



Standard Practice for Validation of Empirically Derived Multivariate Calibrations¹

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1. Scope

1.1 This practice covers requirements for the validation of empirically derived calibrations (**Note 1**) such as calibrations derived by Multiple Linear Regression (MLR), Principal Component Regression (PCR), Partial Least Squares (PLS), Artificial Neural Networks (ANN), or any other empirical calibration technique whereby a relationship is postulated between a set of variables measured for a given sample under test and one or more physical, chemical, quality, or membership properties applicable to that sample.

NOTE 1—Empirically derived calibrations are sometimes referred to as “models” or “calibrations.” In the following text, for conciseness, the term “calibration” may be used instead of the full name of the procedure.

1.2 This practice does not cover procedures for establishing said postulated relationship.

1.3 This practice serves as an overview of techniques used to verify the applicability of an empirically derived multivariate calibration to the measurement of a sample under test and to verify equivalence between the properties calculated from the empirically derived multivariate calibration and the results of an accepted reference method of measurement to within control limits established for the prespecified statistical confidence level.

1.4 *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

E131 Terminology Relating to Molecular Spectroscopy

E1655 Practices for Infrared Multivariate Quantitative Analysis

¹ This practice is under the jurisdiction of ASTM Committee **E13** on Molecular Spectroscopy and Separation Science and is the direct responsibility of Subcommittee **E13.11** on Multivariate Analysis.

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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.

E1790 Practice for Near Infrared Qualitative Analysis

3. Terminology

3.1 For terminology related to molecular spectroscopic methods, refer to Terminology **E131**. For terminology related to multivariate quantitative modeling refer to Practices **E1655**. While Practices **E1655** is written in the context of multivariate spectroscopic methods, the terminology is also applicable to other multivariate technologies.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *accuracy*—the closeness of agreement between a test result and an accepted reference value.

3.2.2 *bias*—the arithmetic average difference between the reference values and the values produced by the analytical method under test, for a set of samples.

3.2.3 *detection limit*—the lowest level of a property in a sample that can be detected, but not necessarily quantified, by the measurement system.

3.2.4 *estimate*—the constituent concentration, identification, or other property of a sample as determined by the analytical method being validated.

3.2.5 *initial validation*—validation that is performed when an analyzer system is initially installed or after major maintenance.

3.2.6 *Negative Fraction Identified*—the fraction of samples not having a particular characteristic that is identified as not having that characteristic.

3.2.6.1 *Discussion*—Negative Fraction Identified assumes that the characteristic that the test measures either is or is not present. It is not applicable to tests with multiple possible outcomes.

3.2.7 *ongoing periodic revalidation*—the quality assurance process by which, in the case of quantitative calibrations, the bias and precision or, in the case of qualitative calibrations, the Positive Fraction Identified and Negative Fraction Identified performance determined during initial validation are shown to be sustained.

3.2.8 *Positive Fraction Identified*—the fraction of samples having a particular characteristic that is identified as having that characteristic.

3.2.8.1 *Discussion*—Positive Fraction Identified assumes