



Standard Guide for Establishing a Linear Correlation Relationship Between Analyzer and Primary Test Method Results Using Relevant ASTM Standard Practices¹

This standard is issued under the fixed designation D7235; 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 will typically be performed to demonstrate that the analyzer meets 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 to Primary Test Method*—For process stream analyzer systems where the application objective is to provide prediction of results from a Primary Test method, once the diagnostic testing is completed, process stream samples will typically be analyzed using both the analyzer system and the corresponding primary test method. A mathematical function will be 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 using an independent data set 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 analyzer and primary test method results during the Initial Validation is maintained. This document provides guidance for item (2) above.

1. Scope*

1.1 This guide covers a general methodology to develop and assess the linear relationship between results produced by a total analyzer system versus the results produced by the corresponding primary test method (PTM) that the analyzer system is intended to emulate, using the principles and approaches outlined in relevant ASTM standard practices and guides.

1.2 This guide describes how the statistical methodology of Practice **D6708** can be employed to assess agreement between the PTM and analyzer results, and, if necessary, develop linear correlation to further improve the agreement over the complete

operating range of the analyzer. For instances where there is insufficient variation in property level to apply the Practice **D6708** multi-level methodology, users are referred to Practice **D3764** to perform a level specific bias evaluation. The correlation relationship information obtained in the application of this guide is applicable only to the material type and property range of the materials representative of those used to perform the assessment. Users are cautioned against extrapolation of the relationship beyond the material type and property range being studied.

1.3 This guide 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 system uses a direct and well-understood measurement principle that is similar to the measurement principle of the primary test method. If the process stream analyzer system uses a different measurement technology from the primary test method, provided that the calibration protocol for the direct output of the analyzer does not require use of the PTM, this practice also applies.

¹ This guide 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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*A Summary of Changes section appears at the end of this standard

1.4 This guide does not apply 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 chemometric or multivariate model development. Users should refer to Practices [E1655](#) and [D6122](#) for detailed correlation and model validation procedures for these types of analyzer systems.

NOTE 1—For example, this guide would apply for the comparison of benzene measurements from a mid-infrared process analyzer system based on Test Method [D6277](#) to those obtained using PTM Test Method [D3606](#), a gas chromatography based test method. For each sample, the mid-infrared spectrum is converted into a single analyzer result using methodology (Test Method [D6277](#)) that is independent of the primary test method (Test Method [D3606](#)). However, when the same analyzer uses a multivariate model to correlate the measured mid-infrared spectrum to Test Method [D3606](#) reference values using the methodology of Practice [E1655](#), this guide does not apply. In this case, the direct output of the analyzer is the spectrum, and the conversion of this multivariate output to an analyzer result require results from the primary test method.

1.5 This guide assumes that the analyzer sampling system is fit for use, and both analyzer and lab systems are in statistical control during the execution of the required tasks. Procedures for testing for proper function of the analyzer sampling system are beyond the scope of this guide. For ascertaining whether the systems are in statistical control, refer to Practice [D6299](#) or other technical equivalent documents.

1.6 *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

- [D3606 Test Method for Determination of Benzene and Toluene in Finished Motor and Aviation Gasoline by Gas Chromatography](#)
- [D3764 Practice for Validation of the Performance of Process Stream Analyzer Systems](#)
- [D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products](#)
- [D5191 Test Method for Vapor Pressure of Petroleum Products \(Mini Method\)](#)
- [D6122 Practice for Validation of the Performance of Multivariate Online, At-Line, and Laboratory Infrared Spectrophotometer Based Analyzer Systems](#)
- [D6277 Test Method for Determination of Benzene in Spark-Ignition Engine Fuels Using Mid Infrared Spectroscopy](#)
- [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](#)

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.

- [D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material](#)
- [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](#)

2.2 American Petroleum Institute Document:³

- [API TP-550 Manual on Installation of Refinery Instruments and Control Systems, Part II, Process Stream Analyzers](#)

3. Terminology

3.1 All of the terminology as defined in Practices [D3764](#), [D6122](#), and [D6708](#) are adopted for this guide.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *primary test method (PTM), n*—test method designated by the user of this guide such that the process analyzer system results are estimates or prediction of PTM results that would have been obtained if applied to the same material.

3.2.1.1 *Discussion*—It is not the intent of this guide to define a test method. Within the context of the intended application of this practice, a PTM can be any user-designated test method that the process analyzer system test results are intended to estimate or predict.

4. Significance and Use

4.1 This guide is intended to be used in conjunction with Practice [D3764](#) (Case 1) and Practice [D6122](#) (Case 2). Methodology in this guide can be used to determine if a linear correlation can improve the performance of the total analyzer system in terms of its ability to predict the results that the PTM would have been if applied to the same material. This methodology, which is based on the same statistical data treatment as Practice [D6708](#), is use to derive the parameters of the linear relationship and to assess the degree of improvement.

4.2 This guide provides developers or manufacturers of process stream analyzer systems with useful procedures for developing the capability of newly designed systems for industrial applications that require reliable prediction of measurements of a specific property by a primary test method of a flowing component or product.

4.3 This guide provides purchasers of process stream analyzer systems with some reliable options for specifying performance requirements for process stream analyzer systems that are used in applications requiring reliable prediction of measurements of a specific property by a primary test method of a flowing component or product.

4.4 This guide provides the user of a process stream analyzer system with useful information on the work process for establishing the PTM prediction relationship and prediction performance.

³ Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, <http://www.api.org>.

4.5 Prediction (correlation) relationship obtained in the application of this guide is applicable only to the material type and property range of the materials used to perform the study. Selection of the property levels and the compositional characteristics of the samples must be suitable for the application of the analyzer system. Users are cautioned against extrapolation of the prediction relationship beyond the material type and property range used to obtain the relationship.

4.6 The degree-of-agreement assessment promoted in this guide is based on the statistical principles articulated in Practice **D6708**, which is purely statistical in nature. No attempt is made in assessing the degree of similarity in the analytical technique between the process analyzer and the PTM; hence, results between the PTM and analyzer unit can be highly correlated, but their measurement principles may be completely different, and may *not* be the principal cause for correlation. Users are therefore cautioned that a high degree of correlation between results does not necessarily imply a high degree of similarity in the measurement principles; nor does it imply a similar degree of agreement can be expected in future measurements. In general, if sample-specific biases are detected, it suggests that the measurement principles may be different, and may affect the degree-of-agreement in future use of the scaling/bias-correction equation. Presence or absence of sample-specific effect can be used as a measure of the robustness of the correlation equation to sample composition or matrix differences.

4.7 Implementation of this guide requires that the process stream analyzer system complies with the following conditions:

4.7.1 Meets the principles set forth in PART II Process Stream Analyzers of API TP-550,

4.7.2 Meets the supplier's recommendation,

4.7.3 Complies with operating conditions specified by the manufacturer,

4.7.4 A predicted PTM algorithm has already been established if necessary, and

4.7.5 Meets applicable quality assurance, data collection and data telemetry protocols.

4.8 After installation or major maintenance, conduct such diagnostic tests as recommended by the manufacturer to demonstrate that the analyzer meets manufacturer's specifications, historical performance levels or both. If necessary, adjust the analyzer system components so as to obtain recommended analyzer output levels for specified reference materials.

4.9 Inspect the entire analyzer system to ensure it is installed properly, is in operating condition, and is properly adjusted after completion of the initial commissioning procedures.

5. Analyzer Calibration Adjustments and Diagnostics Tests

5.1 When an analyzer is initially installed or after major maintenance has been performed, diagnostic tests should be conducted to demonstrate that the analyzer meets 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. Such adjustment may be done in hardware, software or both. This should not be confused with the development of correlation to a PTM, which, is described below.

6. Correlation to Primary Test Method

6.1 General Approach:

6.1.1 Define the sample set to be used for assessment.

6.1.1.1 The material type and property range for which the analyzer system results are to be assessed versus the primary test method is defined.

6.1.1.2 The recommended sample set design criteria for this assessment are:

(1) A minimum of six replicates at each major product/property level combination,

(2) The range of major product/property levels exceed at least two times the published reproducibility of the PTM, and

(3) A minimum of 30 total samples.

6.1.1.3 Replication at a specific level/product combination can be different batches of production material that are nominally similar in property level (within 1.2 times the ASTM reproducibility of the PTM) and composition.

6.1.2 Obtain site precision information for the analyzer system and PTM for the material type and range defined in accordance with the procedures outlined in Practice **D7808** and **D6299**, respectively. If analyzer system precision cannot be obtained using the methodology in Practice **D7808**, manufacturer's published repeatability precision or site precision from other similar systems may be used as a surrogate. Users are cautioned to ensure consistency in statistical definitions between the manufacturer's published repeatability and the site precision statistic as defined in Practices **D6299** and **D7808**.

SAMPLING METHOD

6.1.3 *Line Sampling (Preferred)*—Samples meeting the requirements of the sample set design criteria above are taken in accordance with Practice **D3764**, Line Sample Procedure, at the crosscheck sample points of the analyzer system (see **Fig. 1**), after the sample conditioning subsystem, at a frequency of no more than once per day. Avoid taking this sample at the same time of day to ensure any time-of-day related effect is captured in the dataset.

6.1.4 *Automated Composite Sampling*—For installations that have automated composite sampling systems meeting the requirement of Practice **D4177** or **D7453** (or equivalent), and, the application is intended to provide a predicted PTM result for a batch of homogenous production material using an Flow-Proportioned Average Property Value (FPAPV) calculated from the analyzer system as per Practice **D6624**, the correlation equation can be established using samples taken from the composite sample accumulator at the end of each batch of production meeting the requirements of the sample set design above. While this approach is theoretically equivalent to the line sampling approach, it is vulnerable to failure of assumptions that the requirements of Practices **D6244** and **D4177** or **D7453** are met.

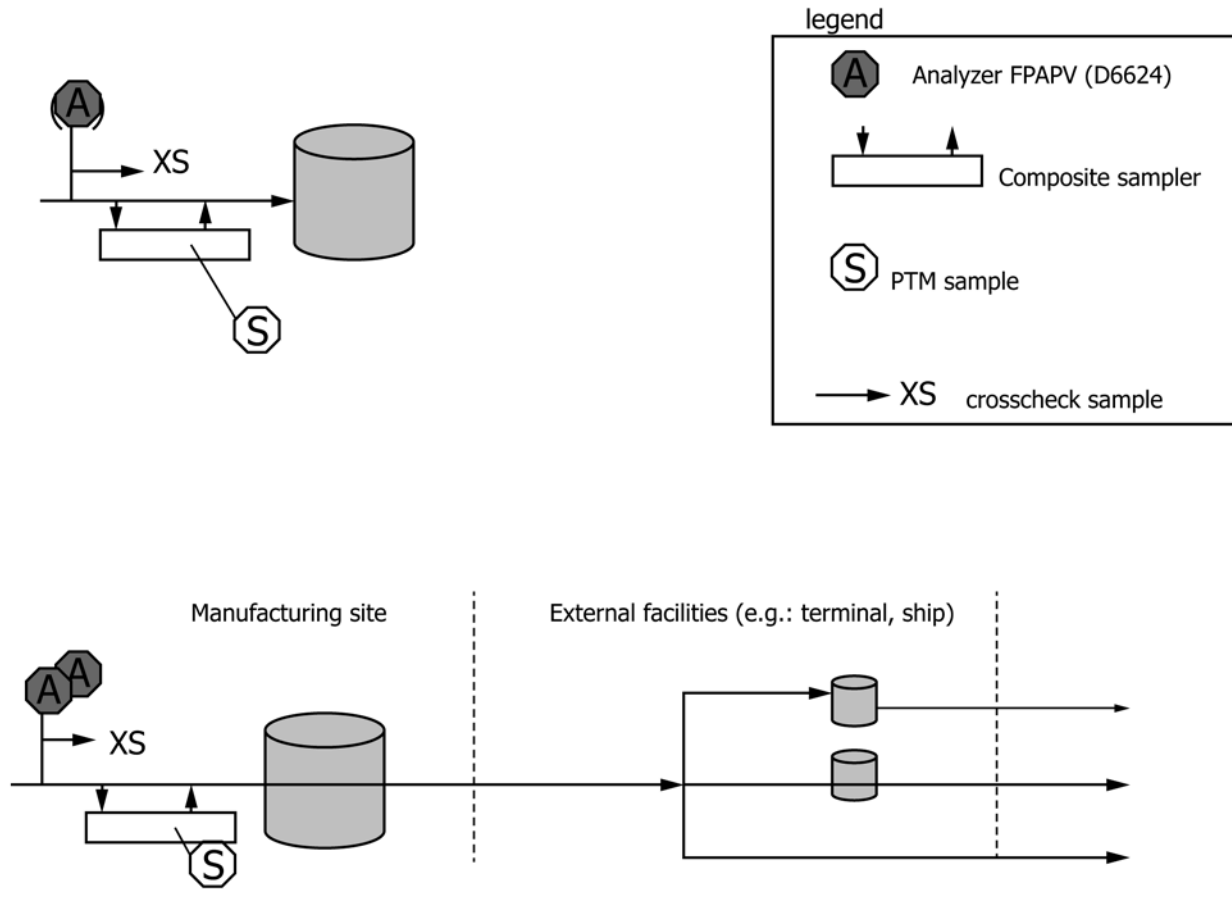


FIG. 1 Common Analyzer System Configurations

ASSESSMENT OF TEST RESULTS

6.1.5 For Line Sampling method, assess the degree of agreement between results generated by the analyzer unit, captured at the time of sampling and the corresponding PTM.

6.1.6 For Automated Composite Sampling method, assess the degree of agreement between the FPAPV value generated in accordance with Practice D6624 using a non-bias corrected analyzer output for each composite sample versus the corresponding PTM result.

6.1.7 Using site precision information, and statistical principles/calculations of Practice D6708 (see Annex A1 for an example of assessment), answer the following questions:

- (A) Is there adequate variation in the property level of the sample set relative to the PTM and analyzer unit site precisions?
- (B) Is there adequate correlation between the test results from the PTM and the analyzer unit?
- (C) Will a scaling/bias correction significantly improve the agreement between the analyzer unit results and the PTM results over and above their combined site precisions?
- (D1) Are there sample-specific biases?
- (D2) If yes to (D1), can these biases be treated as a random effect?
- (D3) If no to (D1), are the residuals randomly scattered?

6.1.7.1 Refer to Table 1 for the assessment outcomes associated with the answers to questions in 6.1.7.

6.1.7.2 If the D6708-based assessment outcome is successful, the scaling/bias-correction from D6708 assessment are mathematically applied to the analyzer unit results to produce a predicted-PTM result by the process analyzer system.

6.1.8 Obtain the dataset comprising (predicted PTM result – PTM result). This dataset is commonly known as the prediction residuals.

6.1.8.1 Plot the residuals in chronological order (a run chart as per Practice D6299).

6.1.8.2 Examine the plot for any cyclic or unusual patterns.

6.1.8.3 Calculate the root-mean-square standard deviation of the residuals (see Annex for example).

6.1.9 Interpret the usefulness of the degree-of-agreement between the scaling/bias-corrected analyzer unit and PTM test results versus the intended analyzer application, taking into account any sample specific biases observed, the magnitude of the standard deviation of the residuals when compared to the PTM site precision standard deviation, and how the scaling/bias-corrected analyzer results is used. It is recommended that this interpretation be carried out by a team comprising subject matter expertise in analytical technology, the manufacturing process that the analyzer system is intended to monitor, and statistical modeling.

6.1.10 If the degree of agreement after scaling/bias-correction is deemed useful, apply the correction to the analyzer unit test results mathematically in the routine use of the process analyzer system.

6.1.11 For total analyzer systems that are used to generate FPAPV (per Practice D6624), the predicted-PTM result can be used to generate a bias-corrected FPAPV that is intended to

TABLE 1 Assessment Outcomes^A

A	B	C	D1	D2	D3	Assessment Outcome
Is there adequate variation in the property level of the sample set relative to the PTM and analyzer unit site precisions?	Is there adequate correlation between the test results from the PTM and the analyzer unit?	Will a scaling/bias correction significantly improve the agreement between the analyzer unit results and the PTM results over and above their combined site precisions?	Are there sample-specific biases?	If yes to (D1), can these biases be treated as a random effect?	If no to (D1), are the residuals randomly scattered?	
Yes	Yes	No	No	N/A	Yes	Pass
Yes	Yes	No	No	N/A	No	Fail
Yes	Yes	No	Yes	Yes	N/A	Pass
Yes	Yes	No	Yes	No	N/A	Fail
Yes	Yes	Yes	No	N/A	Yes	Pass
Yes	Yes	Yes	No	N/A	No	Fail
Yes	Yes	Yes	Yes	Yes	N/A	Pass
Yes	Yes	Yes	Yes	No	N/A	Fail
Yes	No	N/A	N/A	N/A	N/A	Fail
No	N/A	N/A	N/A	N/A	N/A	Fail

^A Boldfaced type indicates reason for failure.

predict the batch quality if the PTM had been used to analyze a representative sample of the homogenized batch of material.

6.1.12 The scaling/bias-correction equation is continually validated using the predicted-PTM result and actual PTM results in accordance with the line sample procedure of Practice **D3764** and the I / MR/ EWMA control charting technique in Practice **D6299**.

6.1.13 For FPAPV applications, the complete FPAPV system should be continually validated using predicted-PTM derived FPAPV and PTM results obtained on samples that the FPAPV is intended to represent, in accordance with the I / MR / EWMA control charting technique in Practice **D6299**.

6.1.14 Work procedures need to be developed to address out-of-control situations for the control charts described above. The specific tasks tend to be application specific, and will depend on the criticality of the application both in a real time sense, and business risk. In general, the following process of elimination should be carried out, in the order presented, to rule out the plausible root causes:

6.1.14.1 Is it due to sample difference between the PTM and the analyzer unit?

6.1.14.2 Is it due to the analyzer unit out of statistical control?

6.1.14.3 Is it due to the primary test method out of statistical control?

6.1.15 If the plausible root causes in **6.1.14** are ruled out, then it is highly likely that the out-of-control situation is due to the sample matrix being significantly different from those used to develop the scaling/bias-correction equation. Repeat the Practice **D6708** assessment using results from more current data set based on the control chart performance.

6.2 *Single-Level Bias Assessment*—Single Level Initial Validations are typically performed using the Line Sample Procedure described in Practice **D3764**.

7. Analyzer System Application Configuration

7.1 **Fig. 1** illustrates two common analyzer system application configurations applicable to this guide. **Figs. 2 and 3** are reproduced from Practice **D3764** to provide additional guidance.

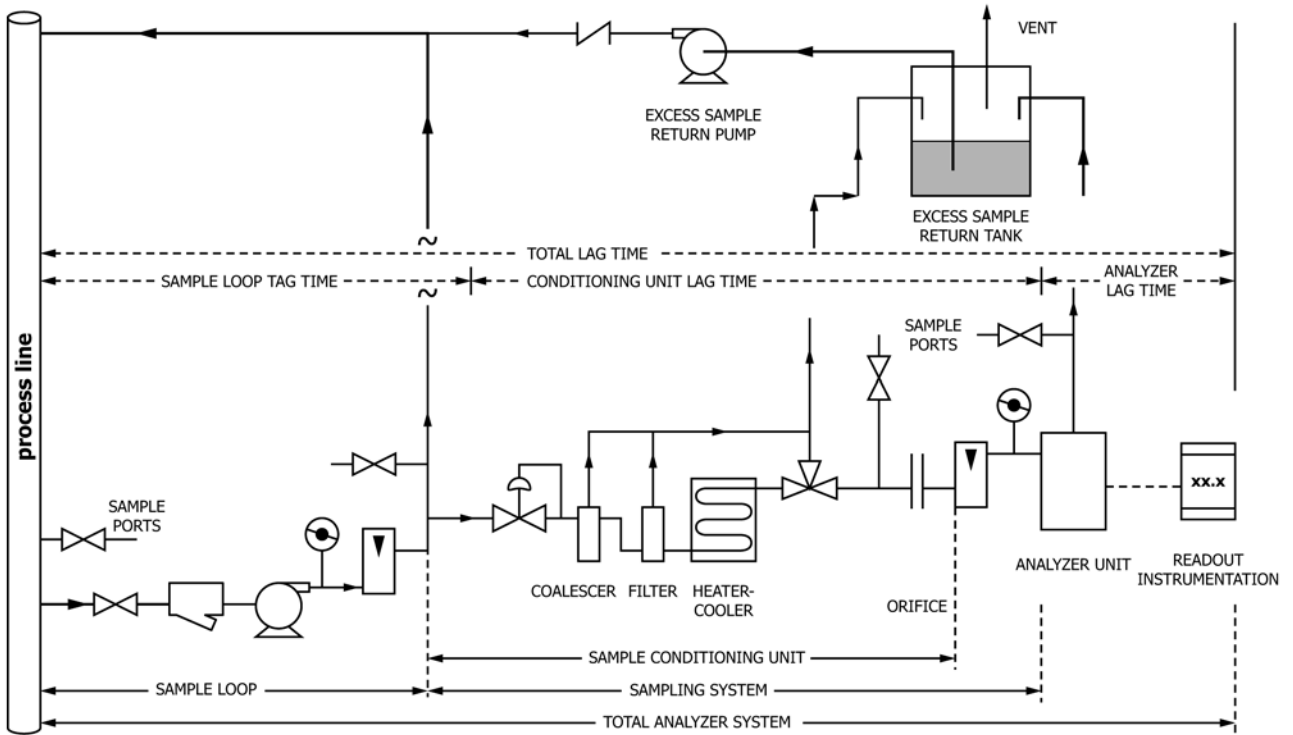


FIG. 2 Total Analyzer System

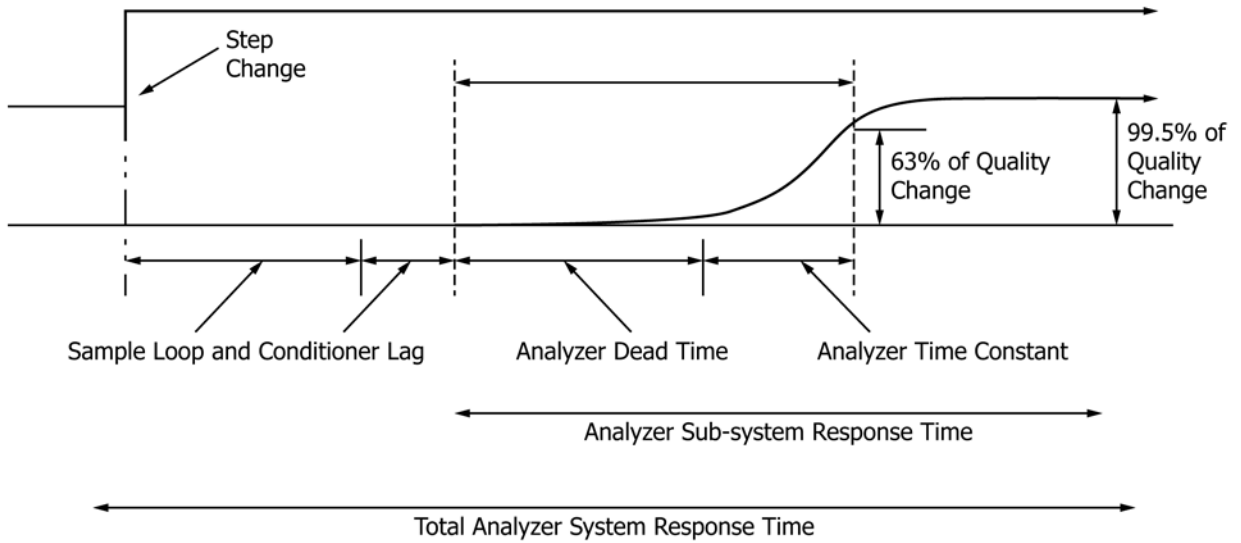


FIG. 3 Analyzer Time Units

ANNEX

(Mandatory Information)

A1. ASSESSMENT EXAMPLE

A1.1 The following is an example of applying the principles articulated in this guide in conjunction with the statistical methodologies from Practices D6299 and D6708 to develop and assess the correlation equation between a field VP analyzer system versus its PTM (D5191).

A1.2 From D6299 QC assessment under site precision conditions, the standard deviations of the field analyzer (X) and lab test method (Y) are:

A1.2.1 Site precision standard deviation for analyzer X: 0.022 psi.

A1.2.2 Site precision standard deviation for lab VP test method: 0.04 psi.

A1.3 Results from X and Y on samples meeting the requirements of the sample set design criteria above, taken in accordance with Practice D3764, Line Sample Procedure, at the line sample points (XS) of the analyzer system (see Fig. 1), after the sample conditioning subsystem, are tabulated below:

X	Site Precision Std Dev for Analyzer X	No. of Analysis	Y	Site Precision Std. Dev for Lab Method Y	No. of Analysis
10.45	0.022	1	10.27	0.04	1
10.44	0.022	1	10.23	0.04	1
10.45	0.022	1	10.18	0.04	1
10.41	0.022	1	10.24	0.04	1

X	Site Precision Std Dev for Analyzer X	No. of Analysis	Y	Site Precision Std. Dev for Lab Method Y	No. of Analysis
8.9	0.022	1	8.82	0.04	1
10.46	0.022	1	10.25	0.04	1
10.47	0.022	1	10.25	0.04	1
10.41	0.022	1	10.3	0.04	1
10.28	0.022	1	10.04	0.04	1
12.59	0.022	1	12.34	0.04	1
12.63	0.022	1	12.24	0.04	1
10.44	0.022	1	10.36	0.04	1
12.52	0.022	1	12.18	0.04	1
12.63	0.022	1	12.43	0.04	1
12.5	0.022	1	12.3	0.04	1
12.65	0.022	1	12.37	0.04	1
14.09	0.022	1	13.78	0.04	1
12.54	0.022	1	12.2	0.04	1
14.04	0.022	1	13.72	0.04	1
15.36	0.022	1	15.01	0.04	1
15.74	0.022	1	15.36	0.04	1
15.7	0.022	1	15.23	0.04	1
15.76	0.022	1	15.27	0.04	1
15.8	0.022	1	15.39	0.04	1
15.75	0.022	1	15.4	0.04	1
15.8	0.022	1	15.45	0.04	1
15.78	0.022	1	15.52	0.04	1

A1.4 The data set above is used as input into ADJD6708 CompTM Version 1.0.21. Output of adjunct is reproduced in Table A1.1. The correlation equation is:

A1.4.1 Predicted lab method Y = 0.9625 (X) + 0.2 psi

TABLE A1.2 Output

CompTM, 28 May 2004, 13:08 - Summary

 TM X: ABB field analyzer vs. Grabner Lab **D5191**
 Can Test Method X distinguish among the samples?

Yes

 TM Y: Grabner results
 Can Test Method Y distinguish among the samples?

Yes

 Are the TMs correlated?
 Selected bias correction, where $y = a + bx$:

 Yes
 Class 2

 $a = 2.0169e-01, b = 9.6255e-01$

Will a bias correction significantly improve their agreement?

Yes

 Are there sample-specific biases?
 Are they random?

 Yes
 Yes

Cross Method Reproducibility:

 $R_{xy}(\text{D6708, Eq. 24}) = \text{SQRT}(4.5762e-04(X)^2 + 0.0000 + 1.6328e-03(Y)^2 + 0.0000)$

End

 CompTM - Details
 28 May 2004, 13:9:47

 TM X: ABB field analyzer vs. Grabner Lab **D5191**
 TM Y: Grabner results

	# of Labs	TSS
Initial		
TM X	27	6.2523e03
TM Y	27	7.9634e04
Intersection		
TM X	27	6.2523e03
TM Y	27	7.9634e04

Significance of difference among samples

Test ratios vs. critical values

Test	Ratio	Critical	Significant?
D6708-01			
6.2.2 TM X F	240.47	1.87	Yes
6.2.3 TM X F	3062.80	1.87	Yes

Bias correction classes

Class	CSSQ
6.3.1.2 Class 0-no correction	1.1346e03
6.3.2.2 Class 1a-constant bias correction	1.4560e02
6.3.3.7 Class 1b-proportional bias correction (when appropriate)	6.7941e01
6.3.4.8 Class 2-linear (proportional + constant bias correction)	5.1026e01

Test ratios vs. critical values

Test	Ratio	Critical	Significant?
6.4.1 Discordance F	1.558e03	1.939	Yes
6.5.2 Class 0 F	2.655e02	3.385	Yes
6.5.3 Class 2 t	2.879e00	2.060	Yes
6.5.3 Class 1a t	2.286e01	2.060	No
6.5.3 Class 1b t	2.879e00	2.060	Yes
6.6 SSBiases chi-sqr.	5.103e01	37.652	Yes

Selected Model

 $\text{Class} = \text{Class 2} \wedge Y = 2.0169e-01 + 9.6255e-01 * X$

J	Residual
6.7.2	Residuals
1	-2.61e00
2	-2.23e00
3	-1.84e00
4	-1.77e00
5	-1.60e00
6	-1.58e00
7	-1.25e00
8	-6.50e-01
9	-4.55e-01
10	-4.39e-01
11	-4.39e-01
12	-1.74e-01

13	9.10e-02
14	1.72e-01
15	2.13e-01
16	3.52e-01
17	4.00e-01
18	4.36e-01
19	5.19e-01
20	8.40e-01
21	8.81e-01
22	1.14e00
23	1.46e00
24	1.57e00
25	1.72e00
26	2.40e00
27	2.84e00

Anderson-Darling on residuals
6.7.2.3 $A^2^* = 0.211$ $A2Crit = 0.752$ Pass

Cross Method Reproducibility
6.7.3 $Rxy(D6708, Eq. 24) = \text{SQRT}(4.5762e-04(X) \wedge 0.0000 + 1.6328e-03(Y \wedge 0.0000))$

SUMMARY OF CHANGES

Subcommittee D02.25 has identified the location of selected changes to this standard since the last issue (D7235 – 14) that may impact the use of this standard. (Approved April 1, 2016.)

(1) Added Practice **D7808** to Referenced Documents and to subsection **6.1.2**.

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