Designation: E1172 – 16

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 variety of thicknesses. The manufacturer should specify the type, thickness, and location of the primary beam filter.

5.4 *Sample Positioning*—The process of positioning a specimen for measurement in a spectrometer involves several components: the specimen holder, the specimen changer, and the specimen rotation mechanism (spinner). These components contribute collectively to the repeatability of positioning the specimen in the optical path and thus to instrument precision.

5.4.1 If provided, a spinner rotates the specimen while it is being exposed to the primary X-ray beam, helping to minimize the influence of surface defects and specimen heterogeneity on analytical results. Specimen rotation rate should be specified by the manufacturer.

5.4.2 Imperfections in the surface of a specimen have the greatest effect on analytical results in spectrometers having a shallow angle of incidence of primary X-rays with respect to the specimen surface or a shallow angle of viewing the secondary X-rays with respect to the specimen surface, or both. The manufacturer should specify these angles.

5.4.3 Maximum specimen size (thickness and diameter) should be specified.

5.5 *Dispersion*—The analyzing crystal is the dispersive device in a wavelength dispersive X-ray spectrometer. A number of crystals providing a range of interplanar spacings are used to disperse the secondary X-rays.

5.5.1 Sequential spectrometers may contain several different crystals mounted on a changer mechanism to allow the analyst to select a specific crystal for the wavelength being measured. Crystals of similar lattice spacing, but different composition, may offer significantly different reflection efficiency.

5.5.2 Each monochromator in a simultaneous instrument has a specified crystal selected in accordance with the expected analytical requirements. The crystal is generally bent and ground to a curve or a logarithmic spiral to focus the diffracted X-rays through the monochromator's exit slit.

5.5.3 The manufacturer should specify each crystal installed in a particular spectrometer according to composition, location (which monochromator or crystal changer position), lattice orientation (when applicable), interplanar spacing, and shape (flat or curved).

5.5.4 The manufacturer should provide an adjustment for rotating the crystal to align it with (or peak) the monochromator.

5.6 *Beam Moderating Devices:*

5.6.1 *Soller Slits*—Soller slits[3] are composed of a series of closely-spaced, thin, parallel plates or tubes. When a Soller slit is placed in the path of a beam of X-rays, only those X-rays that travel parallel to the plates or tube axes will pass through to strike the crystal.

5.6.1.1 Soller slits may be present in several locations. A primary Soller slit is present in the optical path between the specimen and the analyzing crystal. Auxiliary slits may be installed at the detector windows between the detector and the analyzing crystal.

5.6.1.2 It is common for a sequential spectrometer to have a selection of Soller slits with different blade spacing mounted in a changer mechanism. Better resolution is achieved with a fine slit, but at the expense of a loss of intensity.

5.6.1.3 The manufacturer should specify the location and plate spacing of all Soller slits installed in a particular instrument.

5.6.2 *Entrance and Exit Slits*—Both entrance and exit slits are required in a curved-crystal spectrometer. The curved crystal establishes a focusing circle that is similar to the Rowland circle defined by a grating in an atomic emission spectrometer. In contrast, proper focusing in an X-ray fluorescence spectrometer requires that both slits not only be on the focusing circle but also have identical chordal distances from the slits to the crystal. A detector is aimed at the crystal through the exit slit.

5.6.2.1 The manufacturer should specify the sizes of the entrance and exit slit for each monochromator and should provide adjustments to peak each monochromator. Depending upon the manufacturer, peaking may involve movement of the crystal, the exit slit, or both.

5.6.3 *Apertures*—In some spectrometer designs, an aperture is placed in the optical path between the specimen and the entrance slit for the monochromator. The aperture limits the area of the specimen seen by the monochromator. The spectrometer may be equipped with a selection of apertures on a mechanical changer. The manufacturer should specify the sizes of available apertures.

5.6.4 *Attenuators*—When an element is present at a high concentration, it is sometimes desirable to decrease the intensity of its emissions to avoid exceeding the detector's linear counting range. An attenuator, positioned between the specimen and the analyzing crystal, absorbs some of the secondary X-rays and thus lowers the intensity. The manufacturer should specify the location (which monochromator) and the attenuation ratio of all attenuators.

5.6.5 *Masks*—Masks must be properly positioned in the optical path to prohibit secondary X-rays from bypassing the crystal and reaching the detector directly. This is particularly important at large 2θ angles.

5.7 *Detection:*

5.7.1 *Goniometer*—The goniometer in a sequential or hybrid spectrometer is an integral part of its detection system. Adjusting the goniometer varies the angle (called the 2θ angle) between the specimen, crystal, and detector, thus changing the wavelength seen by the detector. The spectrometer therefore can be set to measure the intensity at a specific wavelength.

5.7.1.1 It is critical that the angle indicated by the goniometer readout be an accurate and reproducible indication of the actual 2θ angle. The manufacturer should specify the reading accuracy, reproducibility, and scanning range of the goniometer and provide control of the operation with a range of options for scan speed.

5.7.2 *Detectors*—Detectors commonly used in wavelength dispersive X-ray spectrometers include Geiger-Müller counters, gas-filled and gas-flow proportional counters, and scintillation counters. Each detector is powered by a high voltage power supply, capable of supplying a range of voltages.

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[3] Soller, Walter. "A New Precision X-Ray Spectrometer," *Phys. Rev.*, Vol. 24, 1924, pp. 158–167,

Detectors should be specified according to their type, location within the spectrometer, and, for gas-filled detectors, detector gas and window thickness.

5.7.2.1 A single detector is required for each monochromator in a simultaneous instrument. Sequential instruments, however, generally have several detectors to provide high sensitivity throughout the wavelength range accessible using the monochromator. It is common for two detectors to be mounted and operated in tandem on the detector arm of a sequential spectrometer. The pair may include a gas-flow proportional counter coupled with either a gas-filled proportional counter or a scintillation counter. Another arrangement is to mount several detectors on a changer mechanism.

5.7.2.2 Gas is supplied to gas-flow proportional counters from a cylinder fitted with a pressure regulator. The spectrometer should have a mechanism for controlling the flow and stabilizing the pressure of the detector gas as this is critical to counting stability. Instruments having more than one gas-flow proportional counter require either multiple delivery systems or a manifold to feed all detectors.

5.7.2.3 A detector's sensitivity as a function of X-ray wavelength and count rate linearity is limited. The manufacturer should provide information about the linearity of the detection system. Typically, manufacturers should provide information and software tools to allow a user to measure the performance of a detector with a specimen and to select from a range of signal processing options.

5.8 *Signal Processing and Display:*

5.8.1 *Amplification*—The detector's signal is usually magnified by a preamplifier and a series of amplifiers before being processed further. Most instruments locate the preamplifier at the detector, which makes the instrument less susceptible to electronic noise.

5.8.1.1 Each detector has its own preamplifier. It is common in sequential instruments, however, to direct the output current of all of the preamplifiers into a single amplifier.

5.8.1.2 Simultaneous instruments have separate preamplifiers for each monochromator. In some instruments, these preamplifiers feed current to separate amplifiers while in others, the current signal from each preamplifier is collected on separate capacitors and then read sequentially through a single amplifier.

5.8.1.3 The manufacturer should describe the spectrometer's amplification scheme. Typical amplifier gain settings should also be specified.

5.8.2 *Pulse Height Discrimination*—As voltage pulses pass from the amplifier through the pulse height discriminator, pulses with a voltage below a prescribed lower limit or above a prescribed upper limit are filtered out, and only those pulses between the two limits are allowed to pass for further processing. Pulse height discrimination is effective for removing electronic noise and interferences which are approximately at the same 2θ angle but of a different diffraction order. Typically, software allows for pulse height discriminators to be set for each X-ray wavelength to be measured.

5.8.3 *Other Circuitry*—Other circuits may include those for pulse shaping and automatic dead-time correction. Automatic dead-time correction extends the linear range of the detectors.

5.9 *Software:*

5.9.1 Typically the spectrometer and data processing capabilities are controlled by a sophisticated software operating system that controls the operations of data gathering, calculation of analytical curves, and conversion of the data to final results.

5.9.2 Software design is a major criterion to consider when evaluating an instrument. The data gathering routine should be closely studied as this directly affects the quality of the data. Software should be fully documented.

5.9.2.1 Algorithms for converting measured data to final results may be either fundamental or empirical and should be specified and documented by the manufacturer.

5.9.2.2 The maximum number of standards and corrections that the software can accept should be specified. The manufacturer should specify the maximum number of characters for sample identification and formatting for output.

5.9.2.3 Manufacturers should provide other software routines that are helpful to the analyst. These may include plotting, scanning, instrument diagnostics, and so forth. These ancillary routines should be specified and documented.

6. Performance Criteria

6.1 *Detector Linearity*—The manufacturer should specify the maximum intensity which each detector can accept while maintaining a linear response.

6.1.1 To test a detector's linearity, first select a pure element specimen that will provide an X-ray line within the optimum response range of the detector. Measure that element at a constant voltage while increasing the current stepwise. Prepare a plot of intensity versus current. The plot should curve toward horizontal at the higher intensities. Extrapolate a straight line from the lower, linear portion of the plot upward. The point at which the plot appears to depart from the straight line indicates the intensity at which the detection system's response becomes nonlinear.

6.2 *Precision*—The precision of an X-ray spectrometer is a function of several variables. These include power stability, electronic noise, detector gas stability, counting statistics, and the repeatability of the optical alignment. The mechanical nature of the optical alignment task for sequential spectrometers may make them less precise than their simultaneous counterparts.

6.2.1 To test the precision of an X-ray spectrometer, choose a time for the set of measurements. Next, select a specimen containing an element with a concentration of at least 1000 times its detection limit and make at least 10 consecutive measurements, preferably many more. Measurement conditions should be set to ensure at least 1 000 000 total counts per measurement. The sample cup should be removed and reinserted and all mechanical settings (that is, crystal, collimator, filter, attenuator, aperture, and goniometer setting) changed between measurements. The relative standard deviation of the instrument tested in this way should be in the range of 0.1 % to 0.3 %.

NOTE 2—Because X-ray fluorescence spectrometers, especially wavelength dispersive designs, are highly stable and highly automated, it is appropriate to be concerned about spectrometer stability over periods of time from minutes to hours and even to days. It is important to measure instrument performance over the periods of time needed by the laboratory.

6.3 *Detection Limit*—The detection limit in X-ray spectrometry is a function of the sensitivity (the slope of the analytical curve) and the precision of the background measurement. For guidance on estimation of the detection limit, refer to Section 6.5 in Guide E2857.

7. Safety Features

7.1 *"X-Rays On" Lamp*—The manufacturer should provide one or more lamps that will indicate that the X-ray tube is energized and provide a "fail-safe" design in which the power to the X-ray tube is shut down if a lamp fails to operate.

7.2 *Interlock System*—A safety interlock system (see 5.3.1.2 and 5.3.2.4) should be provided to protect the user from harmful exposure to X-rays or high voltage. It should shut down power to the X-ray tube or to high voltage components if improper or unauthorized access is attempted.

7.3 *Coolant Interlock*—See 5.3.3.2.

7.4 *Emergency Shutdown System*—An emergency shutdown system should be provided to protect the spectrometer in the event of a power failure. A power outage should deactivate the power switch to the X-rays so that the switch must be reset manually. A power failure should also deactivate a solenoid valve to seal the spectrometer chamber so that pump fluid is not pulled into the chamber. The pressure within the spectrometer chamber will then be allowed to increase gradually to atmospheric pressure, thus protecting the gas proportional detector windows from rupture.

8. Keywords

8.1 sequential spectrometer; simultaneous spectrometer; spectrometry; X-ray fluorescence; X-ray spectrometer