



Standard Practice for Validation of the Performance of Multivariate Online, At-Line, and Laboratory Infrared Spectrophotometer Based Analyzer Systems¹

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INTRODUCTION

Operation of a laboratory or 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 (PTM). 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 primary test method result (PPTMR). (3) **Probationary Validation**—Once the relationship between the analyzer output and PTMRs has been established, a probationary validation is performed using an independent but limited set of materials that were not part of the correlation activity. This probationary validation is intended to demonstrate that the PPTMRs agree with the PTMRs to within user-specified requirements for the analyzer system application. (4) **General and Continual Validation**—After an adequate number of PPTMRs and PTMRs have been accrued on materials that were not part of the correlation activity, a comprehensive statistical assessment is performed to demonstrate that the PPTMRs agree with the PTMRs to within user-specified requirements. Subsequent to a successful general validation, quality assurance control chart monitoring of the differences between PPTMR and PTMR is conducted during normal operation of the process analyzer system to demonstrate that the agreement between the PPTMRs and the PTMRs established during the General Validation is maintained. This practice deals with the third and fourth of these activities.

1. Scope*

1.1 This practice covers requirements for the validation of measurements made by laboratory or process (online or at-line) near- or mid-infrared analyzers, or both, used in the calculation of physical, chemical, or quality parameters (that is, properties) of liquid petroleum products. The properties are calculated from spectroscopic data using multivariate modeling methods. The requirements include verification of adequate instrument performance, verification of the applicability of the calibration model to the spectrum of the sample under test, and verification

of equivalence between the result calculated from the infrared measurements and the result produced by the primary test method used for the development of the calibration model. When there is adequate variation in property level, the statistical methodology of Practice D6708 is used to provide general validation of this equivalence over the complete operating range of the analyzer. For cases where there is inadequate property variation, methodology for level specific validation is used.

1.2 Performance Validation is conducted by calculating the precision and bias of the differences between results from the analyzer system (or subsystem) produced by application of the multivariate model, (such results are herein referred to as Predicted Primary Test Method Results (PPTMRs)), versus the Primary Test Method Results (PTMRs) for the same sample set. Results used in the calculation are for samples that are not

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 Performance Assessment and Validation of Process Stream Analyzer Systems.

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