Standard Practice for
Describing and Specifying a Wavelength Dispersive X-Ray Spectrometer[1]

This standard is issued under the fixed designation E1172; 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 practice covers the components of a wavelength dispersive X-ray spectrometer that are basic to its operation and to the quality of its performance. It is not the intent of this practice to specify component tolerances or performance criteria, as these are unique for each instrument. However, the practice does attempt to identify which tolerances are critical and thus which should be specified.

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 to determine the applicability of regulatory limitations prior to use.* Specific safety hazard statements are given in 5.3.1.2 and 5.3.2.4, and in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E2857 Guide for Validating Analytical Methods

3. Terminology

3.1 For terminology relating to X-ray spectrometry, refer to Terminology E135.

4. Significance and Use

4.1 This practice describes the essential components of a wavelength dispersive X-ray spectrometer. This description is presented so that the user or potential user may gain a cursory understanding of the structure of an X-ray spectrometer system. It also provides a means for comparing and evaluating different systems as well as understanding the capabilities and limitations of each instrument.

4.2 It is understood that a laboratory may implement this practice or an X-ray fluorescence method in partnership with a manufacturer of the analytical instrumentation. If a laboratory chooses to consult with an instrument manufacturer, then the following should be considered. The laboratory should have an idea of the alloy matrices to be analyzed, elements and mass fraction ranges to be determined, and the expected performance requirements for each of these elements. The laboratory should inform the instrument manufacturer of these requirements so they may develop an analytical method which meets the laboratory's expectations. Typically, instrument manufacturers customize the instrument configuration to satisfy the end-user's requirements for elemental coverage, elemental precision, and detection limits. Instrument manufacturer developed analytical methods may include specific parameters for sample excitation, wavelengths, inter-element interference corrections, calibration and regression, equipment configuration/installation, and sample preparation requirements. Laboratories should have a basic understanding of the parameters derived by the manufacturer.

5. Description of Equipment

5.1 *Types of Spectrometers*—X-ray spectrometers can be classified as sequential, simultaneous, or hybrid (see 5.1.3).

5.1.1 *Sequential Spectrometers*—The sequential spectrometer disperses and detects secondary X-rays by means of an adjustable monochromator called a goniometer. Secondary X-rays emitted from the specimen pass through a mask that defines the viewed region of the specimen. Next, they enter a collimator, typically a Soller slit, and nonparallel X-rays are eliminated by being absorbed by the blades of the collimator. The parallel beam of X-rays strikes an analyzing crystal that disperses the X-rays according to their wavelengths. The dispersed X-rays are measured by suitable detectors, which may have an attached collimator in front of the entrance window. Adjustment of the goniometer changes the angle between the specimen, crystal, and detector, permitting the measurement of different wavelengths, and therefore, of different elements.

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[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.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

5.1.2 *Simultaneous Spectrometers*—Simultaneous spectrometers use an individual monochromator to measure a selected wavelength of X- rays for each element. A typical monochromator consists of an entrance slit, a curved (focusing) analyzing crystal, an exit slit, and a suitable detector. Secondary X-rays pass through the entrance slit and strike the analyzing crystal, which diffracts the wavelength of interest and focuses it through the exit slit to enter the detector. Some simultaneous instruments use flat crystals.

5.1.3 *Hybrid Spectrometers*—Hybrid spectrometers combine features found in sequential and simultaneous instruments. One type uses a set of fixed monochromators for key X-ray lines and a goniometer for choosing other lines. Another type uses a set of fixed monochromators along with an energy dispersive device for choosing other lines.

5.2 *Spectrometer Environment:*

5.2.1 *Temperature Stabilization*—A means for stabilizing the temperature of the spectrometer should be provided. The degree of temperature control should be specified by the manufacturer. Temperature stability directly affects instrument stability.

5.2.2 *Optical Path:*

5.2.2.1 A vacuum path is generally preferred, especially for the measurement of X-rays of sufficiently low energy (long wavelengths) to be absorbed by air or nitrogen. Instruments capable of vacuum operation should have a vacuum gauge to indicate vacuum level. An airlock mechanism should be provided to evacuate the specimen chamber before opening it to the spectrometer. A means of controlling evacuation time is a desirable feature.

5.2.2.2 A helium path is recommended when measurement of low energy X-rays is required and the specimen (such as a liquid) would be disturbed by a vacuum. Instruments equipped for helium operation should have an airlock for flushing the specimen chamber with helium before introducing the specimen into the spectrometer. A means of controlling helium flush time is a desirable feature. The manufacturer should also provide a means for accurately controlling the pressure of the helium within the spectrometer.

5.2.2.3 Operation with air in the optical path may be an option with some spectrometer designs.

NOTE 1—Some spectrometers do not allow operation in air because high X-ray flux generates ozone that damages elastomers in vacuum seals. Some spectrometers use bellows coupled to micro-switches as the safety interlock to prevent accidental exposure to X-rays by those repairing a spectrometer and to prevent damage resulting from operation with an air-filled optical path.

5.3 *Excitation*—A specimen is excited by X-rays generated by an X-ray tube powered by a high voltage generator. The wavelength distribution and flux of X-rays striking the specimen is varied by changing the power settings to the tube or by inserting filters into the beam path between the tube window and the specimen position.

5.3.1 *X-Ray Tube*—The X-ray tube may be one of two types: end-window or side-window. Depending upon the instrument, either the anode or the cathode is grounded. Cathode grounding permits the window of the X-ray tube to be thinner and thus affords more efficient transmittance of longer wavelengths.

5.3.1.1 X-ray tubes are produced with a variety of targets. The choice of the target material depends upon the wavelengths that require excitation. X-rays from certain materials excite longer wavelengths more efficiently. Other materials are better for exciting shorter wavelengths. Generally the choice of target material is a compromise.

5.3.1.2 X-ray tubes are rated according to maximum power, maximum current, and typical power settings. These should be specified by the manufacturer.

5.3.2 *High Voltage Generator*—The high voltage generator supplies power to the X-ray tube. Its stability is critical to the precision of the instrument.

5.3.2.1 The dc voltage output of the high voltage generator is typically adjustable within the range of 20 kV to 60 kV. Some designs operate at lower voltage and some provide up to 100 kV. Voltage stability, thermal drift, and voltage ripple should be specified. Voltage repeatability should be specified for a programmable generator.

5.3.2.2 The current to the X-ray tube is typically adjustable within the range of 5 mA to 125 mA, with some supplies rated up to 160 mA. Current stability and thermal drift should be specified. Current repeatability should be specified for programmable generators.

5.3.2.3 Voltage and current recovery times should be specified for programmable generators. The software routines which control the generator must delay measurement until the generator recovers from voltage or current changes.

5.3.2.4 Input power requirements should be specified by the manufacturer so the proper power can be supplied when the instrument is installed. Maximum generator power output should be stated.

5.3.3 *Cooling Requirements*—The X-ray tube and some high voltage generators require cooling by either filtered tap water or a closed-loop heat exchanger system.

5.3.3.1 The manufacturer should specify water flow and quality requirements.

5.3.3.2 To protect components from overheating, an interlock circuit that monitors either water coolant flow or temperature or both should shut down power to the X-ray tube whenever these requirements are not met.

5.3.3.3 Water purity is especially critical in cathode-grounded systems because the coolant must be nonconducting. A closed-loop heat exchanger is necessary to supply high purity, low conductivity water. A conductivity gauge may be provided to protect the X-ray tube when conductivity becomes too high. The closed loop may incorporate an ion exchange resin to maintain water purity.

5.3.4 *Primary Beam Filter*—A primary beam filter is commonly used in sequential spectrometers to filter out characteristic emissions from the X-ray tube target when these emissions might interfere with measurement of an analyte element. Primary beam filters are also useful for lowering background in the longer wavelength (lower energy) portion of the spectrum. This serves to increase the peak to background ratio and to lower detection limits.

5.3.4.1 Primary beam filters are made of several different metals (depending upon the X-ray tube target) and come in a