



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.

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 μ m Hg) or less, controlled to ± 3 Pa (± 20 μ m 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.

7.5.1 *Pulse Height Selectors*, used to reduce pulses from higher order X-ray lines and background.

8. Reagents and Materials

8.1 *Purity of Reagents*—All reagents used in this test method shall conform to the “Reagent Grade” specifications of the American Chemical Society⁴. Other chemicals may be used

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

provided it is first ascertained that they are of sufficient purity to permit their use without adversely affecting the expected performance of the analysis.

8.2 *Binders*—Sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7$), polyethylene glycol, fibrous cellulose, or spectrographic grade graphite (–200 mesh, briquetting type).

8.3 *Detector Gas (P-10)*, consisting of a mixture of 90 % argon and 10 % methane, for use with gas-flow proportional counters.

8.4 *Fluxes*—Sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7$), fused and dried; lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$), or lithium tetraborate and boric anhydrite (B_2O_3) mixture (4 g + 6 g).

9. Reference Materials

9.1 *Certified Reference Materials* are available from the National Institute of Standards and Technology⁵ and other certification agencies.

9.2 *Reference Materials* with compositions similar to that of the test specimen and containing varying amounts of the elements to be determined may be used provided they have been previously analyzed in accordance with ASTM test methods. These reference materials shall be homogeneous, and free of voids or porosity.

9.3 The reference materials should cover the concentration ranges of the elements being determined. An appropriate number of reference materials shall be used for each element, depending on the mathematical models being used.

10. Hazards

10.1 Occupational Health and Safety Standards for ionizing radiation⁶ shall be observed at all X-ray emission spectrometer installations. Operating and maintenance personnel shall follow the guidelines of safe operating procedures given in current handbooks and publications from the National Institute of Standards and Technology,^{7,8} the U.S. Government Printing Office,⁹ or similar handbooks on radiation safety, as well as specific state regulations.

10.2 *Monitoring Devices*, either film badges or dosimeters¹⁰ may be worn by all operating and maintenance personnel. Safety practices shall conform to applicable local, state, and federal regulations. To meet local, state, and federal radiation standards, periodic radiation surveys of the equipment for leaks

and excessive scattered radiation shall be made by a qualified person using an ionization-chamber detector.¹¹ The personal film badge survey record, the radiation survey record, and an equipment maintenance record shall be available upon request.

10.3 Special precautions for operators and maintenance personnel shall be posted at the equipment site.

10.4 *Radiation Caution Signs* shall be posted near the X-ray equipment and at all entrances to the radiation area.

10.5 *Fail-Safe “X-Ray On” Warning Lights* shall be used on the equipment.

10.6 Routine checks of safety interlocks shall be documented.

11. Preparation of Reference Materials and Test Specimens

11.1 Throughout the procedure, treat reference materials and test specimens exactly the same way. Consistency in preparation of reference materials and specimens is essential to ensure reproducible results. After the preparation procedure is established, it must be followed exactly. Variations in technique, such as grinding time, abrasive grit size or material, particle size, binder material, sample-binder ratio, briquetting pressure, or holding times, can cause unreliable results.

11.2 *Metal Samples*—Prepare the reference materials and test specimens to provide a clean, flat uniform surface to be exposed to the X-ray beam. For abrasive sanding, select a grit size and use it exclusively for all reference materials and test specimens. See 7.1.1.2 and 7.1.1.3. Refinish the surface of the reference materials and test specimens as needed to eliminate oxidation before measurement.

11.3 *Nonmetallic Samples*—Dry the material. Then reduce it both in particle size and quantity, by crushing and pulverizing integrated with splitting or riffing, ending up with approximately 100 g of material that has a particle size of less than 200 mesh (74 μm).

11.3.1 *Briquettes*—Mix the sample with a suitable binder. (See 8.2.) Ratios of 10 g + 1 g to 20 g + 1 g of sample + binder are common. Grind and blend the sample and binder for a fixed time (generally 2 to 4 min in a disk mill). Press the sample-binder mixture into a briquette using a fixed pressure of 140 to 550 MPa (20 000 to 80 000 psi) and maintaining the pressure for a minimum of 10 s before releasing the briquette. Holding the pressure at 140 MPa (20 000 psi) for about 10 s before increasing it to maximum allows air to escape from the mixture and reduces the possibility of the briquette bursting from internal pressure.

NOTE 2—For some samples, an aluminum cup may be required to support the briquette.

11.3.2 *Fused Beads*—Use a predetermined mix of sample to flux combination. A 0.3 g of sample to 5.0 g of sodium tetraborate or 1.0 g of sample to 4.0 to 10.0 g of lithium tetraborate are commonly used mixtures. Mix weighed amounts of sample and flux and place the mixture in clean platinum/bold or vitreous carbon crucible. Heat at a fixed

⁵ Available from Standard Reference Materials Program, National Institute of Standards and Technology, U.S. Department of Commerce, Gaithersburg, MD 20899.

⁶ ANSI/NBS Handbook 114, General Safety Standard for Installations Using Non-Medical X-Ray and Sealed Gamma-Ray Sources, available from American National Standards Institute, 11 W. 42nd Street, 13th Floor, New York, NY 10036.

⁷ NBS Handbook 76, *Medical X-ray Protection Up to Three Million Volts*, available as NCRP 33 from NCRP Publications, 7910 Woodmont Ave., Suite 1016, Bethesda, MD.

⁸ ANSI N43.2-1977/NBS Handbook 111, Radiation Safety for X-ray Diffraction and Fluorescence Analysis, available from American National Standards Institute, 11 W. 42nd Street, 13th Floor, New York, NY 10036.

⁹ Moore, T. M. and McDonald, D. J., *Radiation Safety Recommendations for X-ray Diffraction and Spectrographic Equipment*, MORP 68-14, 1968, available from National Technical Information Service, Springfield, VA 22161.

¹⁰ Radiation Film Badge Service, available from R. S. Landauer, Jr. and Co., Glenwood Science Park, Glenwood, IL 60425, and Siemens Gammasonics, Inc., Health Physics Services, 2000 Nuclear Drive, Des Plaines, IL 60018, has been found satisfactory.

¹¹ A survey meter that meets the requirements is available from Siemens Gammasonics, Inc., Health Physics Services, 2000 Nuclear Drive, Des Plaines, IL 60018 and Nuclear Associates, Wesbury, L.I., NY 11590.

temperature, usually from 950 to 1100°C, until thoroughly melted. Swirl the crucible several times to ensure a homogeneous fusion and to remove particles from the crucible walls. Fusion time may vary from 2 to 10 min, depending on the sample, flux, and sample to flux ratio.

11.3.2.1 When using platinum/gold crucibles, cast the fused mixture in a preheated (800°C) platinum/gold mold, and allow to solidify and cool in the mold. Remove the bead. It may be beneficial to polish the bead lightly on a 220-grit diamond wheel or equivalent polishing wheel to provide a clean flat surface for analysis.

NOTE 3—For some applications, analysis of the as-cast surface of the bead may be adequate. Each laboratory must determine if polishing is essential for its application. Also, fusion made in a carbon crucible may form a spherically shaped bead upon cooling. It has been reported that a flat bead of acceptable size may be obtained by adding wire ring conforming to inside diameter of the crucible to the melt before it solidifies.

NOTE 4—The addition of a small amount (100 mg) of a halide such as HBr, LiBr, or LiF, may act as release agent when using platinum/gold molds.

12. Preparation of Apparatus

12.1 Prepare and operate the spectrometer in accordance with the manufacturer's instructions, using the specific parameters given for the method.

NOTE 5—It is not within the scope of a method to prescribe details relative to the preparation of the apparatus. For a description and specific details concerning a particular spectrometer, refer to the manufacturer's manual.

12.1.1 *Start-Up*—Follow the manufacturer's instructions for proper warm-up procedures.

12.2 *Tube Power Supply*—Adjust the excitation voltage of the power supply to excite the desired analyte lines. The following equation may be used as a guide:

$$E = 12.4/\lambda_{\text{abs}} \quad (1)$$

where:

E = minimum voltage, keV, required for exciting the line of interest, and

λ_{abs} = wavelength, Å, of the absorption edge of the element of interest.

If a K line is measured, the K absorption edge is used. If an L line is measured, the L absorption edge of highest energy is used, generally the L(III) edge. Ideally, the operating voltage should approximate or exceed 3E.

12.2.1 The X-ray tube voltage and current established as optimum for the method shall be reproduced for subsequent measurements.

12.2.1.1 *X-ray Tube Output Intensity*—The intensity of the continuum is proportional to the current and to the square of the voltage. The intensity of the characteristic line spectrum of the target material is proportional to the current and to the over voltage raised to the 1.7 power. The over voltage (V_o) is the difference between the tube voltage (V_E) and the excitation potential (V_{EP}) of the target material:

$$V_o = V_E - V_{EP} \quad (2)$$

12.2.1.2 *Analyte Line Intensity*—The analyte line intensity is proportional to the tube current and to the voltage raised to

the 1.7 power, assuming that the voltage exceeds the minimum excitation voltage (E).

12.3 *Spectrometer Conditions*—List the analyte, analytical line, crystal, detector, collimation, background location (if determined), and goniometer position (2θ) (for a scanning spectrometer). In general, background measurements need not be made if the peak to background ratio is greater than 10:1, unless background-corrected intensities are required for fundamental parameter calculations. Theoretical 2θ positions should be given since the actual position can vary with individual spectrometers.

12.4 *Proportional Counter Gas Flow*—When a gas-flow proportional counter is used, adjust the flow of the P-10 gas in accordance with the equipment manufacturer's instructions. The detectors should be adequately flushed with detector gas before the instrument is used. Pulse height selector conditions should be checked following a change in P-10 cylinders.

13. Calibration and Standardization

13.1 *Calibration (Preparation of Analytical Curves)*—Using the conditions given in Section 12, measure a series of calibrants that cover the required concentration ranges. Standardants and verifiers (see 13.2) should be run at the same time as the calibrants. Prepare an analytical curve for each element being determined in each alloy type or material. Plot, on the abscissa, the analyte intensities, in terms of counts, counts per second, or relative intensities, (relative intensity is the ratio of the intensity of the analyte in the sample to the intensity obtained for the pure element) versus the corresponding concentrations expressed in percent or parts per million of the elements in the calibrants on the ordinate (refer to Practice E 305). Alternatively, plot the ratios of the intensities of the calibrants to the intensities of one of the calibrants, an internal standard line, or the measured background, as a function of concentration.

13.1.1 Corrections for background (where required), line overlaps, and interelement effects must be properly incorporated into the calibration scheme and selection of calibrants or a bias will be introduced into the calculation of the final results.

13.1.2 To verify the calibration, run the calibrants as unknowns. When nonlinear calibration schemes are used, reference materials other than those used to calibrate should be used to verify the calibration.

13.2 *Verification and Standardization*—Measure the verifiers to see if the readings are within the allowable limits established by a control chart. See Practice E 1329. If necessary, use standardants to make appropriate adjustments according to the instructions in the manufacturer's manual. Refer to Practice E 305 for frequency of verification.

13.2.1 Always measure the verifiers if the detector gas or a major component of the spectrometer has been changed. Recalibrate if required.

14. Procedure

14.1 *Specimen Loading*—If the spectrometer does not have a sample spinner, orient the reference materials and test specimens in the specimen chamber so that the relationship between the X-ray beam and the grinding striations is the same for all measurements. This is essential if the spectrometer is not

equipped with a specimen spinner, but is not necessary when a spinner is used.

14.2 Excitation—Expose the specimen to primary X radiation in accordance with Section 12.

14.3 Radiation Measurements—Obtain and record the counting rate measurement for each element. Either fixed count or fixed time modes may be used. Obtain enough counts so that the statistical error due to counting is acceptable.

NOTE 6—Assuming measurements fit Poisson statistics, where N is the total number of counts, $N^{1/2}$ is a good measure of the statistical counting error, provided N is greater than 100. For example, if 0.1 % counting error is acceptable, 10^6 counts, with $N^{1/2} = 10^3$, yielding a relative percent error of $100 \times 10^3/10^6$ or 0.1 %. For a counting error of 0.5 %, the total counts required drops to 40 000 counts. The time required for measurement is the total counts required divided by the intensity. For an intensity of 1600 counts per second, to obtain a 0.5 % error, a count time of 25 s is required. To obtain 0.1 % error, a count time of 625 s is required. These are ideal precisions that can be attained only if other instrumental errors are negligible.

14.4 Spectral Interferences—X-ray spectrometers may not completely resolve X-ray line pairs for some line combinations (for example, manganese K- α and chromium K- β ; vanadium K- α and titanium K- β ; and phosphorus K- α and molybdenum L1). Therefore, care must be exercised in the interpretation of X-ray line intensities when both elements are present. In some cases, alternative X-ray lines without interference can be used. Otherwise, mathematical calculations must be used to correct for the interferences. See Guide E 1622.

14.5 Replicate Measurements—Make a single measurement on each test specimen. Assuming that the conditions in 14.3 are met, the accuracy of an X-ray spectrometric analysis is not improved significantly by making multiple measurements on the same surface of the specimen. Confidence in the accuracy of the analysis of the material will improve by making multiple measurements on freshly prepared surfaces of the same specimen or by analyzing more than one specimen. Precision can be increased by increasing the counting time. See Note 6.

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15. Calculation of Results

15.1 Using the intensities from the test specimen and the appropriate analytical curves, determine the concentration of the various elements.

15.1.1 The intensities may be entered directly into a computer that is programmed with mathematical algorithms to convert the data directly into concentrations. These algorithms must provide corrections for any spectral interferences and matrix effects. See Section 6 and 14.4.

15.1.2 If mathematical calculations must be made for inter-element effects, refer to the equipment manufacturer's manual for the recommended procedure for the instrument being used. Computers are normally interfaced to the X-ray spectrometers employing such correction schemes. See also Guide E 1361.

NOTE 7—For systems without computers, concentration may be determined using graphs or tables, but care must be exercised to be sure that errors from line overlap and matrix effects do not occur. The tables or graphs must be made from reference materials that closely match the specimens being analyzed.

16. Precision and Bias

16.1 Precision—An acceptable number of laboratories should test the method in accordance with an acceptable interlaboratory test such as Practice E 1060. Precision data, including repeatability (precision within laboratories), R_1 , and reproducibility (precision between laboratories), R_2 , should be provided in table(s).

16.2 Bias and Accuracy—If acceptable true concentrations are known for the samples used in the interlaboratory test, calculate accuracy in accordance with Practice E 1060 and show the results in a table.

16.2.1 Unless the accuracy data shows a systematic deviation from acceptable true concentrations, a statement should be made that the test method shows no significant bias.

17. Keywords

17.1 spectroscopy; X-ray