



# Standard Guide for Quality System in Petroleum Products and Lubricants Testing Laboratories<sup>1</sup>

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 ( $\epsilon$ ) 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,<sup>2</sup> 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,<sup>3</sup> and STP 1209.<sup>4</sup>

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:<sup>5</sup>

- D 3244 Practice for Utilization of Test Data to Determine Conformance with Specifications
- D 4182 Practice for Evaluation of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke
- D 4621 Guide for Quality Management in an Organization That Samples or Tests Coal and Coke
- D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance

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

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

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

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

E 456 Terminology Relating to Quality and Statistics

E 548 Guide for General Criteria Used for Evaluating Laboratory Competence<sup>6</sup>

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

E 994 Guide for Calibration and Testing Laboratory Accreditation Systems General Requirements for Operation and Recognition<sup>7</sup>

E 1301 Guide for Proficiency Testing by Interlaboratory Comparisons

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

2.2 ISO Standards:<sup>8</sup>

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 derived as: (1) a theoretical or established value, based on

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<sup>2</sup> “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.

<sup>3</sup> ASTM STP 15D, *ASTM Manual on Presentation of Data and Control Chart Analysis*, ASTM International, W. Conshohocken, PA.

<sup>4</sup> ASTM STP 1209, *ASTM Manual on Total Quality Management*, ASTM International, W. Conshohocken, PA.

<sup>5</sup> 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.

<sup>6</sup> Withdrawn.

<sup>7</sup> Withdrawn.

<sup>8</sup> 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.

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 then 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: 06

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 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<sup>9,10,11</sup> 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. 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 D 6299. The generally accepted practices are outlined in 8.9.2 through 8.12.4.

8.9.2 Test QC samples on a regular schedule. Principal factors to be considered for determining the frequency of testing include: (1) 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.

8.9.2.1 If site precision for a specific test has not been established as defined by Practice D 6299, then the recommended frequency for analysis of QC samples is one QC out of

<sup>9</sup> Cali, J. P., *Anal. Chem.*, 48, 802A, 1976.

<sup>10</sup> Uriano, G. A., and Gravatt, C. C., *CRC Crit. Revs. in Anal. Chem.*, 6, 361, 1977.

<sup>11</sup> Alvarez, R., Rasberry, S. D., and Uriano, G. A., *Anal. Chem.*, 54, 1226A, 1982.

every ten samples analyzed. Alternatively, one QC sample is analyzed each day that samples are analyzed, whichever is more frequent.

8.9.2.2 Once the site precision has been established as defined by Practice **D 6299**, 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).

(1) For standard test methods with PR (as defined in **10.2**) less than 4 and a TPI (as defined in **10.1**) less than 0.8, consult **10.3** and the Standard Test Method for appropriate corrective action.

(2) For standard test methods with PR (as defined in **10.2**) greater than or equal to 4 and a TPI (as defined in **10.1**) less than 1.6, consult **10.3** and the Standard Test Method for appropriate corrective action.

8.9.2.3 **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.

8.9.2.4 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.

8.9.2.5 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 **8.9.2.2** and **8.9.2.4**.

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

8.9.4 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.

8.9.5 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, have

concentration values that are within the calibration range of the 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**.

**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

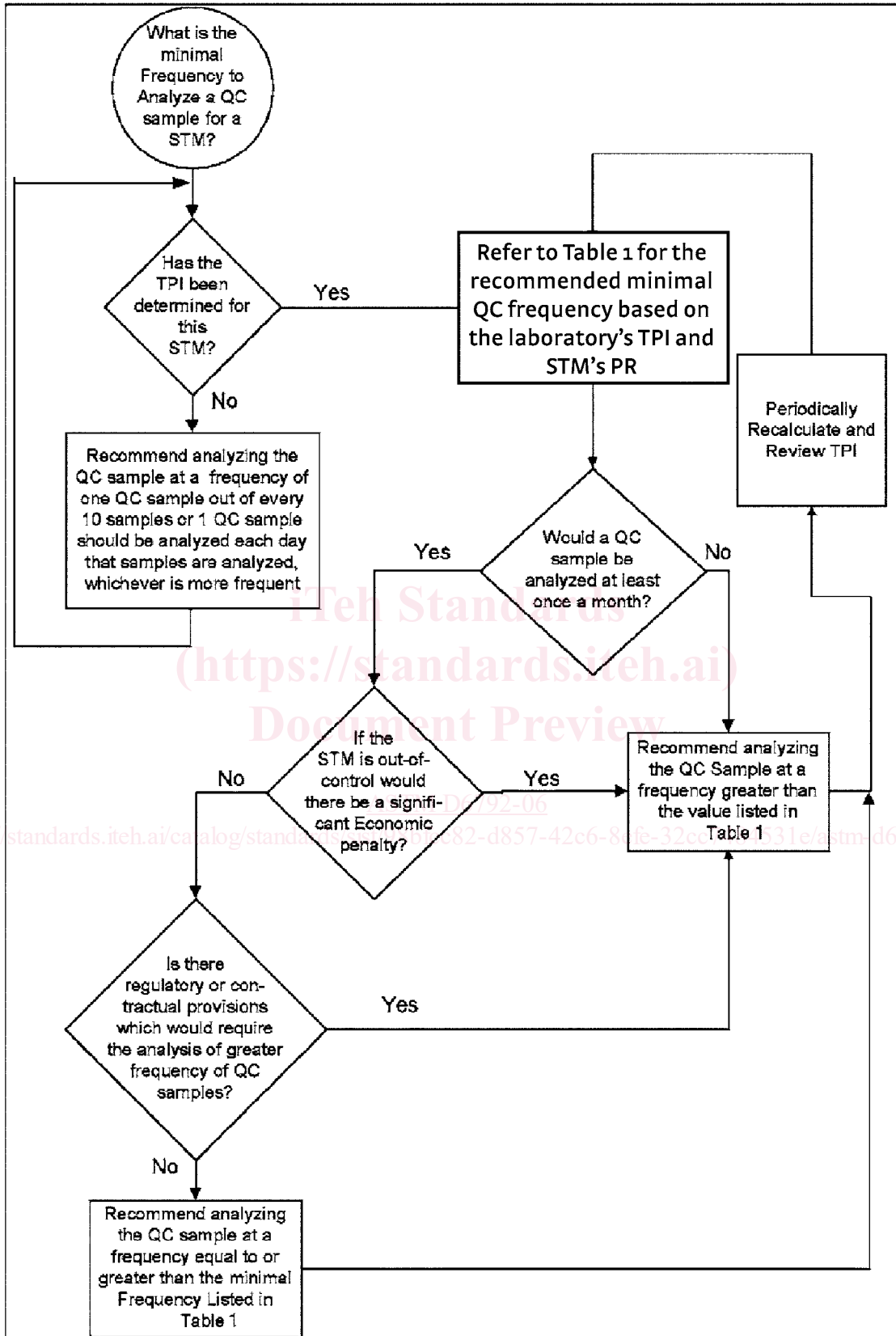


FIG. 1 Flowchart for QC Frequency