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# Standard Guide for X-Ray Emission Spectrometric Analysis[1]

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## 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 Materials[2]

E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves[3]

E 1060 Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis[3]

E 1257 Guide for Evaluating Grinding Materials Used for Surface Preparation in Spectrochemical Analysis[3]

E 1329 Practice for Verification and the Use of Control Charts in Spectrochemical Analysis[3]

E 1361 Guide for Correction of Interelement Effects in X-ray Spectrometric Analysis[3]

E 1622 Practice for Correction of Spectral Line Overlap in Wavelength-Dispersive X-Ray Spectrometry[3]

## 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 nonmetallic 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

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

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

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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 compensate for interelement or matrix effects. Various mathematical correction procedures are commonly utilized. See Guide E 1361. Any of these that will achieve the necessary analytical accuracy is acceptable.

NOTE 1—Interelement effects are not interferences in the spectrometric sense, but will contribute to errors in the analysis if not properly addressed. Caution must be used with mathematical models used in computers to be sure that sufficient data is provided to adequately compensate for these effects. Reference materials which were not used in the calibration should be analyzed as unknowns to verify the calibration.

6.3 Additionally, interferences may occur from Compton lines or characteristic lines generated by the target material of the X-ray tube. These may be reduced or eliminated by the use of primary beam filters, but this will cause some loss of analyte line intensity.

6.4 *Errors From Metallurgical Structure*—Because the analyte intensity is affected by the mass absorption coefficient of the sample and mathematical models assume a homogeneous sample, an error may result if the analyte exists in an inclusion. For example, in a steel which contains carbon and carbide formers such as titanium and niobium, the titanium may exist in a titanium-niobium carbide which has a lower mass absorption coefficient than iron for the titanium K-α line. The intensity for titanium is higher in this sample than it would be if the titanium were in solid solution.

## 7. Apparatus

7.1 *Specimen Preparation Equipment for Metals*:

7.1.1 *Surface Grinder or Sander With Abrasive Belts or Disks, or Lathe*, capable of providing a flat, uniform surface on both the reference materials and test specimens.

7.1.1.1 Abrasive disks are preferred over belts because the platen on a belt sander tends to wear and produce a non-flat surface on the specimen. If belt sanders are used, care must be exercised to be sure the platen is flat.

7.1.1.2 The grinding material should be selected so that no significant contamination occurs for the elements of interest during the sample preparation. (Refer to Guide E 1257.)

7.1.1.3 Grinding belts or disks shall be changed at regular, specified intervals in order that changes in abrasive grit due to repeated use does not affect the repeatability of the roughness of the sample finish. This is particularly important in alloys which exhibit smearing of a softer component over the sample matrix.

7.2 *Specimen Preparation Equipment for Powders*:

7.2.1 *Jaw Crusher or Steel Mortar and Pestle*, for initial crushing of lumps.

7.2.2 *Plate Grinder or Pulverizer*, with one static and one rotating disk for further grinding.

7.2.3 *Rotary Disk Mill or Shatterbox*, with hardened grinding containers and timer control for final grinding.

7.2.4 *Briquetting Press*, providing pressures of up to 550 MPa (80 000 psi). The press shall be equipped with a mold assembly that provides a briquette that is compatible with the X-ray specimen holder.

7.2.5 *Fusion Equipment*, with a timer, capable of heating the sample and flux to at least 1000°C and homogenizing the melt.

7.2.6 *Fusion Crucibles*, compatible with the flux and sample type:

7.2.6.1 *Vitreous Carbon*, 20 to 30-mL capacity, with flat bottom 30 to 35 mm in diameter.

7.2.6.2 *95 % Platinum/5 % Gold Alloy*, with 30 to 35-mL capacity.

7.2.7 *Platinum/Gold Casting Mold (95 %/5 %)*, 30 to 35-mL capacity, with flat bottom 30 to 40 mm in diameter.

7.2.8 *Polishing Wheel*, suitable for polishing the fused button to obtain a flat uniform surface for irradiation.

7.3 *Excitation Source*:

7.3.1 *X-Ray Tube Power Supply*, providing a stable voltage of sufficient energy to produce secondary radiation from the specimen for the elements specified.

7.3.1.1 The instrument may be equipped with an external line voltage regulator or a transient voltage suppressor.

7.3.2 *X-Ray Tubes*, with targets of various high-purity elements, that are capable of continuous operation at potentials and currents that will excite the elements to be determined.

7.4 *Spectrometer*, designed for X-ray emission analysis, and equipped with specimen holders and a specimen chamber. The chamber may contain a specimen spinner, and must be equipped for vacuum or helium-flushed operation for the determination of elements of atomic number 20 (calcium) or lower.

7.4.1 *Analyzing Crystals*, flat or curved crystals with optimized capability for the diffraction of the wavelengths of interest. This may also include synthetic multi-layers for low atomic number elements.

7.4.2 *Collimator*, for limiting the characteristic X rays to a parallel bundle when flat crystals are used in the instrument. For curved crystal optics, a collimator is not necessary, but is replaced by entrance and exit slits.

7.4.3 *Masks*, for restricting the incident beam pattern on the specimen.

7.4.4 *Detectors*—sealed or gas-flow proportional counters and scintillation counters are most commonly used.

7.4.5 *Vacuum System*, for the determination of elements whose radiation is absorbed by air. The system shall consist of a vacuum pump, gage, and electrical controls to provide automatic pumpdown of the optical path, and maintain a controlled pressure, usually 13 Pa (100 µm Hg) or less, controlled to ± 3 Pa (± 20 µm Hg).

7.5 *Measuring System*, consisting of electronic circuits capable of amplifying and shaping pulses received from the detectors. The system shall be equipped with an appropriate data output device.

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