



Standard Guide for X-Ray Emission Spectrometric Analysis¹

This standard is issued under the fixed designation E 1621; 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 guide covers guidelines for developing and describing analytical procedures using a wavelength-dispersive X-ray spectrometer.

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

E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves³

E 1060 Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis³

E 1257 Guide for Evaluating Grinding Materials Used for Surface Preparation in Spectrochemical Analysis³

E 1329 Practice for Verification and the Use of Control Charts in Spectrochemical Analysis³

E 1361 Guide for Correction of Interelement Effects in X-ray Spectrometric Analysis³

E 1622 Practice for Correction of Spectral Line Overlap in Wavelength-Dispersive X-Ray Spectrometry³

3. Terminology

3.1 **Definitions**—For definitions of terms used in this guide, refer to Terminologies E 135 and E 1361, Section.

4. Summary of Guide

4.1 The test specimen is prepared with a clean, uniform, flat surface. It may be prepared by grinding, polishing, or lathing a metal surface or by fusing or briquetting a powder. This surface is irradiated with a primary source of X rays. The secondary X rays produced in the specimen are dispersed according to their wavelength by means of crystals or synthetic multilayers. Their

intensities are measured by suitable detectors at selected wavelengths and converted to counts by the detector. X-ray measurements are made based on the time required to reach a fixed number of counts, or on the total counts obtained for a fixed time. Concentrations of the elements are determined from the measured intensities of analyte X-ray lines using analytical curves prepared with suitable reference materials. Either a fixed multi-channel simultaneous system or a sequential monochromator system may be used to provide determinations of the elements.

5. Significance and Use

5.1 X-ray fluorescence spectrometry can provide an accurate and precise determination of metallic and many non-metallic elements. This guide covers the information which should be included in an X-ray spectrometric analytical method and provides direction to the analyst for determining the optimum conditions needed to achieve acceptable accuracy.

5.2 The accuracy of an analysis is a function of the calibration scheme, the sample preparation, and the sample homogeneity. Close attention to all aspects of these areas is necessary to achieve the best results.

6. Interferences

6.1 Line overlaps, either total or partial, may occur for some elements. Fundamental parameter equations require that the net intensities be free from line overlap effects. Some empirical schemes incorporate line overlap corrections in their equations. See Guide E 1622 for correction of line overlap effects.

6.2 Interelement effects or matrix effects may exist for some elements. An empirical way to compensate for these effects is to prepare a series of calibration curves which cover the designated concentration ranges to be analyzed. A large suite of carefully designed reference materials is necessary for this procedure. A series of samples in which all elements are relatively constant, except for the analyte, is necessary for each analyte which can be affected by other elements in the matrix. In addition, several series for the same analyte may be necessary, if the analyte is subject to large effects from some other element in the matrix. The composition of the specimen being analyzed must match closely the composition of the reference materials used to prepare the particular calibration curves.

6.2.1 Alternatively, mathematical methods may be used to

¹ This guide 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 E01.20 on Fundamental Practices.

Current edition approved July 15, 1994. Published September 1994.

² *Annual Book of ASTM Standards*, Vol 03.05.

³ *Annual Book of ASTM Standards*, Vol 03.06.