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Standard Guide for Selecting Components for Energy-Dispersive X-Ray Fluorescence (XRF) Systems[1]

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.

ε[1] 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 Materials[2]

E 181 General Methods for Detector Calibration and Analysis of Radionuclides[3]

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 XRF[4] 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

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

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

[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.

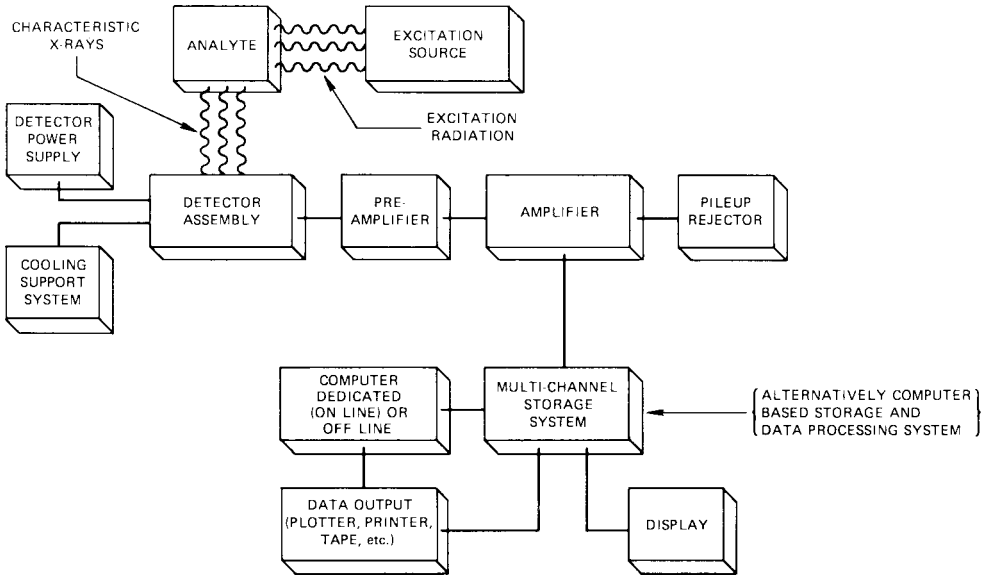

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FIG. 1 Function Block Diagram of XES System

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(T1), 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 μs 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

pileup rejection circuitry. (See 6.4.) Most commercial shaping amplifiers in these applications are nuclear instrument module (NIM) standard modules.

6.4 Pulse pileup rejection is employed to exclude from the processed data those signals with amplitudes that may be altered by the close occurrence in time of two detected events. Pileup rejection units generally permit the selection of a resolving time within which the occurrence of two events causes the generation of a logic signal. The logic signal is used to reject the time-coincident linear signal in a circuit designed for this purpose. The latter circuit can be incorporated in the pileup rejection module, in the data acquisition unit (multichannel analyzer), or in a separate gating module. The reduction of the influence of pulse pileup can also be obtained with circuits which apply “baseline restoration” to the pulse train, and it is possible to combine both baseline restoration and pulse pileup techniques. A high overlap of pulses can result in loss of the true zero of reference to give an erroneous measure of pulse amplitude. Pole-zero cancellation reduces these effects, but where a resolution of better than 0.1 % at high rates (~50 kHz) is needed, baseline restoration appears to be a practical solution. Both passive and active methods have been developed with the active methods showing greater superiority at higher count rates. These circuits can either be incorporated in the main amplifier, or they can be part of the pulse amplitude analyzer system.

6.5 Counting losses caused by pulse pileup or electronics deadtime increase with increased counting rates. Accurate quantitative X-ray analysis requires that data be normalized to correct for these variable losses. Corrections are performed by counting the number of events accumulated in a peak generated by a fixed-frequency electronic pulser, or a radioisotopic source in fixed counting geometry, or by increasing the counting time of the data acquisition system based on an electronic analysis of counting losses. Commercial units are available for automatic correction for count rate losses.

6.6 Gain stabilization to maintain a constant relationship between X-ray energy and electronic pulse height or multichannel analyses channel may be required for applications in some environments. The overall system gain can be stabilized using electronic means or computer software techniques.

7. Data Acquisition and Analysis

7.1 *Multichannel Analyzer (MCA)*:

7.1.1 The most frequently employed unit for storage and counting of pulses from the shaping amplifier is the MCA. MCA memory consists of a minimum number of locations (channels) to which counts are assigned based on the amplitudes of pulses associated with detected events. Each linear amplified signal is digitized (assigned a numerical value) proportional to its amplitude. One count per event is stored in the channel corresponding to the digital quantity. Since the number of channels is finite, the channels store the counts of events within (continuous) energy bins of the amplified pulse height distribution. A two-dimensional display of the number of counts versus channel shows peaks in the appropriate channels corresponding to the X-ray energies. The range (in volts) of pulse height that can be digitized is often MCA-controllable. Computer control of MCA data acquisition is a common mode of operation.

7.1.2 The most basic form of MCA data analysis involves a hand integration of peak areas. MCA memory is frequently part of computer memory or accessible to a computer central processor. In this case, data analysis can be automated. Most MCA systems contain either preprogrammed software or firmware controlled by mechanical operation of buttons or switches, which perform, for example, peak integration, energy calibrations, or resolution measurements. They may also provide programmable memory for increased automation or for more complex or specific data analysis, which may include fitting peaks and backgrounds. As a third option, the MCA can be interfaced to a computer for direct access to the analyzer memory and functions or for the transfer of stored data to the computer for analysis. The analysis software should be accessible to the user for editing or modification to suit the needs of the particular application. Source listings of the analysis programs of commercial software packages should be available to the user.

7.2 *Single Channel Analyzer (SCA)*:

7.2.1 The function of a single MCA channel is performed by the SCA. This is an electronic component that generates a logic signal for each linear input signal that the amplitude of the input signal falls within a window defined by certain voltage limits. The range and spacing of the limits are adjustable on the SCA. An SCA rather than an MCA might be employed for reasons of economy, portability, or space constraint. Its use is limited to measurements of relatively simple spectra from samples with well-defined components or for survey measurements where high accuracy is not required. A scaler is normally used to count the SCA logic output signals.

7.2.2 The simplest form of SCA data analysis involves counting the number of events in a window that brackets a peak of interest. In this case a peak must be well resolved from neighboring peaks and have negligible background. When background is nonnegligible, two measurements of equal count duration can be made with a fixed-width window set to bracket the peak in one case and the background in the other. The background count can then be subtracted from the peak count. This procedure can be simplified to a single measurement if two SCAs are em-ployed. The use of multiple SCAs and logical summing units can give directly the number of background-subtracted counts in one or more well-resolved peaks from a single measurement.

8. Photon Excitation Sources

8.1 Radioisotopic sources are convenient and low-cost methods of producing X-ray photoexcitation. The choice of radioisotope, source strength, and geometry is dependent on application. ^{109}Cd for L X-ray excitation and ^{57}Co for K X-ray excitation of nuclear material are typical sources. Useful source strengths are generally determined by system count rate capability. Sources in an annular geometry provide excellent uniformity of intensity across the sample surface and a tight counting geometry. (See Fig. 2.) Point sources are also used.

8.2 X-ray generators provide a source of photons with variable energy and intensity. The X-ray generator should provide X rays of energy at least slightly greater than the absorption edge energy of interest. The X-ray tube, available

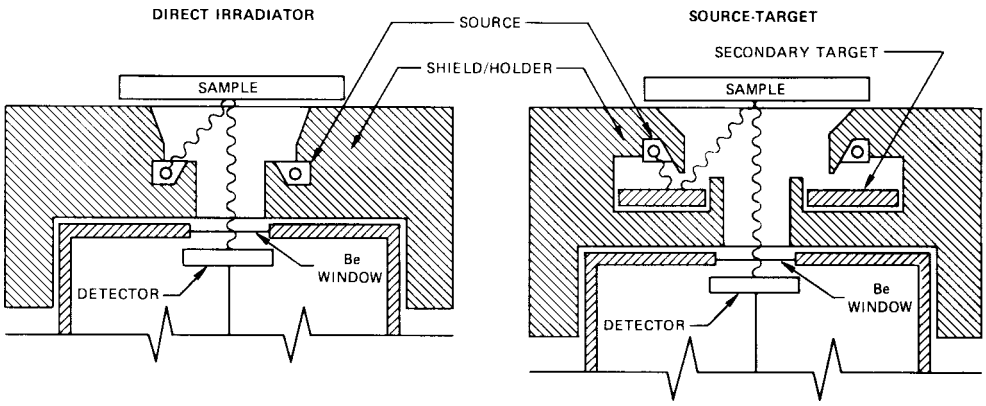

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FIG. 2 Schematic Representation of Radioisotope Excitation System with Annular Source Configuration, (a) Direct Irradiator; (b) Secondary Target Irradiator

with a target(s) of various high-purity elements, should be capable of uninterrupted operation up to the potentials and currents required for the particular application.

8.3 The radiation from radioisotopic sources and X-ray generators can be modified by the use of filters or secondary sources or can be utilized in the direct mode.

9. Keywords

9.1 energy dispersive x-ray fluorescence; instrument components