



Standard Practice for Establishing and Controlling Spectrochemical Analytical Curves¹

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^{ε1} NOTE—Section 9 was added editorially in January 1995.

1. Scope

1.1 This practice provides guidance for establishing and controlling spectrochemical analytical curves. The preparation of analytical curves and their routine control are considered as separate although interrelated operations. This practice is applicable to optical emission spectrographs, optical emission spectrometers, or X-ray emission spectrometers with linear or logarithmic readouts.

1.2 *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:

- E 116 Practice for Photographic Photometry in Spectrochemical Analysis²
- E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials²
- E 178 Practice for Dealing with Outlying Observations³
- E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data⁴

3. Terminology

3.1 For definitions of terms used in this practice, refer to Terminology E 135.

4. Summary of Practice

4.1 Systematic and random errors that occur in obtaining data are reviewed. Background corrections are considered for linear and logarithmic readout systems. Calibration procedures

are given, including the reference materials to be used and the generation of data. Procedures are provided for constructing the analytical curve, fitting a regression curve and evaluating curve fit. Control of curve shift and rotation is described.

5. Significance and Use

5.1 This practice is intended as a fundamental guide for the calibration, standardization, and daily control of the analytical curves on optical emission spectrographs and spectrometers, and X-ray spectrometers.

6. Precautions

6.1 Systematic Errors:

6.1.1 *Systematic Errors Due to Incorrect Calibration*—In the procedure for quantitative spectrochemical analysis, the initial construction of the analytical curve relates element concentration and spectral intensity or intensity ratio. The accuracy of the calibration may be affected by a number of factors, such as incorrect element concentrations, heterogeneity of the reference materials, spectral interferences, and matrix effects. Such errors may cause a rotation or bodily displacement of the analytical curve, thereby leading to systematic errors in the analytical data generated.

6.1.1.1 Calibration errors due to incorrect element concentrations may be minimized by the use of certified reference materials. When these are used, one or more other reference materials for which the chemical compositions have been carefully determined by approved methods of analysis, such as ASTM or BSI (British Standards Institute), may be included to detect whether systematic errors exist because of differences in the metallurgical condition of the certified reference materials and the specimens. In the absence of certified reference materials, it is helpful to use several reference materials from a variety of sources to detect systematic differences in these materials. In general, the use of a large number of reference materials will aid in the detection and rejection of those which are obviously inaccurate. In cases where it is necessary to synthesize reference materials, the synthesis of each one

¹ This practice is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.20 on Fundamental Practices and Measurement Traceability.

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² Annual Book of ASTM Standards, Vol 03.05.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Annual Book of ASTM Standards, Vol 03.06.

