



# Standard Practice for Qualifying Spectrometers and Spectrophotometers for Use in Multivariate Analyses, Calibrated Using Surrogate Mixtures<sup>1</sup>

This standard is issued under the fixed designation E 2056; 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 relates to the multivariate calibration of spectrometers and spectrophotometers used in determining the physical and chemical characteristics of materials. A detailed description of general multivariate analysis is given in Practice E 1655. This standard refers only to those instances where surrogate mixtures can be used to establish a suitable calibration matrix. This practice specifies calibration and qualification data set requirements for interlaboratory studies (ILSs), that is, round robins, of standard test methods employing surrogate calibration techniques that do not conform exactly to Practices E 1655.

NOTE 1—For some multivariate spectroscopic analyses, interferences and matrix effects are sufficiently small that it is possible to calibrate using mixtures that contain substantially fewer chemical components than the samples that will ultimately be analyzed. While these surrogate methods generally make use of the multivariate mathematics described in Practices E 1655, they do not conform to procedures described therein, specifically with respect to the handling of outliers.

1.2 This practice specifies how the ILS data is treated to establish spectrometer/spectrophotometer performance qualification requirements to be incorporated into standard test methods.

NOTE 2—Spectrometer/spectrophotometer qualification procedures are intended to allow the user to determine if the performance of a specific spectrometer/spectrophotometer is adequate to conduct the analysis so as to obtain results consistent with the published test method precision.

1.2.1 The spectroscopies used in the surrogate test methods would include but not be limited to mid- and near-infrared, ultraviolet/visible, fluorescence and Raman spectroscopies.

1.2.2 The surrogate calibrations covered in this practice are: multilinear regression (MLR), principal components regression (PCR) or partial least squares (PLS) mathematics. These calibration procedures are described in detail in Practices E 1655.

1.3 For surrogate test methods, this practice recommends limitations that should be placed on calibration options that are

allowed in the test method. Specifically, this practice recommends that the test method developer demonstrate that all calibrations that are allowed in the test method produce statistically indistinguishable results.

1.4 For surrogate test methods that reference spectrometer/spectrophotometer performance practices, such as Practices E 275, E 387, E 388, E 579, E 925, E 932, E 958, E 1421, E 1683, E 1866 or E 1944, this practice recommends that instrument performance data be collected as part of the ILS to establish the relationship between spectrometer/spectrophotometer performance and test method precision.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- D 6277 Test Method for Determination of Benzene in Spark-Ignition Engine Fuels by Mid Infrared Spectroscopy<sup>2</sup>
- D 6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants<sup>3</sup>
- E 131 Terminology Relating to Molecular Spectroscopy<sup>3</sup>
- E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers<sup>3</sup>
- E 387 Test Method for Estimating Stray Radiant Power of Spectrophotometers by the Opaque Filter Method<sup>3</sup>
- E 388 Test Method for Spectral Bandwidth and Wavelength Accuracy of Fluorescence Spectrophotometers<sup>3</sup>
- E 579 Test Method for Limit of Detection of Fluorescence of Quinine Sulfate<sup>3</sup>
- E 691 Practice for Conducting an Inter-Laboratory Study to Determine the Precision of a Test Method<sup>4</sup>
- E 925 Practice for the Periodic Calibration of Narrow Band-Pass Spectrophotometers<sup>3</sup>
- E 932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrometers<sup>3</sup>
- E 958 Practice for Measuring Practical Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers<sup>3</sup>
- E 1421 Practice for Describing and Measuring Performance

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and is the direct responsibility of Subcommittee E13.11 on Chemometrics.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 05.03.

<sup>3</sup> Annual Book of ASTM Standards, Vol 03.06.

<sup>4</sup> Annual Book of ASTM Standards, Vol 14.02.

