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Standard Guide for Selecting Components for Energy-Dispersive X-Ray Fluorescence (XRF) Systems¹

This standard is issued under the fixed designation C 982; 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.

^{ε1} NOTE—Keywords were added editorially in March 1998.

1. Scope

1.1 This guide describes the components for an energy-dispersive X-ray fluorescence (XRF) system for materials analysis. It can be used as a reference in the apparatus section of test methods for energy-dispersive X-ray fluorescence analyses of nuclear materials.

1.2 The components recommended include X-ray detectors, signal processing electronics, data acquisition and analysis systems, and excitation sources that emit photons (See Fig. 1).

1.3 Detailed data analysis methods are not described or recommended, as they may be unique to a particular analysis problem. Some applications may require the use of spectrum deconvolution to separate partially resolved peaks or to correct for matrix effects in data reduction.

1.4 *This standard does not purport to address all of the safety problems, 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 181 General Methods for Detector Calibration and Analysis of Radionuclides³

3. Significance and Use

3.1 This guide describes typical prospective analytical X-ray fluorescence systems that may be used for qualitative and quantitative elemental analysis of materials related to the nuclear fuel cycle.

3.2 Standard methods for the determination of materials

using energy-dispersive XRF⁴ usually employ apparatus with the components described in this document.

4. Hazards

4.1 XRF equipment analyzes by the interaction of ionizing radiation with the sample. Applicable safety regulation and standard operating procedures must be reviewed prior to the use of such equipment. (See NBS Handbook 111.⁵)

4.2 Instrument performance may be influenced by environmental factors such as heat, vibration, humidity, dust, stray electronic noise, and line voltage stability. These factors and performance criteria should be reviewed with equipment manufacturers.

4.3 The quality of quantitative XRF results can be dependent on a variety of factors, such as sample preparation and mounting. Consult the specific analysis method for recommended procedures.

4.4 Sample chambers are available commercially for operation in air, vacuum, or helium atmospheres, depending upon the elements to be determined and the physical form of the sample.

5. Energy Dispersive X-Ray Detectors

NOTE 1—Because of the rapid improvement in detector and electronics technologies, the most up-to-date information on XRF components is found in manufacturers' literature. Lists of vendors of XRF equipment can be found in compilations such as the "Guide to Scientific Instruments," published by the American Association for the Advancement of Science, Washington, DC.

5.1 Energy-dispersive X-ray detectors can be used to detect X rays with energies from approximately 1 to 100 keV; however, a single-type detector usually cannot satisfy all the

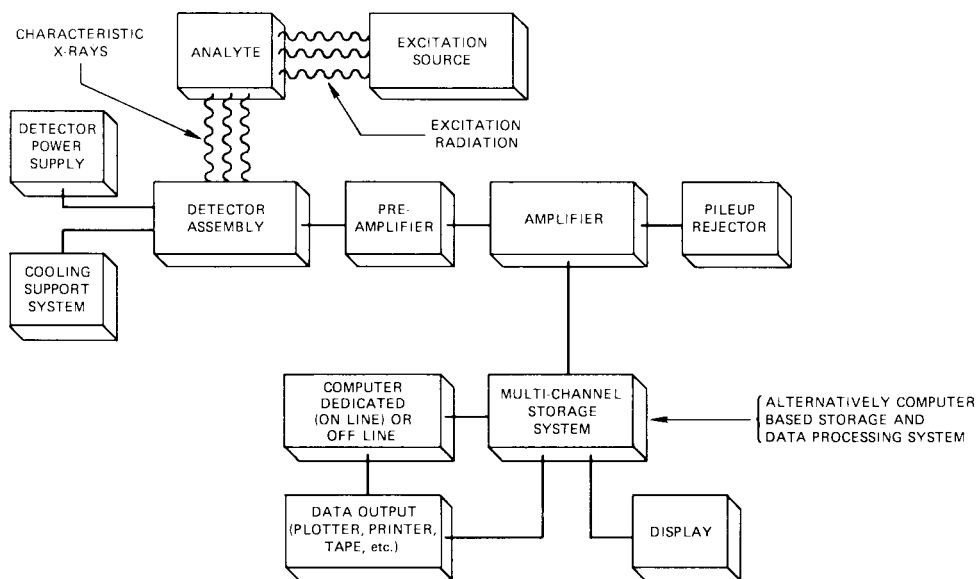
⁴ General References for XRF include Bertin, Eugene P., *Principles and Practices of X-Ray Spectrometric Analysis*, Second Ed., Plenum Press, New York-London, 1975, Jenkins, Ron, *An Introduction to X-Ray Spectrometry*, Heyden and Sons, Ltd., London, New York, Rhine, 1974, and Woldseth, Rolf, *All You Ever Wanted to Know About X-Ray Energy Spectrometry*, First Ed., Kevex Corporation, Burlingame, CA, 1973.

⁵ *Radiation Safety for X-Ray Diffraction and X-Ray Fluorescence Analysis Equipment*, National Institute of Standards and Technology, Washington, DC.

¹ This guide is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test. Current edition approved Nov. 25, 1988. Published February 1989.

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

³ *Annual Book of ASTM Standards*, Vol 12.02.



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FIG. 1 Function Block Diagram of XES System

requirements of efficiency and energy resolution over such a wide energy range.

5.2 The energy resolution (Terminology E 135) of a detector is usually specified by the FWHM (full width at half maximum) of the full energy peak of an X ray (or γ ray) of a particular energy and at a specified count rate. The FWTM (full width at one-tenth maximum) of the full-energy peak or the peak-to-background ratio, or both, may also be specified. "High resolution" (small values of FWHM) detectors are required to separate X rays of similar energy emitted by different elements.

5.3 The dimension of the detector "active volume" is usually specified for X-ray detection applications, allowing the efficiency of the device for detecting a particular energy X ray to be estimated.

5.4 XRF analysis systems requiring high-resolution detectors employ semiconductor ("solid state") detectors.

5.4.1 Lithium-drifted silicon, Si(Li), detectors are usually used for applications requiring the detection of X rays from 1 to 40 keV. Si(Li) resolution is commonly specified at 5.9 keV and 1000 cps. Resolutions of 145 to 160 eV (FWHM) are typical.

5.4.2 Germanium, Ge, detectors or lithium-drifted germanium, Ge(Li), detectors are usually employed to detect X rays in the high-energy region of the X-ray spectrum. Ge resolution is commonly specified at 122 keV and 1000 cps when the detector is to be used for X-ray detection. Resolutions of 500 eV (FWHM) are typical.

5.4.3 Both Ge(Li) and Si(Li) detectors must be operated at 77°K (liquid nitrogen temperature). Si(Li) detectors can be stored at room temperature. Germanium detectors must be stored at 77°K (Standard E 181).

5.4.4 Improvements in technology can extend the useful energy range of a particular detector type or result in the development of new detector materials. Manufacturers should be consulted for the latest information.

5.5 Scintillation detectors such as NaI(Tl), or gas-filled

proportional counters, may be used in certain applications in which the high resolution of the semiconductor detectors is not required.

5.6 All types of X-ray detectors have entrance windows of a low-Z material (for example, Be on solid-state detectors or plastic film on gas-filled counters) to minimize X-ray absorption.

6. Signal Processing Electronics

6.1 The bias power supply required to operate X-ray detectors must be capable of delivering sufficient high voltage and current for the particular detector. The power shall be regulated and have ripple and noise content below the 10-mV level.

6.2 A preamplifier converts the charge pulse caused by an ionizing event in the detector to a voltage signal. It must also minimize electronic noise that would degrade the spectrum resolution. The preamplifier is typically charge sensitive and uses a field-effect transistor. The noise content, gain, and count-rate capability must be compatible with the particular detector and application. "Resistive feedback" and "pulsed-optical feedback" are two types of preamplifier techniques suitable for high-resolution spectroscopy. The preamplifier is usually supplied as an integral part of the detector, and the detector manufacturer should be consulted to determine a suitable preamplifier.

6.3 A shaping amplifier is used to integrate the preamplifier pulse for a well-defined duration, differentiate the pulse to provide an acceptable shape, and amplify the pulse to an appropriate voltage. Typical shaping times vary from 1 to 40 μ s depending on the detector count rate and the required energy resolution. Amplifier outputs are approximately ten volts maximum. The amplification factor is variable so that the output pulse height spectrum corresponds to the desired range of X-ray energies. Most shaping amplifiers provide power to the preamplifier. High-resolution amplifiers are equipped with adjustments for signal shaping to optimize the resolution at a given rate. Some high-resolution amplifiers are equipped with