This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

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Standard Practice for Validation of the Performance of Multivariate Online, At-Line, Field and Laboratory Infrared Spectrophotometer, and Raman Spectrometer Based Analyzer Systems[1]

This standard is issued under the fixed designation D6122; 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 laboratory or process stream analyzer system typically involves five sequential activities. *(1)* **Correlation**—Prior to the initiation of the procedures described in this practice, a multivariate model is derived which relates the spectrum produced by the analyzer to the Primary Test Method Result (PTMR). *(1a)* **If the analyzer and Primary Test Method (PTM) measure the same material**, then the multivariate model directly relates the spectra to PTMR collected on the same samples. Alternatively *(1b)* **if the analyzer measures the spectra of a material that is subjected to treatment prior to being measured by the PTM**, then the multivariate model relates the spectra of the untreated sample to the PTMR for the same sample after treatment. *(2)* **Analyzer Qualification**—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 *(3)* **Local Validation**—A local validation is performed using an independent but limited set of materials that were not part of the correlation activity. This local validation is intended to demonstrate that the agreement between the Predicted Primary Method Test Results (PPTMRs) and the PTMRs are consistent with expectations based on the multivariate model. *(4)* **General Validation**—After an adequate number of PPTMRs and PTMRs have been accrued on materials that were not part of the correlation activity and which adequately span the multivariate model compositional space, a comprehensive statistical assessment can be performed to demonstrate that the PPTMRs agree with the PTMRs to within user-specified requirements. *(5)* **Continual Validation**—Subsequent to a successful local or 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, fourth, and fifth of these activities.

"Correlation where analyzer measures a material which is subjected to treatment before being measured by the PTM" as outlined in this practice can be applied to biofuels where the biofuel material is added at a terminal or other facility and not included in the process stream material sampled by the analyzer at the basestock manufacturing facility. The "treatment" shall be a constant percentage addition of the biofuels material to the basestock material. The correlation is deemed valid only for the specific percentage addition and type of biofuel material used in its development.

1. Scope*

1.1 This practice covers requirements for the validation of measurements made by laboratory, field, or process (online or at-line) infrared (near- or mid-infrared analyzers, or both), and Raman analyzers, used in the calculation of physical, chemical, or quality parameters (that is, properties) of liquid petroleum products and fuels. 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 that the uncertainties associated with the degree of agreement between

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

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