



Standard Practice for Testing Homogeneity of Materials for Development of Reference Materials¹

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^{ε1} NOTE—Section 12 was added editorially in June 1996.

1. Scope

1.1 This practice is suitable for testing the homogeneity of metals, either in solid or powdered form, and finely ground oxide materials that are intended for use as reference materials in X-ray emission, or optical emission spectroscopy, or both. The criteria for acceptance of the test specimens as reference materials, however, must be previously determined by the user for meeting his specific requirements.

1.2 The procedure is designed primarily for testing specimens by X-ray emission spectrometry or optical emission spectroscopy, or both. However, the practice could be easily adapted for use with other instrumental techniques such as atomic absorption spectrophotometry.

1.3 This procedure can be applied to one or more elements in a specimen provided the signal-to-background ratio is not a limiting factor.

1.4 This practice includes one method, if desired, to correct for systematic or periodic (sinusoidal) drift in the instrument with time through the use of a control reference material.

NOTE 1—**Caution:** If serious drift occurs (for example, unstable power supply, X-ray tube, etc.) erroneous conclusions may be obtained from the data analysis.

1.5 *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 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials²

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods³

E 178 Practice for Dealing with Outlying Observations³

¹ This practice is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.22 on Statistics and Quality Control.

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² Annual Book of ASTM Standards, Vol 03.05.

³ Annual Book of ASTM Standards, Vol 14.02.

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data⁴

3. Terminology

3.1 **Definitions**—For definitions of terms used in this practice, refer to Terminology E 135, and Practices E 177 and E 178.

3.2 **Definitions of Terms Specific to This Standard:**

3.2.1 **drift**—a gradual systematic or sinusoidal change in instrument readings with time.

3.2.2 **homogeneity**—as defined in this practice, is statistically acceptable differences among means of specimens in the test.

4. Summary of Practice

4.1 This procedure, which is based on statistical methods (1-6),⁵ consists of stepwise instructions for testing homogeneity of candidate reference materials. The candidate materials are selected as described in Section 6, and then measured by either X-ray emission or optical emission spectroscopy (see Sections 7 and 8). The resultant data are corrected for instrumental drift, if desired (see Section 10), and then tabulated (see Table 1, Table X1.3, and Table X1.4) to facilitate the statistical calculations that are performed according to Section 9. The homogeneity of the material is determined from the results of the data analysis.

4.2 This procedure *requires* that repeated measurements on the same specimen have sufficient precision (that is, repeatability) through appropriate selection of instrumental parameters so that any significant difference among specimens can be detected with confidence.

4.3 This procedure requires that there be an absence of outliers in the data (see Practice E 178).

NOTE 2—**Caution:** The use of Practice E 178 dealing with outliers should be done with extreme care to ensure that values are not discarded that may be valid for the analysis.

5. Significance and Use

5.1 The purpose of this practice is to ensure the quality of

⁴ Annual Book of ASTM Standards, Vol 03.06.

⁵ The boldface numerals in parentheses refer to the list of references at the end of this practice.