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Standard <u>GuidePractice</u> for Generating a Process Stream Property Value through the Application of a Process Stream Analyzer¹

This standard is issued under the fixed designation D7825; 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 and provides a workflow overview of the necessary steps related to generating a Process Stream Property Value obtained from the application of a process stream analyzer.

1.2 Generating a Process Stream Property Value from the application of a process stream analyzer requires the use of several ASTM standards. These standards describe procedures to collect a representative sample, establish and validate the relationship to the primary test method, and calculate a property value with an expected uncertainty. Each standard builds or prepares data, or both, to be used in another standard. The workflow process culminates to produce a process stream analyzer result that represents a user defined batch of product. The sequence in which the standards are to be utilized is defined in this guide.

1.3 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:²

D3764 Practice for Validation of the Performance of Process Stream Analyzer Systems

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

- D6122 Practice for Validation of the Performance of Multivariate Online, At-Line, and Laboratory Infrared Spectrophotometer Based Analyzer Systems
- D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
- D6624 Practice for Determining a Flow-Proportioned Average Property Value (FPAPV) for a Collected Batch of Process Stream Material Using Stream Analyzer Data
- D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material
- D7235 Guide for Establishing a Linear Correlation Relationship Between Analyzer and Primary Test Method Results Using Relevant ASTM Standard Practices

D7278 Guide for Prediction of Analyzer Sample System Lag Times

- D7453 Practice for Sampling of Petroleum Products for Analysis by Process Stream Analyzers and for Process Stream Analyzer System Validation
- D7808 Practice For Determining the Site Precision of a Process Stream Analyzer on Process Stream Material

E1655 Practices for Infrared Multivariate Quantitative Analysis

E2617 Practice for Validation of Empirically Derived Multivariate Calibrations

3. Terminology

3.1 Definitions—Please refer to the individually cited ASTM standards for definitions.

3.2 Acronyms:

¹ This test method practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.25 on Performance Assessment and Validation of Process Stream Analyzer Systems.

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² 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.

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3.2.1 *FPAPV(s)*—Flow Proportional Average Property Value(s)

3.2.2 MLR-Multilinear Regression

3.2.3 PCR-Principal Components Regression

3.2.4 PLS-Partial Least Squares

3.2.5 PSPV(s)—Process Stream Property Value(s)

3.2.6 PTM-Primary Test Method

3.2.7 PTMR(s)—Primary Test Method Result(s)

3.2.8 PPTMR(s)—Predicted Primary Test Method Result(s)

3.2.9 QC-Quality Control

3.2.10 UAR(s)—Uncorrected Analyzer Result(s)

4. Significance and Use

4.1 The standards employed in the Process Stream Analyzer PSPV Generation Flow Diagram each have a specific deliverable that when combined into a single system produces a PSPV enabling the representation of product by process stream analyzer.

4.2 The description of each standard in the process provides the user with an overview of the application of the standard in the process for developing a PSPV.

5. Flow Diagram and Work Process

5.1 A flow chart showing the process for generating a PSPV is shown in Fig. 1.

5.2 The various standards shown in the flow chart are applied in sequence, building on the results of the previous standards. The end result is a PSPV which is expected to agree with a PTMR for the same material to within the user-specified requirements.

6. Supporting Practices

6.1 While practices D6299 and D6708 do not appear directly as part of the Flow Diagram/work process, the methodologies described in these two practices are incorporated by reference.

6.2 D6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance:

6.2.1 The statistical quality control procedures and tools described in D6299 are an integral part to the validation of analyzers by D3764 or D6122. ASTM D7825-18

6.3 D6708 Standard Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material:

6.3.1 D6708 covers statistical methodology for assessing the expected agreement between two standard test methods that purport to measure the same property of a material, and for deciding if a simple linear bias correction can further improve the expected agreement.

6.3.2 Practices D3764, D6122 and D7235 which are part of the PSPV generation work process all make use of the statistical methodology described in D6708.

7. Sampling and Sample Delivery

7.1 D7453 Practice for Sampling of Petroleum Products for Analysis by Process Stream Analyzer System Validation:

7.1.1 Sampling is the initial process in the generation of a process stream property value, equally important as any other process in the chain it provides the material that everything will be based on. The sampling standard provides guidance on how to collect a representative sample from the sample stream and deliver it to the desired sample destination (line sample point, on-line analyzer, or composite sampler).

7.1.2 This sampling method is focused on sample stream delivery without the contamination that can be found in process streams. The end use of the sample is to determine physical properties of the sample so filtering and coalescing are required to protect the on-line analytical system. Sampling methods like D4177 are designed to collect a representative amount of sediment and water so are not conducive to proper analyzer system operation.

7.2 D7278 Standard Guide for Predicting and Measuring Lag Times for On-Line Sampling:

7.2.1 The time it takes for a sample to travel from the process stream to the analyzer inlet or line sample point, including the required flush volume, is a critical piece of information when establishing the relationship between the on-line analyzer and the primary test method (D7235) and also the validation of the on-line system (D3764 and D6122).

7.2.2 The lag time needs to be taken into account when collecting samples and recording analyzer reading to correlate with lab sample results and process conditions.

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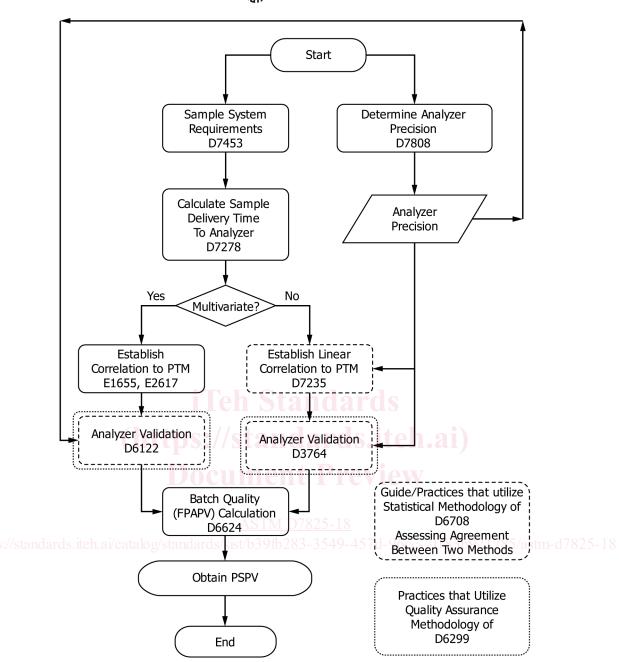


FIG. 1 Process Stream Analyzer PSPV Generation Flow Diagram

8. Analyzer System Site Precision

8.1 D7808 Standard Guide for Determining the Site Precision of a Process Stream Analyzer on Process Stream Material: 8.1.1 To properly apply D7235, and as an extension of D7235 the application of D6708, the user needs to have determined the analyzer site precision over the expected process stream operating range. The process employed to determine the Site Precision of a Process Stream Analyzer on Process Stream Material results can be utilized as part of a QC program as described in D6299.

8.1.2 D3764 and D6122 require that the user has determined analyzer site precision in order to verify that the process analyzer system is at steady state during the validation process.

8.1.3 To reliably calculate the analyzer site precision, infrastructure must be in place to repeatedly introduce aliquots of one or more bulk samples under the same conditions as the sample stream.

9. Analyzer Calibration

9.1 D7235 Standard Guide for Establishing a Linear Correlation Relationship between Analyzer and Primary Test Method Results using Relevant ASTM Standard Practices:



9.1.1 Guide D7235 covers a general methodology to develop and access the linear relationship between uncorrected analyzer results (UARs) produced by a total analyzer system versus results produced by the corresponding primary test method (PTMRs) that the analyzer is intended to emulate.

9.1.2 Guide D7235 describes how the statistical methodology of Practice D6708 is employed to access the agreement between the PTM and the analyzer results, and if necessary, develop a linear correlation to improve the agreement over the complete operating range of the analyzer.

9.1.3 Guide D7235 applies two either of the following two cases: (1) the process stream analyzer system and the primary test method are based on the same measurement principle(s), or, (2) the process stream analyzer system uses a direct and well-understood measurement principle that is similar to the measurement principle of the primary test method.

9.1.4 If the process stream analyzer system uses a different measurement technology from the PTM, this practice also applies provided that the calibration protocol for the direct output of the analyzer does not require use of the PTM.

9.1.5 Procedures are described to ensure that the sample set used to generate the linear correlation are representative of the material type and property range for the intended analyzer service, and to ensure that the set provides adequate variation in property level.

9.1.6 Preferably, line samples are collected in accordance with Practice D3764 from a sampling point after the sample conditioning system. Taking into account the analyzer lag time (D7278), corresponding analyzer results are obtained. The line samples are measured by the PTM. The UARs and PTMRs are accessed using the statistical methodology of D6708 to determine if there is an adequate linear relationship to allow PSPVs values to be estimated based on UARs.

9.1.7 Alternatively, the statistical methodology of D6708 can be used to correlate PTMRs obtained from composite samples to FPAPVs generated from the analyzer results using the methodology of D6624.

9.1.8 PPTMRs are generated by applying the linear correlation to the measured analyzer result.

9.2 E1655 Standard Practices for Infrared Multivariate Quantitative Analysis:

9.2.1 Practice E1655 may be used to develop the calibration if the process stream analyzer system utilizes an indirect or mathematically modeled measurement principle such as chemometric or multivariate analysis techniques where results from PTM are required for the development of the chemometric or multivariate model.

Note 1—While the practices described within E1655 deal specifically with mid- and near-infrared analysis, much of the mathematical and procedural detail contained therein is also applicable to other analytical methods. The user of E1655 is cautioned that typical and best practices for multivariate quantitative analysis using data from other multivariate analytical techniques may differ from that described in E1655 for mid- and near-infrared spectroscopies.

9.2.2 E1655 describes procedures for collection and treating data for developing multivariate calibrations. Multivariate mathematics are applied to correlate spectra measured for a set of calibration samples to PTMRs for this same set. The resultant multivariate calibration model is applied to the analysis of the spectrum of an unknown sample to estimate the PPTMR for that sample. Application of E1655 is limited to models developed by MLR, PCR or PLS.

9.2.3 E1655 describes procedures for validating the calibration model. This validation tests for bias in model predictions, and for the expected agreement between the PPTMRs and PTMRs. Validation of the model is intended to demonstrate the multivariate models capabilities, but it is not intended as a measure of analyzer performance. The performance of multivariate analyzer systems must be validated using procedures described in practice D6122.

9.2.4 E1655 describes statistical tests which are employed to detect when samples being analyzed exceed the range for which the multivariate model has been validated. The analysis of such "outlier" samples represents an extrapolation of the model, and there is lower confidence that PPTMRs predicted by extrapolation will agree with PTMRs.

9.3 E2617 Standard Practice for Validation of Empirically Derived Multivariate Calibrations:

9.3.1 If the multivariate model is developed using techniques other than MLR, PCR or PLS, then the model itself should be validated using E2617. As with E1655, the validation of the model is intended to demonstrate the multivariate models capabilities, but it is not intended as a measure of analyzer performance.

9.3.2 For calibrations developed using Practice E2617, Practice D6122 is still used to validate analyzer performance.

10. Validation of Process Analyzer Performance

10.1 D3764 Practice for Validation of Process Analyzers:

10.1.1 For analyzers where Guide D7235 was used to establish the linear correlation relationship between UARs and PTMRs, Practice D3764 is applied for the validation of performance.

10.1.2 After the analyzer system has been calibrated per the manufactures requirements, and the correlation between the system and a PTM has been completed (D7235) and implemented, the analyzer system is ready for validation. Analyzer validation is proving that all the previous steps in the work process have been successfully carried out and that the analyzer system produces PPTMRs that predict the PTM results to within the user's expectations and requirements.

10.1.3 After an analyzer is installed, or major maintenance is conducted, a probationary validation is performed to demonstrate that the PPTMRs agree with the PTMRs to within the user-specified requirements for the analyzer system. The probationary validation is conducted using a limited set of materials which were not used in developing the linear correlation. Once the analyzer passes the probationary validation, it may be put into service to generate PPTMRs.