Designation: E 1172 – 87 (Reapproved 2001)

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

This standard is issued under the fixed designation E 1172; 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 describes 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. The document does, however, attempt to identify which of these 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 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.

1.3 There are several books and publications from the National Institute of Standards and Technology[2] and the U.S. Government Printing Office[3,4] which deal with the subject of X-ray safety. Refer also to Practice E 416[5].

2. Referenced Documents

2.1 *ASTM Standards:*

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[5]

E 416 Practice for Planning and Safe Operation of a Spectrochemical Laboratory[5]

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data[5]

3. Terminology

3.1 For terminology relating to X-ray spectrometry, refer to Terminology E 135.

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.

5. Description of Equipment

5.1 *Types of Spectrometers*—X-ray spectrometers can be classified as sequential, simultaneous, or a combination of these two (hybrid).

5.1.1 *Sequential Spectrometers*—The sequential spectrometer disperses and detects secondary X rays by means of an adjustable monochromator called a goniometer. In flat-crystal instruments, secondary X rays are emitted from the specimen and nonparallel X rays are eliminated by means of a Soller slit (collimator). The parallel beam of X rays strikes a flat analyzing crystal which disperses the X rays according to their wavelengths. The dispersed X rays are then measured by suitable detectors. Adjusting the goniometer varies the angle between the specimen, crystal, and detector, permitting the measurement of different wavelengths and therefore different elements. Sequential instruments containing curved-crystal optics are less common. This design substitutes curved for flat crystals and entrance and exit slits for collimators.

5.1.2 *Simultaneous Spectrometers*—Simultaneous spectrometers use separate monochromators to measure each element. These instruments are for the most part of fixed configuration, although some simultaneous instruments have a scanning channel with limited function. 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 where it is measured by the detector. Some simultaneous instruments use flat crystals, but this is less common.

5.1.3 *Hybrid Spectrometers*—Hybrid spectrometers combine features found in sequential and simultaneous instruments. They have both fixed channels and one or more fully functional goniometers.

5.2 *Spectrometer Environment*:

[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] NBS Handbook, *X-Ray Protection*, HB76, and NBS Handbook 111, ANSI N43.2-1971, available from National Institute of Standards and Technology, Gaithersburg, MD 20899.

[3] *Radiation Safety Recommendations for X-Ray Diffraction and Spectrographic Equipment*, No. MORP 68-14, 1968, available from U.S. Department of Health, Education, and Welfare, Rockville, MD 20850.

[4] U.S. Government Handbook 93, *Safety Standards for Non-Medical X-Ray and Sealed Gamma-Ray Sources*, Part 1, General, Superintendent of Documents, available from U.S. Government Printing Office, Washington, DC 22025.

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

5.2.1 *Temperature Stabilization*—A means for stabilizing the temperature of the spectrometer shall be provided. The degree of temperature control shall 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 analysis of light elements (long wavelengths). Instruments capable of vacuum operation shall have a vacuum gage to indicate vacuum level. An airlock mechanism shall also be provided to pump down the specimen chamber before opening it to the spectrometer. Pump down time shall be specified by the manufacturer.

5.2.2.2 A helium path is recommended when light element analysis is required and the specimen (such as a liquid) would be disturbed by a vacuum. Instruments equipped for helium operation shall have an airlock for flushing the specimen chamber with helium before introducing the specimen into the spectrometer. Helium flushing time shall be specified by the manufacturer. The manufacturer shall also provide a means for accurately controlling the pressure of the helium within the spectrometer.

5.2.2.3 An air path is an option when the instrument is not equipped for vacuum or helium operation. Light element analysis and some lower detection limits are sacrificed when operating with an air optical path.

5.3 *Excitation*—A specimen is excited by X rays generated by an X-ray tube which is powered by a high voltage generator and is usually cooled by circulating water. The intensity of the various wavelengths of X rays striking the specimen is varied by changing the power settings to the tube or by inserting filters into the beam path.

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 the longer excitation 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 the longer wavelengths more efficiently. Other materials are better for exciting the 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. **Warning:** It is important that the user be protected from exposure to harmful X rays. Standard warning labels shall warn the user of the possibility of exposure to X rays. Safety interlock circuits (7.3) shall shut down power to the X-ray tube whenever protective shielding is removed.

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 d-c voltage output of the high voltage generator is typically adjustable within the range of 10 to 100 kV. Voltage stability, drift with temperature, and voltage ripple should be specified. Voltage repeatability should be specified for a programmable generator, which is frequently used in sequential systems.

5.3.2.2 The current to the X-ray tube is typically adjustable within the range of 5 to 100 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. **Warning:** Safety is a primary concern when dealing with high voltage. Safety interlock circuits (7.3) and warning labels shall protect the user from coming in contact with high voltage. The interlock system shall shut down the generator when access to high voltage is attempted. Circuits shall be provided to protect the X-ray tube from power and current overloads.

5.3.3 *Water 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 shall 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 shall 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 since this requires the coolant to be nonconducting. A closed-loop heat exchanger is necessary to supply high purity cooling water. A conductivity gage shall monitor water coolant purity in these systems and shall shut down power to the X-ray tube when coolant purity is below requirements.

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

5.3.4.1 Primary beam filters are made of several different metals (depending upon the X-ray tube's target) and come in various 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 in a spectrometer for analysis involves several components; the specimen holder, the specimen changer, and the specimen rotation mechanism (spinner). These components contribute collectively to the reproducibility of positioning the specimen in the optical path and thus, to instrument precision. The design of these components should therefore be regarded critically.