



Designation: D 3764 – 01

An American National Standard

Standard Practice for Validation of Process Stream Analyzer Systems¹

This standard is issued under the fixed designation D 3764; 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.

INTRODUCTION

Operation of a process stream analyzer system typically involves four sequential activities. (1) **Analyzer Calibration**—When an analyzer is initially installed, or after major maintenance has been performed, diagnostic testing is performed to demonstrate that the analyzer meets the manufacturer's specifications and historical performance standards. These diagnostic tests may require that the analyzer be adjusted so as to provide predetermined output levels for certain reference materials. (2) **Correlation**—Once the diagnostic testing is completed, process stream samples are analyzed using both the analyzer system and the corresponding primary test method. A mathematical function is derived that relates the analyzer output to the primary test method (PTM). The application of this mathematical function to an analyzer output produces a predicted PTM result. (3) **Initial Validation**—Once the relationship between the analyzer output and primary test method results has been established, an initial validation is performed to demonstrate that the predicted PTM results agree with those from the primary test method within the tolerances established from the correlation activities and with no statistically observable systemic bias. (4) **Continual Validation**—During normal operation of the process analyzer system, quality assurance testing is conducted to demonstrate that the agreement between the analyzer and primary test method results during the initial validation is maintained. This practice deals primarily with the third and fourth of these activities.

1. Scope

1.1 This practice describes procedures and recommendations for the validation of a total process analyzer system or its subsystems, or both, used in the direct measurement of physical or chemical characteristics of petroleum and petrochemical products. Procedures for initial validation and subsequent continuous quality assurance of system performance are described.

1.2 Validation is achieved by statistical assessment of results generated for common materials by the total analyzer system or its subsystem versus results generated by an ASTM or other established primary test method (PTM).

1.2.1 For analyzers used in product certification, the analyzer system precision determined by the statistical assessment is typically compared to the site precision for the PTM.

1.2.2 For other analyzer applications, analyzer system precision determined by the statistical assessment is compared to prespecified performance criteria based on the intended use.

1.3 Two procedures for validation are described: the line sample procedure and the validation reference material (VRM) injection procedure.

1.4 Only the analyzer system or subsystem downstream of the VRM injection point or the line sample extraction point is being validated by this practice.

1.5 The line sample procedure is limited to applications where material can be safely withdrawn from the sampling point of the analyzer unit without significantly altering the property of interest.

1.6 Validation information obtained in the application of this practice is applicable only to the type and property range of the materials used to perform the validation.

1.7 Procedures for conducting an initial validation are described. These procedures are typically conducted at installation or after major maintenance once the system mechanical fitness-for-use has been established.

1.8 Procedures for the continual validation of system performance are described. These procedures are typically applied at a frequency commensurate with the criticality of the application.

1.9 This practice applies if the process stream analyzer system and the primary test method are based on the same measurement principle(s), or, if the process stream analyzer

¹ This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.25 on Validation of Process Analyzers.

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system uses a direct and well-understood measurement principle that is similar to the measurement principle of the primary test method it is intended to predict.

1.10 This practice is not intended for use if the process stream analyzer system utilizes an indirect or mathematically modeled measurement principle such as chemometric or multivariate analysis techniques. Users should refer to Practice **D 6122** for detailed validation procedures for these types of analyzer systems.

1.11 This practice does not address procedures for diagnosing causes of validation failure.

1.12 This practice does not address the methodology for establishing the correlation equation used to generate predicted PTM results using analyzer outputs, nor the expected prediction error. The former is assumed to have been correctly developed as part of the analyzer application development work.

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

2. Referenced Documents

2.1 ASTM Standards:

D 1265 Practice for Sampling Liquefied Petroleum (LP) Gases (Manual Method)²

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products³

D 5842 Practice for Sampling and Handling of Fuels for Volatility Measurements⁴

D 6122 Practice for Validation of Multivariate Process Infrared Spectrophotometers⁵

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance⁵

E 456 Terminology Relating to Quality and Statistics⁶

F 307 Practice for Sampling Pressurized Gas for Gas Analysis⁷

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 scientific principles, (2) an assigned or certified value, based on experimental work of some national or international organization, or (3) a consensus or certified value, based on collaborative experimental work under the auspices of a scientific or engineering group.

3.1.2 *precision, n*—the closeness of agreement between independent test results obtained under stipulated conditions. **E 456**

3.1.3 *repeatability conditions, n*—conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time. **E 456**

3.1.4 *reproducibility conditions, n*—conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment. **E 456**

3.1.5 *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**

3.1.5.1 *Discussion*—A measurement system may comprise multiple instrument being used for the same test method.

3.1.6 *site precision, n*—2.77 times the standard deviation of results obtained under site precision conditions. **D 6299**

3.2 Definitions of Terms Specific to This Standard:

3.2.1 Analyzer System Items:

3.2.1.1 *analyzer output, n*—a signal (pneumatic, electrical, or digital), proportional to the property being measured that is suitable for readout or control instrumentation external to the analyzer system.

3.2.1.2 *analyzer system result, n*—the measured property reading, in the accepted property measurement units, that is displayed by the analyzer unit readout instrumentation or transmitted to end user of the analyzer system.

3.2.1.3 *analyzer unit, n*—the instrumental equipment necessary to automatically measure the physical or chemical property of a process or product stream sample using either an intermittent or a continuous technique.

3.2.1.4 *analyzer unit repeatability, n*—2.77 times the standard deviation of results obtained from repetitive analysis of the same material directly injected into the analyzer unit under repeatability conditions.

3.2.1.5 *continuous analyzer unit, n*—an analyzer that measures the property value of a process or product stream on a continuous basis and dynamically displays the instantaneously updated analyzer output.

3.2.1.6 *intermittent analyzer unit, n*—a cyclic type analyzer that performs a measurement sequence on samples from a process or product stream and displays a new analyzer output at the conclusion of each cycle.

3.2.1.7 *total analyzer system, n*—the complete analyzer system inclusive of the sample loop, sample conditioning unit, analyzer unit, readout instrumentation, and excess sample return system (see **Fig. 1**).

3.2.2 Time Unit Items—General Terms:

3.2.2.1 *analyzer unit cycle time, n*—for intermittent analyzers, the time interval between successive updates of the analyzer output.

² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 05.02.

⁴ Annual Book of ASTM Standards, Vol 05.03.

⁵ Annual Book of ASTM Standards, Vol 05.04.

⁶ Annual Book of ASTM Standards, Vol 14.02.

⁷ Annual Book of ASTM Standards, Vol 15.03.

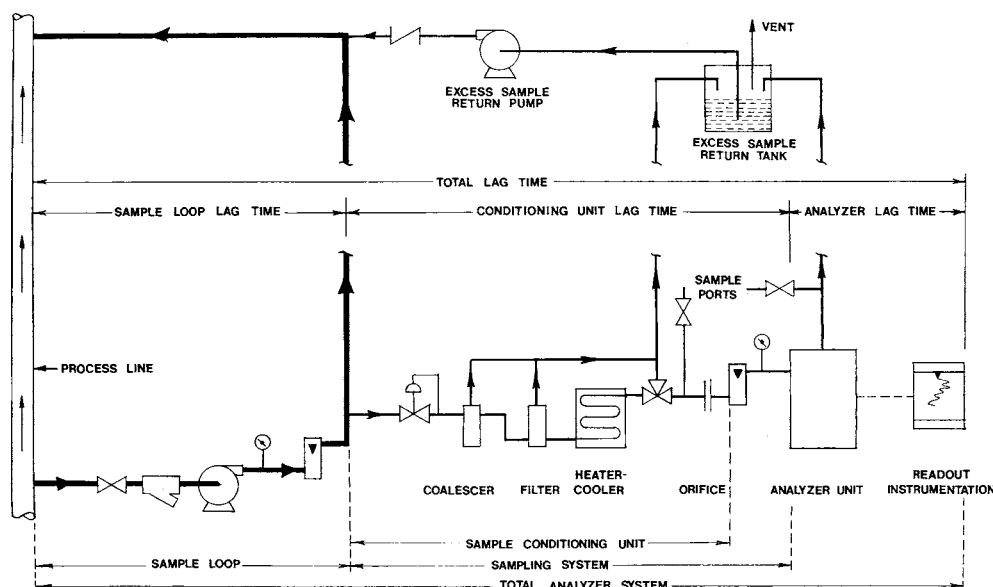


FIG. 1 Total Analyzer System

3.2.2.2 *analyzer unit dead time, n* —the time interval between the introduction of a step change in property characteristic at the inlet of the analyzer unit and the initial indication of analyzer response to this change.

(1) *Discussion*—For intermittent analyzers, if the analyzer dead time is less than one analyzer unit cycle time, the analyzer unit dead time cannot be directly measured.

3.2.2.3 *analyzer unit response time, n* —(see Fig. 2) the time interval between the introduction of a step change in property characteristic at the inlet of the analyzer unit and when the analyzer output indicates a value corresponding to 99.5 % of the subsequent change in analyzer results;

(1) *Discussion*—For continuous and intermittent analyzers with sufficiently short cycle times, the total analyzer response time is the analyzer dead time plus three times the analyzer unit time constant. For intermittent analyzers with long cycle times, the analyzer unit response time is effectively equal to the analyzer unit cycle time. For intermittent analyzers with intermediate cycle times, the analyzer unit response time should be defined as the multiple of the analyzer unit cycle time needed to exceed 99.5 % response.

3.2.2.4 *analyzer unit time constant, n* —(see Fig. 2) the time interval between the initial response of the analyzer unit to a

step change in property characteristic and when the analyzer output indicates a value corresponding to 63 % of the subsequent change in analyzer results.

(1) *Discussion*—For intermittent analyzers, if the analyzer unit time constant is less than one analyzer unit cycle time, the analyzer time constant cannot be directly measured.

3.2.2.5 *lag time, n* —the time required for material to travel from Point A to Point B in the total analyzer system (Points A and B are user-defined)

(1) *Discussion*—Lag time is a function of an analyzer system design parameters such as length and diameter of lines, number of fittings, flow restrictions, and the flow rate of the material (process or product stream) through the analyzer system (see Fig. 1 and Fig. 2).

3.2.2.6 *sample conditioning unit lag time, n* —the time required for material to travel from the start of the sample conditioning unit to the analyzer unit inlet.

3.2.2.7 *sample loop lag time, n* —the time required for material to travel from the process takeoff point of the sample loop to start of the sample conditioning unit.

3.2.2.8 *total analyzer system response time, n* —(see Fig. 2) The time interval between when a step change in property characteristic at the sample loop inlet and when the analyzer

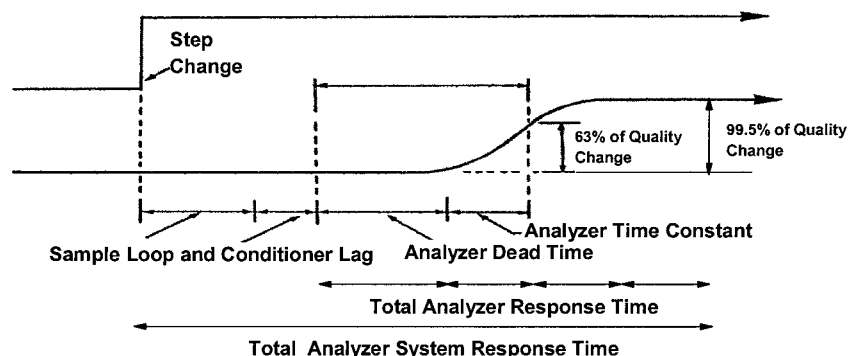


FIG. 2 Analyzer Time Units