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An American National Standard

Standard Practice for Quality Management Systems in Petroleum Products, Liquid Fuels, and Lubricants Testing Laboratories[1]

This standard is issued under the fixed designation D6792; 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 practice covers the establishment and maintenance of the essentials of a quality management system in laboratories engaged in the analysis of petroleum products, liquid fuels, and lubricants. It is designed to be used in conjunction with Practice D6299.

NOTE 1—This practice is based on the quality management concepts and principles advocated in ANSI/ISO/ASQ Q9000 standards, ISO/IEC 17025, ASQ Manual,[2] and ASTM standards such as D3244, D4182, D4621, D6299, D6300, D7372, E29, E177, E456, E548, E882, E994, E1301, E1323, STP 15D,[3] and STP 1209.[4]

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[5]

D630 Specification for Olive Oil Chip Soap (Type A, Straight; Type B, Blended) (Withdrawn 1979)[6]

D3244 Practice for Utilization of Test Data to Determine Conformance with Specifications

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4182 Practice for Evaluation of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke (Withdrawn 2010)[6]

D4621 Guide for Quality Management in an Organization That Samples or Tests Coal and Coke (Withdrawn 2010)[6]

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants

D6617 Practice for Laboratory Bias Detection Using Single Test Result from Standard Material

D7372 Guide for Analysis and Interpretation of Proficiency Test Program Results

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E456 Terminology Relating to Quality and Statistics

E548 Guide for General Criteria Used for Evaluating Laboratory Competence (Withdrawn 2002)[6]

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E994 Guide for Calibration and Testing Laboratory Accreditation Systems General Requirements for Operation and Recognition (Withdrawn 2003)[6]

E1301 Guide for Proficiency Testing by Interlaboratory Comparisons (Withdrawn 2012)[6]

E1323 Guide for Evaluating Laboratory Measurement Practices and the Statistical Analysis of the Resulting Data

E2476 Guide for Risk Assessment and Risk Control as it

[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.94 on Coordinating Subcommittee on Quality Assurance and Statistics.

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[2] "Quality Assurance for The Chemical and Process Industries: A Manual of Good Practices," 1987, available from American Society for Quality (ASQ), 600 N. Plankinton Ave., Milwaukee, WI 53203. www.asq.org.

[3] ASTM STP 15D, *ASTM Manual on Presentation of Data and Control Chart Analysis*, ASTM International, W. Conshohocken, PA.

[4] ASTM STP 1209, *ASTM Manual on Total Quality Management*, ASTM International, W. Conshohocken, PA.

[5] 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.

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

***A Summary of Changes section appears at the end of this standard**

Impacts the Design, Development, and Operation of PAT Processes for Pharmaceutical Manufacture

2.2 *ISO Standards:*[7]

ISO Guide 30 Terms and Definitions Used in Connection with Reference Materials

ISO Guide 73 Risk Management – Vocabulary

ISO 4259-3 Petroleum and related products – Precision of measurement methods and results – Part 3: Monitoring and verification of published precision data in relation to methods of test

ISO 4259-4 Petroleum and related products – Precision of measurement methods and results – Part 4: Use of statistical control charts to validate 'in-statistical-control' status for the execution of a standard test method in a single laboratory

ISO 9000 Quality Management Systems – Fundamentals and Vocabulary

ANSI/ISO/ASQ Q9000 Quality Management System Standards

ISO/IEC 17000 Conformity Assessment – Vocabulary and general principles

ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories

ISO 31000 Risk Management – Guidelines

2.3 *Other Standards:*

40 CFR 80 Regulation of Fuels and Fuel Additives[8]

3. Terminology

3.1 *Definitions:*

3.1.1 For a more extensive list of terms used by laboratories engaged in the analysis of petroleum products, liquid fuels, and lubricants refer to Terminology D4175.

3.1.2 More extensive lists of terms related to quality management systems are found in ISO 9000 and ISO/IEC 17000, terms related to risk management are found in ISO Guide 73 and ISO 31000, and terms related to quality and statistics are found in Practice D630 and Terminology E456.

3.1.3 *accepted reference value, ARV, n*—a value that serves as an agreed upon reference for comparison, and which is derived as: (*1*) a theoretical or established value, based on scientific principles, (*2*) an assigned value, based on experimental work of some national or international organization such as the U.S. National Institute of Standards and Technology (NIST), or (*3*) a consensus value, based on collaborative experimental work under the auspices of a scientific or engineering group. **E456**

3.1.4 *accuracy, n*—the closeness of agreement between a test result and an accepted reference value. **E456**

3.1.5 *audit, n*—a systematic examination of a laboratory's quality management system documentation and related activities by an internal or external team to determine conformance to the applicable quality management system standard, such as described in this practice.

3.1.6 *bias, n*—the difference between the population mean of the test results and an accepted reference value. **E456**

3.1.7 *calibration standard, n*—a material with a certified value for a relevant property, issued by or traceable to a national organization such as NIST, and whose properties are known with sufficient accuracy to permit its use to evaluate the same property of another sample.

3.1.8 *certified reference material, CRM, n*—a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by a traceable certificate or other documentation which is issued by a certifying body. **ISO Guide 30**

3.1.9 *correction, n*—action to eliminate a detected nonconformity. **ISO 9000**

3.1.9.1 *Discussion*—A correction may be performed before, during, or after corrective action. **ISO 9000**

3.1.9.2 *Discussion*—Corrections are typically one-time fixes that immediately address the nonconformity, but may not prevent recurrence.

3.1.10 *corrective action, n*—action taken to eliminate the cause of a nonconformity and to prevent recurrence. **ISO 9000**

3.1.10.1 *Discussion*—There can be more than one cause for a nonconformity. **ISO 9000**

3.1.10.2 *Discussion*—Corrective action is taken to prevent recurrence whereas preventive or continuous improvement actions are taken to prevent occurrence. **ISO 9000**

3.1.11 *measurand, n*—the measurable quantity subject to measurement.

3.1.12 *nonconformity, n*—non-fulfillment of a requirement. **ISO 9000**

3.1.12.1 *Discussion*—The non-fulfilment of the requirement may render the quality of the product or service unacceptable, indeterminate, or not according to specified requirements and may be identified through several sources.

3.1.13 *outlier, n*—a result far enough in magnitude from other results so as to be considered not a part of the set. **D6300**

3.1.14 *precision, n*—the closeness of agreement between test results obtained under prescribed conditions. **E456**

3.1.15 *proficiency testing, n*—determination of a laboratory's testing capability by evaluating its test results in interlaboratory exchange testing or crosscheck programs.

3.1.15.1 *Discussion*—One example is the ASTM D02 committee's proficiency testing programs in a wide variety of petroleum products and lubricants, many of which may involve more than a hundred laboratories.

3.1.16 *quality assurance (QA), n*—a system of activities, the purpose of which is to provide to the producer and user of a product, measurement, or service the assurance that it meets the defined standards of quality with a stated level of confidence.

3.1.16.1 *Discussion*—Quality assurance includes quality planning and quality control.

[7] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[8] Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.access.gpo.gov.

3.1.17 *quality control (QC), n*—a planned system of activities whose purpose is to provide a level of quality that meets the needs of users; also the uses of such a system.

3.1.18 *quality control sample (QC sample), n*—for use in quality assurance program to determine and monitor the precision and stability of a measurement system; a stable and homogenous material having physical or chemical properties, or both, similar to those of typical samples tested by the analytical measurement system. The material is properly stored to ensure sample integrity, and is available in sufficient quantity for repeated long-term testing. **D6299**

3.1.19 *reference material (RM), n*—a material with accepted reference value(s), accompanied by an uncertainty at a stated level of confidence for desired properties, which may be used for calibration or quality control purposes in the laboratory.

3.1.19.1 *Discussion*—Sometimes these may be prepared "in-house" provided the reference values are established using accepted standard procedures.

3.1.20 *repeatability, n*—the quantitative expression of the random error associated with a single operator in a given laboratory obtaining repetitive results with the same apparatus under constant operating conditions on identical test material. It is defined as the difference between two such results at the 95 % confidence level. **D6300**

3.1.21 *reproducibility, n*—a quantitative expression of the random error associated with different operators using different apparatus, and so forth, each obtaining a single result on an identical test sample when applying the same method. It is then defined as the 95 % confidence limit for the difference between two such single and independent results. **D6300**

3.1.22 *risk, n*—effect of uncertainty on objectives. **ISO 31000**

3.1.22.1 *Discussion*—An effect is a deviation from the expected and can be positive, negative, or both, and can address, create, or result in opportunities and threats. **ISO 31000**

3.1.23 *risk appetite, n*—organization's approach to assess and eventually pursue, retain, take, or turn away from risk. **ISO Guide 73**

3.1.24 *risk assessment, n*—overall process of risk identification, risk analysis, and risk evaluation. **ISO Guide 73**

3.1.24.1 *Discussion*—Risk assessments typically utilize an evidence based approach to assist the decision making process when evaluating the impact of risk to the laboratory. The risk should be evaluated in terms of consequence to the business and likelihood of recurrence when no mitigation of the risk is implemented.

3.1.25 *risk management, n*—coordinated activities to direct and control an organization with regard to risk. **ISO 31000**

3.1.26 *risk tolerance, n*—organization's readiness to bear the risk after risk treatment in order to achieve its objectives. **ISO Guide 73**

3.1.26.1 *Discussion*—Risk tolerance can be influenced by legal or regulatory requirements. **ISO Guide 73**

3.1.27 *site precision (R'), n*—the value below which the absolute difference between two individual test results obtained under site precision conditions may be expected to occur with a probability of approximately 0.95 (95 %). It is defined as 2.77 times the standard deviation of results obtained under site precision conditions. **D6299**

3.1.28 *site precision conditions, n*—conditions under which test results are obtained by one or more operators in a single site location practicing the same test method on a single measurement system using test specimens taken at random from the same sample of material over an extended period of time spanning at least a 15 day interval. **D6299**

3.1.29 *traceability, n*—property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *precision ratio (PR), n*—an estimate of relative magnitude of repeatability and reproducibility. The PR for a given standard test method can provide information on the relative significance between variation caused by different operators and laboratories compared to a single operator in a laboratory performing the standard test method.

3.2.2 *test performance index (TPI), n*—an approximate measure of a laboratory's testing capability, defined as the ratio of test method reproducibility (R) to site precision (R').

3.3 *Acronyms:*

3.3.1 *NIST*—National Institute of Standards and Technology, Gaithersburg, MD.

4. Significance and Use

4.1 A petroleum products, liquid fuels, and lubricants testing laboratory plays a crucial role in product quality management and customer satisfaction. It is essential for a laboratory to provide quality data. This document provides guidance for establishing and maintaining a quality management system in a laboratory.

4.1.1 The word 'customer' can refer to both customers internal and external to the laboratory or organization.

5. General Quality Requirements for the Laboratory

5.1 Establishment and maintenance of a quality management system shall include stated objectives in the following areas: a laboratory's adherence to test method requirements, calibration and maintenance practices, and its quality control program. Laboratory quality objectives should encompass the laboratory's continuous improvement goals as well as meeting customer requirements.

5.2 Management shall appoint a representative to implement and maintain the quality management system in the laboratory.

5.3 Laboratory management shall review the adequacy of the quality management system and the activities of the laboratory for consistency with the stated quality objectives at least annually.

5.4 The quality management system shall have documented processes for:

5.4.1 Sampling and sample management (see Section 6),

5.4.2 Data, records, and document management and control (see Section 7),

5.4.3 Control and implementation of test methods (see Section 8),

5.4.4 Equipment calibration, verification, and maintenance (see Section 9),

5.4.5 Quality control (see 10.1),

5.4.6 Proficiency testing (see 10.2),

5.4.7 Audits (see 10.3),

5.4.8 Customer feedback (see 10.4),

5.4.9 Control of nonconforming work and corrective action (see 10.5),

5.4.10 Ensuring that procured services and materials meet the contracted requirements, and

5.4.11 Ensuring that personnel are adequately trained to obtain quality results (see Section 11).

6. Sampling and Sample Management

6.1 When samples are obtained by laboratory staff, these samples shall be obtained in accordance with applicable industry standards.

6.2 The elements of sample management shall include at a minimum:

6.2.1 Procedures for sample receipt into the laboratory and assigning it a unique identifier.

6.2.2 Criteria for sample acceptance.

6.2.2.1 Procedures for addressing and notifying the customer when received samples deviate from requirements or are not suitable for testing.

6.2.3 Procedures for sample handling.

6.2.3.1 In cases where industry standards for sample handling are applicable and referenced within industry standard test methods, they shall be utilized (for example, Practice D4057).

6.2.4 Procedures for sample storage and retention. Items to consider when creating these procedures include:

6.2.4.1 Applicable government—local, state, or national—regulatory requirements or customer contract agreements.

6.2.4.2 Type of sample containers required to preserve the sample,

6.2.4.3 Control of access to the retained samples to protect their validity and preserve their original integrity,

6.2.4.4 Storage equipment and conditions,

6.2.4.5 Required safety precautions, and

6.2.4.6 Customer requirements.

6.2.5 Procedures for sample disposal in accordance with applicable government regulatory requirements.

NOTE 2—This may be handled through a separate chemical hygiene or waste disposal plan.

7. Data, Records, and Document Management and Control

7.1 *Reports of Analysis:*

7.1.1 The work carried out by a laboratory shall be covered by a certificate or report that accurately, clearly, objectively, and unambiguously contains all the information in accordance with the test method reporting, specification, customer, or accreditation authority requirements, or combinations thereof, and all other relevant information required for interpretation of the results.

NOTE 3—Reports are typically printed or electronic for external customers, however reports for internal customers may just be an entry in a Laboratory Information Management System (LIMS) or equivalent system.

7.1.2 The intent of the report should be to meet 7.1.1; however, some report requirements may be specified by the customer or an accreditation authority and if so these shall be followed. Other items in the lab report may include:

7.1.2.1 Name and address of the testing laboratory,

7.1.2.2 Unique identification of the report (such as serial number) on each page of the report including version identification if the report has been updated,

NOTE 4—Occasionally, a report may be updated and a version identification will enable one version of the report to be distinguished from another. This is necessary to determine which report version was the original and which is the most current. Simple conventions such as last updated date are useful means of version identification.

7.1.2.3 Name and contact information of the customer,

7.1.2.4 Order identification number (if relevant for the work undertaken),

7.1.2.5 Description and unique identification of the test sample(s) including comments on the sample condition if it is likely to have an adverse effect on the sample integrity,

7.1.2.6 Date of receipt of the test sample(s) and date(s) of performance of test, as appropriate,

7.1.2.7 Identification of the test specification, test method(s), or testing procedures used,

7.1.2.8 Description of the sampling procedure, and or sub-sampling (where relevant to the application of the results),

7.1.2.9 Any deviations, additions to or exclusions from the specified test method requirements,

7.1.2.10 Disclosure of any nonstandard sampling procedure, test method, or procedure utilized,

7.1.2.11 Measurements, examinations, and derived results including units of measurement, supported by tables, graphs, sketches, and photographs as appropriate, and any failures identified,

7.1.2.12 Product or sample specifications or limits, if applicable,

7.1.2.13 The test results with units of measurement and, where relevant, a statement of the measurement uncertainty,

7.1.2.14 Identification of any test results provided by an external laboratory,

7.1.2.15 Identification of the person(s) authorizing the final report, and

7.1.2.16 A statement on the laboratory policy regarding the reproduction of test reports.

7.1.3 Items to be included in laboratory reports should be specified by laboratory management or agreements with customers, or both.

7.1.4 Procedures for corrections or additions to a test report after issue shall be established.

7.2 *Reporting and Rounding the Data:*

7.2.1 The reporting requirements specified in the test method or procedure shall be used (unless specifically required otherwise by the customer or applicable regulations).

7.2.2 If rounding is performed, the rounding protocol of Practice E29 should be used unless otherwise specified in the method, procedure, or governing specification.

7.2.3 The test results should be reviewed and authorized before reporting to the customer.

7.3 *Records of Calibration, Verification, and Maintenance:*

7.3.1 Procedures shall be established for the management of instrument calibration records. The procedures used shall be in compliance with that required in the test method reported. Such records usually indicate the instrument calibrated, method or procedure used for calibration, the dates of last and next scheduled calibrations, the person performing the calibration, the values obtained before and after calibration, permissible tolerances, and the metrological traceability (if applicable) of the calibration materials used (that is, certified values). Records may be electronic.

7.3.2 Procedures shall be established for the management of instrument maintenance records. Such records usually indicate the instrument maintained, description of the maintenance performed, the dates of last and next maintenance, and the person performing the maintenance. Records may be electronic.

NOTE 5—For instruments that require calibration, calibration and maintenance records may be combined.

7.4 *Quality Control (QC) Testing Records:*

7.4.1 The laboratory shall have documented procedures for creating and maintaining records for analysis of QC samples where it has been identified as a QC tool. The QC procedures used shall be in compliance with that required in the test method reported. It is recommended that such records include the QC sample name, material, and source (if known), the test method(s) for which it is to be used, the assigned values and their uncertainty where applicable, and values obtained upon analysis. Additionally, it is recommended that the receipt date or the date the QC material was put into active use in the laboratory be documented, along with the expiration date (if applicable).

7.4.2 Procedures for initial setup and re-evaluation of the chart control limits, ongoing evaluation of the control charts, and retaining completed control charts should be established. It is recommended that these records include the date the control charts were changed and the reason for the change.

7.5 *Record Retention and Disposal:*

7.5.1 The record retention and disposal system should suit the laboratory's particular circumstances and comply with any existing regulations and customer requirements.

7.5.2 All data shall be maintained, archived, and disposed according to laboratory, company, customer, or regulatory agency requirements, or a combination thereof.

7.5.3 Procedures for retaining and disposal of records, including electronic, of all original observations, calculations and derived data, calibration records, and final test reports for an appropriate period shall be established. The records for each test should contain sufficient information to permit satisfactory replication of the test and recalculation of the results, and if changes have been made from the original record then these changes can be tracked to a previous or original record.

7.5.4 The original records shall be held in a suitable environment to prevent loss, damage, deterioration, unauthorized access, or amendment. A system shall exist that allows locating the required documents in a reasonable period of time.

7.5.5 All issued reports should be retained and disposed of in line with customer or regulatory requirements.

7.6 *Document Control:*

7.6.1 Document, implement, and maintain procedures to describe the control of documents in the laboratory. These procedures shall establish the process by which only authorized and appropriate (typically the most current) version of documents are available to the staff in the area of use. As a minimum, for laboratory procedures (for example, standard operating procedures, SOPs) and laboratory test procedures (if used), a document control process shall address version/revision identification, unique identification, authorization for use by relevant personnel, review and update period, and a system whereby unintended use of obsolete documents is prevented.

7.7 *Laboratory Information Management System (LIMS):*

7.7.1 The LIMS system in use should suit the laboratory's circumstances and can be electronic or non-electronic in nature or a mixture of both.

7.7.2 Procedures for backup and recovery of electronic data shall be established.

7.7.3 Procedures shall be established for changes to LIMS system and should include authorization, documentation, and validation of the change.

7.7.4 Procedures shall be established to protect the system from unauthorized access and tampering to maintain the integrity of the data reported.

7.7.5 Calculations and data transfers performed directly from instruments should be validated as accurate on a periodic basis.

8. Test Methods

8.1 The laboratory shall have documented test methods and procedures for performing the required tests.

8.2 The test methods that are stated in the product specifications or agreements with customers shall be used for sample analysis.

8.3 The laboratory shall have procedures for reporting modifications to the test method requirements.

8.4 The laboratory shall have procedures which include a review, justification and approval for modifying any test method.

8.4.1 Any modification shall be documented and accepted by the customer prior to testing the customer's samples.

8.5 The test methods shall be maintained up-to-date and be readily available to the laboratory staff.

8.5.1 If a previous version of a test method is required a version number, revision number or year of adoption as appropriate should indicate its identity.

NOTE 6—Some specifications or regulations may require the use of a specific year version of a test method.

9. Equipment Calibration, Verification, and Maintenance

9.1 *Calibration and Verification:*

9.1.1 Perform calibrations and verifications as specified in the test methods and maintain these records.

9.1.2 If an instrument is found to be out of calibration, or does not pass the verification and the situation cannot be immediately addressed, then the instrument shall be taken out of operation and tagged as such until the situation is corrected (see 10.5).

9.1.3 The performance of apparatus and equipment used in the laboratory but not calibrated in that laboratory (that is, precalibrated, vendor supplied) should be verified by using a documented, technically valid procedure at periodic intervals.

9.1.4 Procedures shall be established to verify the measuring and testing equipment in-calibration status, at a scheduled frequency, including equipment pre-calibrated by the vendor. Items to consider when creating these procedures include:

9.1.4.1 Records of calibration, verification and maintenance (see 7.3),

NOTE 7—The calibration and verification frequency and protocol may vary with the instrument type, test method requirements and its frequency of use.

9.1.4.2 Traceability to national or international standards for calibration records,

NOTE 8—Where the concept of traceability to national or international standards of measurement is not applicable, the testing laboratory shall provide satisfactory evidence of test result accuracy (for example, by participation in a program of interlaboratory comparisons or use of CRMs).

9.1.4.3 Customers and test method requirements and,

9.1.4.4 Corrective actions (see 10.5).

9.2 *Calibration Standards and Materials:*

9.2.1 Use the calibration material as specified in the test method.

9.2.2 Calibration material shall be appropriate for the method and characterized with the accuracy demanded by the analysis to be performed. Quantitative calibration standards should be prepared from constituents of known purity. Use the primary calibration standards or CRMs specified or allowed in the test method.

9.2.3 Where appropriate, values for reference materials should be produced following the certification protocol used by NIST[9] or other standards issuing bodies, and, should be traceable to the International System of Units (SI) units of measurement, if required or appropriate.

9.2.4 Expiry dates of calibration standards shall be documented. If the expiry date is not stated by the vendor then this should be established by a documented technically valid procedure. Do not use this material beyond the assigned expiry date.

9.3 *Verification Standards and Materials:*

9.3.1 Use the verification material as specified in the test method.

9.3.2 Materials analyzed in proficiency testing programs meeting the requirements of Practice D6300 or ISO 4259-3 may be used as verification materials, provided no obvious bias or unusual frequency distribution of results are observed. The consensus value is most likely the value closest to the true value of this material; however, the uncertainty attached to this mean value will be dependent on the precision and the total number of the participating laboratories.

NOTE 9—Some proficiency testing material may change composition once the sample is opened.

9.3.3 Secondary standards may be used to verify calibration of equipment.

9.3.4 Expiry dates of verification material shall be documented. Do not use this material beyond the assigned expiry date.

9.4 The laboratory shall establish procedures for the storage of reference materials in a manner to ensure their safety, integrity, and protection from contamination (see 6.2.4).

9.5 *Maintenance:*

9.5.1 Document and maintain procedures and schedules for performing maintenance and preventative maintenance of inspection, testing and measuring equipment.

9.5.2 Perform maintenance as required and maintain records that contain all relevant data and indicate completion of the maintenance activity.

9.5.3 A reliability strategy should be developed which may include the following:

9.5.3.1 Equipment age and performance,

9.5.3.2 Back-up equipment justification based on utilization,

9.5.3.3 Critical spare parts, and

9.5.3.4 Inventory of equipment.

10. Performance Measures

10.1 *Quality Control:*

10.1.1 *Quality Control Practices:*

10.1.1.1 Quality control practices shall be established to assess applicable test methods used by the laboratory.

10.1.1.2 Use of appropriate quality control charts or other quality control practices shall be established for each test method performed by the laboratory unless specifically excluded (see Practice D6299). Document cases where quality control practices are not employed and include the rationale.

10.1.1.3 This practice advocates the regular testing of quality control samples with timely interpretation of test results. This practice also advocates using appropriate control charting techniques to ascertain the in-statistical-control status of test methods in terms of precision, bias (if a standard is being used), and method stability over time. For details concerning QC sample requirements and control charting techniques, refer to Practice D6299. The generally accepted practices are outlined in 10.1.1.4 through 10.1.5.4.

10.1.1.4 QC sample testing frequency shall be established for each applicable test method. Principal factors to be considered for determining the frequency of testing shall include: (*1*)

[9] The most current protocols for NIST certification of reference materials can be found in NIST SP260–136 (2020 Edition) found at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2020.pdf.

frequency of use of the analytical measurement system, (*2*) criticality of the parameter being measured and business economics, (*3*) established system stability and precision performance based on historical data, (*4*) regulatory requirements, (*5*) contractual provisions, and (*6*) test method requirements. Minimum QC sample testing specified in regulations or in the test method shall prevail over any larger interval determined below in (*1*) through (*4*).

(1) If site precision for a specific test has not been established as defined by Practice D6299, then the recommended frequency for analysis of QC samples is one QC out of every ten samples analyzed. Alternatively, one QC sample is analyzed each day that samples are analyzed, whichever is more frequent.

(2) Once the site precision has been established as defined by Practice D6299, and to ensure similar quality of data is achieved with the documented method, the minimal QC frequency may be adjusted based on the Test Performance Index (TPI) and the Precision Ratio (PR).

(3) Table 1 provides recommended minimal QC frequencies as a function of PR and TPI. For those tests, which are performed infrequently, for example less than 25 samples are analyzed monthly, it is recommended that at least one QC sample be analyzed each time samples are analyzed.

(4) In many situations, the minimal QC frequency as recommended by Table 1 may not be sufficient to ensure adequate statistical quality control, considering, for example, the significance of use of the results. Hence, it is recommended that the flowchart in Fig. 1 be followed to determine if a higher QC frequency should be used.

(5) The TPI should be recalculated and reviewed at least annually. Adjustments to QC frequency should be made based on the recalculated TPI by following sections 10.1.1.4(*1*) and (*2*).

10.1.1.5 QC testing frequency, QC samples, and their test values shall be recorded.

10.1.1.6 All persons who routinely operate the system shall participate in generating QC test data. QC samples should be treated as regular samples.

NOTE 10—Avoid special treatment of QC samples designed to "get a better result." Special treatment seriously undermines the integrity of precision and bias estimates.

10.1.1.7 The laboratory may establish random or blind testing, or both, of QC or other known materials.

10.1.2 *Test Method Performance Assessment:*

TABLE 1 Minimal QC Frequency as a Function of Test Performance Index

TPI for Standard Test Methods with PR<4	TPI for Standard Test Methods with PR≥4	Nominal QC Frequency (1 QC out of every X Samples) Values of X	Approximate Percentage of QC Samples/ Total Analyses
Not determined	Not determined	10	9
<0.8	<1.6	10	9
0.8–1.2	1.6–2.4	20	5
1.2–2.0	2.4–4.0	35	3
>2.0	>4.0	40	2

10.1.2.1 A laboratory should review their precision obtained for multiple analyses on the same sample. The site precision (R') of the QC samples can be compared with the reproducibility or repeatability given in the standard test methods to indicate how well a laboratory is performing against the industry standards.

(1) The test performance index (TPI) can be used to compare the precision of the laboratory measurements with the published reproducibility of a standard test method. The term TPI is defined as:

$$\text{test performance index} = \frac{\text{test method reproducibility}}{\text{site precision}} \quad (1)$$

NOTE 11—The ASTM International Committee D02 sponsored Proficiency Test Program employs a test performance index based on the ratio of the published ASTM reproducibility to the reproducibility calculated from the program data. This index is termed the TPI (Industry) to distinguish from the definition in 10.1.2.1(*1*).

10.1.2.2 A precision ratio (PR) is determined for a given published test method so that the appropriate action criteria may be applied for a laboratory's TPI. The PR for a published test method estimates the influence that non-site specific variations has on the published precision. The PR can be calculated by dividing the test method's reproducibility by the repeatability as shown in Eq 2.

$$\text{Precision Ratio, PR} = \frac{\text{Test Method reproducibility (R)}}{\text{Test Method repeatability (r)}} \quad (2)$$

where the ratio of R/r is calculated to the nearest integer (that is, 1, 2, 3, 4, …).

(1) A test method with PR greater than or equal to 4, for the purpose of this practice, is deemed to exhibit a significant difference between repeatability and reproducibility. For further explanation on why the greater than or equal to 4 criterion was chosen, please see Appendix X2.

10.1.2.3 A laboratory's TPI may be a function of the sample type being analyzed and variations associated with that laboratory. As general guidelines Table 2 may be used once the TPI of that laboratory and the PR of the published standard test method has been calculated.

(1) A laboratory may choose to set other benchmarks for TPI, keeping in mind that site precision of an adequately performing laboratory cannot, in the long run, exceed the practically achievable reproducibility of the method when PR is less than 4 or approaches repeatability when PR is much greater than 4.

NOTE 12—Experience has shown, for some methods, published reproducibility is not in good agreement with the precision achieved by participants in well-managed proficiency testing programs. Users should consider this fact when evaluating laboratory performance using TPI.

10.1.2.4 A laboratory's site precision (R') that is significantly worse than the published test method reproducibility may indicate poor performance. As appropriate, investigate to determine the root cause for this performance so that corrective action can be undertaken if necessary.

10.1.3 *Quality Control Sample and Test Data Evaluation:*

10.1.3.1 QC samples should be stable and homogeneous materials having physical or chemical properties, or both, representative of the actual samples being analyzed by the test method. This material shall be well-characterized for the