determine the cause (X3.6), and discard client sample results obtained since the last CM was tested.

X3.5 *Dealing with Progressive (Pattern) Changes*—It is difficult to provide specific steps to follow when patterns (X3.3.2-X3.3.4) indicate that some progressive change has occurred. Often, it is too late to reconstruct what has happened in time to reanalyze affected client sample. At the least, if client sample results have been transmitted, the client should be informed that a problem may have existed during the analysis of his samples. Technical evaluations should be made.

X3.6 *Technical Evaluations*—Options for discovering the source of an out-of-control situation will vary with the type of

excursion and the test method. Some guidance for evaluating problems can normally be deduced from steps in the specific test method. It would also be prudent to have developed beforehand a general "response-to-excursions" SOP. The test procedure must be tracked from beginning to end and a series of checkpoints established to determine the location of the error.

X3.6.1 Once the problem has been corrected, the procedure should be validated using reference materials or control materials. Any test samples in the same batch or that were tested since the system was know to be in control should be retested.

X3.7 *Preventing Recurrence*—Whenever an excursion has occurred, and a solution has been found, action should be taken to prevent recurrence of the situation. The analyst (and, where appropriate, a supervisor) should spend the necessary time to consider in detail what actions could be taken to minimize the opportunity for such a problem to recur, and then those actions should be taken.

X3.8 *Documentation*—It is extremely important that all excursions be documented thoroughly. There should be a logbook for each test procedure, in which records are maintained about the occurrence of control excursions. The quality manager or supervisor and the analyst should record in the logbook the details of how the problem was solved and what was done to prevent recurrence. The record should include all data generated during the correction process and any validation results obtained on reference materials or control materials. The information should be circulated to all affected supervisors and laboratory personnel. Excursion reports should be maintained in a central repository or near the location of the analysis equipment, or both.

X3.9 *Responsibilities*

X3.9.1 Generally, the analyst is responsible for initial detection of any excursion. The analyst should record the occurrence in the procedure logbook. The analyst should then follow the initial procedures (previously stated) for finding causes of excursions. If the cause is discovered and corrected, it should be documented in the logbook.

X3.9.2 If the analyst concludes that the client sample data associated with the CM excursion may be incorrect, or if there is no simple explanation for the excursion, the analyst should contact the supervisor or another superior.

X3.9.3 The supervisor should work with the analyst to further investigate the cause and to decide whether to accept or reject any affected test sample results.

X3.9.4 The supervisor should record sufficient information in the logbook or in an excursion report to familiarize the technical manager with all of the steps that have been taken to find the source of the excursion and to correct it.

X3.9.5 If no solution can be found, the supervisor should contact the technical manager for further instructions.

X3.9.6 The technical or quality manager is responsible for communicating information about the excursion to appropriate analysts or other affected personnel.

X3.9.7 The laboratory manager is responsible for communicating information to clients if results that have already been reported are suspected to be incorrect because of the discovery of an out-of-control situation.

X4. PROCEDURES FOR IMPROVING PRECISION OR ACCURACY OF TEST RESULTS

X4.1 *Introduction*—Although complete coverage of the topic of improving analytical methods is beyond the scope of this guide, several aspects of statistical or supervisory control have the effect of improving precision, accuracy, or reliability of analytical results without making substantial changes in analytical or test methods.

X4.2 *Replication*—It is a simple statistical fact that, when the procedure is in control, the reliability of the mean of a number of independent measurements improves as the number of measurements increases. In general, for values randomly obtained from a normally distributed population of values, the expected reliability of the mean of n readings is $\sqrt{n}$ times the reliability of a single reading. A second important fact is that the first few replications improve the reliability much more than would the same number of additional replications. The improvement from averaging four observations rather than reporting an individual observation is to increase the reliability of the reported (mean) result two times; but averaging nine replicates increases the reliability by only three times over a single observation. The nature of analyzing or testing materials is such that care is to be used when improved reliability of results is sought in this manner.

X4.2.1 First, it is generally not difficult to justify making two, or even four, replicate observations for measurements in very important situations. It is rarely justified to perform the same assay more than nine times.

X4.2.2 The second consideration is even more important. Truly independent replications are obtained only by replication of the entire test process (which includes sampling and sample preparation). Replications of less than the entire process usually produces much less increase in the reliability of the final reported value than would be expected from the square root law. By way of illustration, the mean of determinations for calorific values of two separate samples taken from a rail car of coal will probably be closer to the true calorific value of coal in that car than the value produced from the mean of two successive readings of a calorimeter from one of those samples.

X4.2.3 Replicating analyses to obtain greater accuracy is no substitute for practicing good quality control.

X4.3 *Improvements in Equipment and Staff Skills*:

X4.3.1 Proper maintenance of equipment with periodic checks on its performance using reference materials is necessary to minimize the instrumental- or equipment-related errors. Each piece of equipment has a logbook in which is recorded

information about the equipment performance, including maintenance performed upon the equipment; instances of a change in supply of carrier or reactant gases; pertinent calibrations or associated calibrations; and dates, times, and initials of the person(s) performing the task.

X4.3.2 Proper training of personnel in good techniques and in understanding the significance of each step performed is necessary if a laboratory is to approach the accuracy that test methods and equipment are capable of delivering. This is the reason for requiring ongoing training of personnel. Involving all laboratory personnel in the planning and implementation of a quality-control program can instill an attitude of pride in good work and confidence in one's ability to perform well.