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An American National Standard

Standard Practice for Validation of Multivariate Process Infrared Spectrophotometers[1]

This standard is issued under the fixed designation D 6122; 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 practice covers requirements for the validation of measurements made by on-line, process near- or mid-infrared analyzers, or both, used in the calculation of physical, chemical, or quality parameters of liquid petroleum products. The parameters 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 method used for the development of the calibration model.

1.2 This practice does not cover procedures for establishing the calibration model used by the analyzer. Calibration procedures are covered in Practices E 1655 and references therein.

1.3 This practice is intended as a review for experienced persons. For novices, this practice will serve as an overview of techniques used to verify instrument performance, to verify model applicability to the spectrum of the sample under test, and to verify equivalence between the parameters calculated from the infrared measurement and the results of the primary method measurement.

1.4 This practice teaches and recommends appropriate statistical tools, outlier detection methods, for determining whether the spectrum of the sample under test is a member of the population of spectra used for the analyzer calibration. The statistical tools are used to determine if the infrared measurement results in a valid property or parameter estimate.

1.5 The outlier detection methods do not define criteria to determine whether the sample, or the instrument is the cause of an outlier measurement. Thus, the operator who is measuring samples on a routine basis will find criteria to determine that a spectral measurement lies outside the calibration, but will not have specific information on the cause of the outlier. This practice does suggest methods by which instrument performance tests can be used to indicate if the outlier methods are responding to changes in the instrument response.

1.6 This practice is not intended as a quantitative performance standard for the comparison of analyzers of different design.

1.7 Although this practice deals primarily with validation of on-line, process infrared analyzers, the procedures and statistical tests described herein are also applicable to at-line and laboratory infrared analyzers which employ multivariate models.

1.8 *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 consult and 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 3764 Practice for Validation of Process Stream Analyzers[3]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[3]

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance[4]

E 131 Terminology Relating to Molecular Spectroscopy[5]

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near-Infrared Spectrophotometers[5]

E 456 Terminology Relating to Quality and Statistics[6]

E 932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrophotometers[5]

E 1421 Practice for Describing and Measuring Performance

[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 for Petroleum and Petroleum Products.

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[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 03.06.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

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