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An American National Standard

Standard Guide for Quality System in Petroleum Products and Lubricants Testing Laboratories[1]

This standard is issued under the fixed designation D 6792; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This guide covers the establishment and maintenance of the essentials of a quality system in laboratories engaged in the analysis of petroleum products and lubricants. It is designed to be used in conjunction with Practice D 6299.

NOTE 1—This guide is based on the quality management concepts and principles advocated in ANSI/ISO/ASQ Q9000 standards, ISO Guide 17025, ASQC Manual,[2] and ASTM standards such as D 3244, D 4182, D 4621, D 6299, D 6300, E 29, E 177, E 456, E 548, E 882, E 994, E 1301, E 1323, 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 and health practices and determine the applicability of regulatory requirements prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 3244 Practice for Utilization of Test Data to Determine Conformance with Specifications[5]

D 4182 Practice for Evaluation of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke[6]

D 4621 Guide for Quality Management in an Organization that Samples or Tests Coal and Coke[6]

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance[7]

D 6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants[7]

D 6617 Practice for Laboratory Bias Detection Using Single Test Result from Standard Material[8]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[9]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[9]

E 456 Terminology Relating to Quality and Statistics[9]

E 548 Guide for General Criteria Used for Evaluating Laboratory Competence[10]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[9]

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[11]

E 994 Guide for Calibration and Testing Laboratory Accreditation Systems General Requirements for Operation and Recognition[9]

E 1301 Guide for Proficiency Testing by Interlaboratory Comparisons[9]

E 1323 Guide for Evaluating Laboratory Measurement Practices and the Statistical Analysis of the Resulting Data[9]

2.2 *ISO Standards:*[12]

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

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

ISO Standard 4259 Petroleum Products—Determination and Application of Precision Data in Relation to Methods of Test

ANSI/ISO/ASQ Q9000 Quality Management System Standards

3. Terminology

3.1 *Definitions:*

3.1.1 *accepted reference value, ARV, n*—a value that serves as an agreed upon reference for comparison, and which is

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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.

[3] *ASTM STP 15D*, "ASTM Manual on Presentation of Data and Control Chart Analysis," available from ASTM International Headquarters.

[4] *ASTM STP 1209*, "ASTM Manual on Total Quality Management," available from ASTM International Headquarters.

[5] *Annual Book of ASTM Standards*, Vol 05.02.

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

[7] *Annual Book of ASTM Standards*, Vol 05.03.

[8] *Annual Book of ASTM Standards*, Vol 05.04.

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

[10] Discontinued; See 2001 *Annual Book of ASTM Standards*, Vol 14.02.

[11] *Annual Book of ASTM Standards*, Vol 03.05.

[12] Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.

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

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. **E 456**

3.1.2 *accuracy*, *n*—the closeness of agreement between a test result and an accepted reference value. **E 456**

3.1.3 *audit*, *n*—a systematic examination of a laboratory's quality system procedure and related activities by an internal or external team to determine whether these procedures or activities are implemented according to the documented system.

3.1.4 *bias*, *n*—the difference between the population mean of the test results and an accepted reference value. **E 456**

3.1.5 *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.

3.1.6 *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**

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

3.1.8 *outlier*, *n*—a result far enough in magnitude from other results so as to be considered not a part of the set. **D 6300**

3.1.9 *precision*, *n*—the closeness of agreement between test results obtained under prescribed conditions. **E 456**

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

3.1.10.1 *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.

3.1.11 *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.

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

3.1.12 *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.

3.1.13 *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. **D 6299**

3.1.14 *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.

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

3.1.15 *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. **D 6300**

3.1.16 *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 the defined as the 95 % confidence limit for the difference between two such single and independent results. **D 6300**

3.1.17 *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. **D 6299**

3.1.18 *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. **D 6299**

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

3.3 *Acronyms:*

3.3.1 *NIST*, *n*—National Institute of Standards and Technology (formerly called National Bureau of Standards), Gaithersburg, MD.

4. Significance and Use

4.1 A petroleum products 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 system in a laboratory.

5. General Quality Requirements for the Laboratory

5.1 Establishment and maintenance of a quality 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 system in the laboratory.

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

5.4 The quality system shall have documented processes for:

5.4.1 Sample management (see Section 6),

5.4.2 Data and record management (see Section 7),

5.4.3 Producing accurate, reliable, and properly represented test results (see Section 8),

5.4.4 Audits and proficiency testing (see Section 9),

5.4.5 Corrective and preventive action (see Section 11),

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

5.4.7 Ensuring that personnel are adequately trained to obtain quality results.

6. Sample Management

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

6.1.1 Procedures for unique sample identification.

6.1.2 Criteria for sample acceptance.

6.1.3 Procedures for sample handling.

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

6.1.4.1 Applicable government—local, state, or national—regulatory requirements for shelf life and time-dependent tests that set product stability limits,

6.1.4.2 Type of sample containers required to preserve the sample,

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

6.1.4.4 Storage conditions,

6.1.4.5 Required safety precautions, and

6.1.4.6 Customer requirements.

6.1.5 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 and Record Management

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 and unambiguously presents the test results and all other relevant information.

NOTE 3—This report may be an entry in a Laboratory Information Management System (LIMS) or equivalent system.

7.1.2 The following items are suggested for inclusion in laboratory reports:

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,

7.1.2.3 Name and address of the customer,

7.1.2.4 Order number,

7.1.2.5 Description and identification of the test sample,

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

7.1.2.7 Identification of the test specification, method and procedure,

7.1.2.8 Description of the sampling procedure, where relevant,

7.1.2.9 Any deviations, additions to or exclusions from the specified test requirements, and any other information relevant to a specific test,

7.1.2.10 Disclosure of any nonstandard test method or procedure utilized,

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

7.1.2.12 Minimum-maximum product specifications, if applicable,

7.1.2.13 A statement of the measurement uncertainty (where relevant),

7.1.2.14 Any other information which might be required by the customer,

7.1.2.15 A signature and job title of person(s) accepting technical responsibility for the test report and the date of issue, and

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

7.1.3 Items actually 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.

7.2.2 If rounding is performed, the rounding protocol of Practice E 29 should be used unless otherwise specified in the method or procedure.

7.3 *Records of Calibration and Maintenance*:

7.3.1 Procedures shall be established for the management of instrument calibration records. Such records usually indicate the instrument calibrated, method or procedure used for calibration, the dates of last and next calibrations, the person performing the calibration, the values obtained during calibration, and the nature and traceability (if applicable) of the calibration standards (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, the dates of last and next maintenance, and the person performing the maintenance. Records may be electronic.

NOTE 4—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. It is recommended that such records include the sample name and source, the test(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 date put into active QC use in the laboratory be documented, along with the expiration date (if applicable).

7.4.2 Procedures for 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*:

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

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

7.5.3 Procedures for retaining a record 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.

7.5.4 The records shall be held in a safe and secure storage. A system shall exist that allows locating the required documents in a reasonable period of time.

8. Producing Accurate, Reliable, and Properly Represented Test Results

8.1 The laboratory shall have documented test methods and procedures for performing the required tests. These shall be maintained up-to-date and be readily available to the laboratory staff. The test methods that are stated in the product specifications or agreed upon with customers should be used for sample analysis.

8.2 The laboratory shall have procedures for the approval, documentation, and reporting of deviations from the test method requirements or the use of alternative methods.

8.3 Procedures shall be established to ensure that measuring and testing equipment is calibrated, maintained properly, and is in statistical control. Items to consider when creating these procedures include:

8.3.1 Records of calibration and maintenance (see 7.3),

8.3.2 Calibration and maintenance schedule,

NOTE 5—The calibration frequency may vary with the instrument type and its frequency of use, some needing calibration before each set of analyses, others requiring calibration at less frequent periods, or triggered by a QC chart out-of-statistical-control situation.

8.3.3 Traceability to national or international standards,

NOTE 6—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).

8.3.4 Requirements of the test method or procedure,

8.3.5 Customer requirements, and

8.3.6 Corrective action (see Section 11).

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

8.5 Calibration standards 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.

8.5.1 Where appropriate, values for reference materials should be produced following the certification protocol used by NIST[13,14,15] or other standards issuing bodies, and, should be traceable to national or international standard reference materials, if required or appropriate.

8.5.2 The materials analyzed in proficiency testing programs meeting the requirements of Practice D 6300 or ISO 4259 may be used as reference 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.

8.6 The laboratory shall establish procedures for the storage of reference materials in a manner to ensure safety, integrity, and non-contamination (see 6.1.4).

8.7 Records of instrument calibration shall be maintained (see Section 7).

8.8 If an instrument is found to be out of calibration, the instrument shall be taken out of operation and tagged as such until the situation is corrected (see Section 11).

8.9 *Quality Control Practices*:

8.9.1 This guide advocates the practice of regularly testing quality control samples with timely interpretation of test results using appropriate control charting techniques to ascertain the in statistical control status of test methods in terms of method stability over time, precision, and bias. For details concerning QC sample requirements, test frequency, and control charting techniques, refer to Practice D 6299. The generally accepted practices are outlined in 8.9.2-8.9.4.

8.9.2 QC testing frequency, QC samples to be used, and their test values shall be documented.

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

NOTE 7—Avoid special treatment of QC samples designed to "get a better result." Special treatment seriously undermines the integrity of precision and bias estimates.

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

8.10 *Quality Control Sample and Test Data Evaluation*:

8.10.1 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 analyses of interest, available in sufficient quantities and have concentration values that are within the calibration range of the

[13] Cali, J. P., *Anal. Chem.*, 48, 802A, 1976.

[14] Uriano, G. A., and Gravatt, C. C., *CRC Crit. Revs, in Anal. Chem.*, 6, 361, 1977.

[15] Alvarez, R., Rasberry, S. D., and Uriano, G. A., *Anal. Chem.*, 54, 1226A, 1982.

test method, and reflect the most common values tested by the laboratory. For QC testing that is strictly for monitoring the test method stability and precision, the QC sample expected value is the control chart centerline, established using data obtained under site precision conditions. For regular QC testing that is intended to assess test method bias, RMs, or CRMs with independently assigned ARVs should be used. The results should be assessed in accordance with Practice D 6299 requirements for check standard testing. For infrequent QC testing for bias assessment, refer to Practice D 6617.

NOTE 8—It is not advisable to use the same sample for both a calibrant and a QC sample. It is not advisable to use the same chemical lot number for both a calibrant and a QC sample.

8.10.2 If the QC material is observed to be degrading or changing in physical or chemical characteristics, this shall be immediately investigated and, if necessary, a replacement QC material shall be prepared for use.

NOTE 9—In a customer-supplier quality dispute, it may be beneficial to provide the customer with the laboratory's test results on QC material to demonstrate testing proficiency. Practice D 3244 may be useful.

8.11 *Quality Control Charts*:

8.11.1 QC sample test data should be promptly plotted on a control chart and evaluated to determine if the results obtained are within the method specifications and laboratory-established control limits. The charts used should be appropriate for the testing conditions and statistical objectives. Corrective action should be taken and documented for any analyses that are out-of-control (see Section 11).

NOTE 10—Charts such as individual, moving average and moving range, exponentially weighted moving average, or cumulative summation charts may be used as appropriate. Refer to Practice D 6299 for guidance on plotting these charts.

8.11.1.1 The charts should indicate the test method, date when the QC analyses were performed, and who performed them. Analysis of test samples should not be reported until the QC data are assessed and the testing process is verified to be in statistical control.

8.11.2 Adequate training should be given to the analysts to enable them to generate and interpret the charts.

8.11.3 It is suggested that the charts be displayed prominently near the analysis workstation, so that all can view and, if necessary, help in improving the analyses.

8.11.4 Supervisory and technical personnel should periodically review the QC charts.

8.11.5 The laboratory should establish written procedures outlining the appropriate interpretation of QC charts and responses to out-of-statistical-control situations observed. All laboratory analysts involved in the QC sample analyses should be trained on these procedures.

8.11.5.1 When an out-of-statistical-control situation has been identified, remedial action should be taken before analyzing further samples. In all such cases, run the QC sample and ensure that a satisfactory result can be obtained before analyzing *unknown* samples.

NOTE 11—A generic checklist for investigating the root cause of unsatisfactory analytical performance is given in Appendix X1.

8.11.6 Out-of-control situations may be detected by one or more analyses. In these cases, it may be necessary to retest samples analyzed during the period between the last in-control QC data point and the QC data point that triggered the out-of-statistical-control notice (or event) using retained samples and equipment known to be in control. If the new analysis shows a difference that is statistically different from the original results, and the difference exceeds the established site precision of that test, the laboratory should decide on what further actions are necessary (see Section 11).

8.12 *Revision of Control Charts*—QC chart revision is covered in detail in Practice D 6299. Control charts shall be revised only when the existing limits are no longer appropriate. As a guideline, revisions may be needed when:

8.12.1 Additional information becomes available,

8.12.2 The process has improved,

8.12.3 A new QC material is initiated and the mean value is different than the previous QC material, or

8.12.4 There are major changes to the test procedure.

9. Audits and Proficiency Testing

9.1 *Audits*:

9.1.1 A laboratory shall have a system to periodically review its own practices to confirm continued conformance to the laboratory's documented quality system. Even if the laboratory is subjected to a formal external audit (for example, as a requirement of ANSI/ISO/ASQ Q9000), it is important to have internal audits since the internal reviewers may be more familiar with their laboratory's requirements than the external auditors.

9.1.2 Audits of test methods should be conducted to confirm adherence to the documented test methods. The performance of the entire test should be observed and checked against the official specified test method. An annual audit of test methods is recommended.

NOTE 12—These audits may be part of the quality system audits or may be separate.

9.1.3 Audit results shall be promptly documented. The team shall report the audit results to management having the authority and responsibility to take corrective action and to its management.

9.1.4 The findings and recommendations of these internal audits shall be reviewed by the laboratory management and acted upon to correct the deficiencies or nonconformances.

9.1.5 The effectiveness of any corrective actions taken in response to an audit shall be verified. The follow-up results shall be documented as required by the quality system procedures or laboratory policy, or both.

9.2 *Proficiency Testing*:

9.2.1 Participation in regularly conducted interlaboratory exchanges or cross-check programs, where typical samples are tested by multiple test facilities using a specified (ASTM) test protocol, can provide a cost-effective alternative to regular CRM testing. Pending on adequate frequency, participants may control chart their deviations from the consensus values established by the exchange averages to ascertain if their measurement processes are non-biased. The precision of these exchange performance data can also be assessed against

precision established by in-house QC sample testing for consistency (see Practice D 6299 for details).

9.2.2 Participation in proficiency testing shall not be considered as a substitute for in-house quality control, and vice versa.

10. Test Method Precision Performance Assessment

10.1 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:

$$\text{test performance index} = \frac{\text{test method reproducibility}}{\text{site precision}}$$

10.2 The TPI may be a function of the sample type being analyzed. As general guidelines the following may be used:

10.2.1 A TPI greater than 1.2 indicates that the performance is probably satisfactory relative to ASTM published precision.

10.2.2 A TPI greater than 0.8 and less than 1.2 indicates performance may be marginal and the laboratory should consider method review for improvement.

10.2.3 A TPI less than 0.8 suggests that the method as practiced at this site is not consistent with the ASTM published precision. Either laboratory method performance improvement is required, or the ASTM published precision does not reflect precision achievable. Existing interlaboratory exchange performance (if available) should be reviewed to determine if the latter is plausible.

10.2.4 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.

NOTE 13—Experience has shown, for some methods, published reproducibility is not in good agreement with the precision achieved by participants in well-managed crosscheck programs. Users should consider this fact when evaluating laboratory performance using TPI.

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

10.4 A laboratory precision significantly worse than the published test method reproducibility may indicate poor performance. An investigation should be launched to determine the root cause for this performance so that corrective action can be undertaken if necessary. Such a periodic review is a key feature of a laboratory's continuous improvement program.

11. Corrective and Preventive Action

11.1 The need for corrective and preventive action may be indicated by one or more of the following unacceptable situations:

11.1.1 Equipment out of calibration,

11.1.2 QC sample out of control,

11.1.3 Out of method specification,

11.1.4 Out of product, material, or process specification data,

11.1.5 Outlier or unacceptable trend in an interlaboratory cross-check program,

11.1.6 Nonconformance identified in an external or internal audit,

11.1.7 Nonconformance identified during review of laboratory data or records, and

11.1.8 Customer complaint.

11.2 When any of these situations occur, the root cause should be investigated and identified. Procedures for investigating root cause should be established. Items to consider when creating these procedures include:

11.2.1 Determining when the test of equipment was last known to be in control,

11.2.2 Identifying results that may have been adversely affected,

11.2.3 How to handle affected results already reported to a customer,

11.2.4 What to do if the root cause cannot be determined, and

11.2.5 What to do if it is determined that the original data is correct.

11.3 It is possible that the analytical results are correct, even if they don't meet specifications. Procedures should consider this possibility. See Appendix X1 for a checklist for investigating the root cause of unsatisfactory analytical performance. Procedures should also be established for the identification and implementation of appropriate corrective and preventive action so that the situation does not reoccur. This may involve:

11.3.1 Training or retraining personnel,

11.3.2 Reviewing customer specifications,

11.3.3 Reviewing test methods and procedures,

11.3.4 Establishing new or revised procedures,

11.3.5 Instrument maintenance and repair,

11.3.6 Re-preparation of reagents and standards,

11.3.7 Recalibration of equipment,

11.3.8 Reanalysis of samples, and

11.3.9 Additional QC sample analysis.

11.3.10 The situation, root cause, and corrective/preventive action taken should be documented promptly. A corrective and preventive action report is a suitable format for documentation. The report should be reviewed and approved by management and then verified for effectiveness of corrective/preventive action.

11.4 Quality control charts (see 8.11) are a method of preventive action and should be evaluated on a regular basis to prevent, when possible, out-of-statistical-control situations.

11.5 *Customer Complaints*—A procedure shall exist to follow-up on customer complaints or non-conformances brought to the laboratory's attention by a client. The result of such investigation should be communicated to the customer as soon as practical.

11.6 *Training*—Laboratory training should cover at a minimum the following areas: safety, test methods, and company policies and procedures. Records of training should be maintained.

12. Relationship with Other Quality Standards

12.1 Some laboratories in the petrochemicals testing area have been registered to ISO Standard 17025. There are a number of similarities between the ISO Standard and this guide in key areas of managing laboratory quality. For example:

Requirement	ISO Standard 17025	ASTM Guide D 6792
Quality System	4.2	5.1
Document Control	4.3	8.1; 8.2
Contract Review	4.4	5.4.6
Complaints	4.8	11.5
Corrective Action	4.10	11; Appendix X1
Preventive Action	4.11	11.4
Control of Records	4.12	7.3.1; 7.4; 7.5
Internal Audits	4.13	9.1
Management Reviews	4.14	5.3
Personnel	5.2	5.4.7
Calibration	5.6.2.1	8.3–8.8
Sample Handling	5.8	6.1
Quality Control Procedures	5.9	8.9
Use of Quality Control Materials	5.9.a	8.10
Proficiency Testing	5.9.b	9.2
Data Reports	5.10	7.1

12.2 *Measurement Uncertainty*—For test methods under the jurisdiction of Committee D02, measurement uncertainty as required in ISO 17025, as practiced by a laboratory, can be estimated by multiplying 2× the site precision standard deviation as defined in Practice D 6299.

NOTE 14—The complexity and empirical nature of the majority of D02 methods preclude the application of rigorous measurement uncertainty algorithms. In many cases, interactions between the test method variables and the measurand cannot be reasonably estimated due to the covariance of the variables that affect the measurand. The site precision approach estimates the combined effects of these variables on the total uncertainty for the measurand.

13. Keywords

13.1 audit; calibration; control charts; proficiency testing; quality assurance; quality control; test performance index

APPENDIX

(Nonmandatory Information)

X1. A CHECKLIST FOR INVESTIGATING THE ROOT CAUSE OF UNSATISFACTORY ANALYTICAL PERFORMANCE

X1.1 To identify why a laboratory's data may have been considered a statistical outlier or to improve the precision, or both, the following action items (not necessarily in the order of preference) are suggested. There may be additional ways to improve the performance.

X1.1.1 Check the results for typos, calculation errors, and transcription errors.

X1.1.2 Reanalyze the sample; compare to site precision, or, if not available, test method repeatability.

X1.1.3 Check the sample for homogeneity or contamination, and that a representative sample has been analyzed.

X1.1.4 Review the test method and ensure that the latest version of the ASTM test method is being used. Check the procedure step–by–step with the analyst.

X1.1.5 Check the instrument calibration.

X1.1.6 Check the statistical quality control chart to see if the problem has been developing earlier.

X1.1.7 Check the quality of the reagents and standards used, and whether they are expired or contaminated.

X1.1.8 Check the equipment for proper operation against the vendor's operating manual.

X1.1.9 Perform maintenance or repairs, or both, on the equipment following guidelines established by the vendor.

X1.1.10 After the problem has been resolved, analyze a certified reference material if one is available, or the laboratory quality control sample, to ascertain that the analytical operation is under control.

X1.1.11 Provide training to new analysts and, if necessary, refresher training to experienced analysts.

X1.1.12 Document the incident and the learnings for use in the future if a similar problem occurs.

SUMMARY OF CHANGES

Subcommittee D02.94 has identified the location of selected changes to this standard since the last issue (D 6792 – 02) that may impact the use of this standard.

(*1*) Added a new definition of term *measurand*.
(*2*) Added ISO Standard 17025 as a referenced document.
(*3*) Added 12.1, comparing this guide with ISO Standard 17025.
(*4*) Added 12.2 on measurement uncertainty.