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

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1. Scope

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

1.2 The components recommended include X-ray detectors, signal processing electronics, excitation sources, and dispersing crystals.

1.3 Detailed data analysis procedures are not described or recommended, as they may be unique to a particular analysis problem. Some applications may require the use of complex computer software during data reduction to correct for matrix effects.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[2]

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 analyses of materials related to the nuclear fuel cycle.

3.2 Standard test methods for the determination of materials using wavelength-dispersive XRF[3] usually employ apparatus with the components described in this guide.

4. Technical Precautions

4.1 XRF equipment analyzes by the interaction of ionizing radiation with the sample. Applicable safety regulation and standard operating procedures must be reviewed before use of such equipment. All current XRF spectrometers are equipped with safety interlocks to prevent accidental penetration of X-ray beam by the user. Do not override these interlocks without proper training or a second person present during such operation. (See NBS Handbook 111[4])

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 on the elements to be determined and the physical form of the sample.

5. Excitation Sources

5.1 *X-Ray Generator*— The X-ray generator should consist of, but not be limited to, an X-ray power supply with an output rating of at least 3000-W constant power. The voltage should be adjustable from at least 10 to 60 kV in not greater than 5-kV increments (100-kV generators are available if the specific application requires). Tube current should be adjustable from at least 5 to 80 mA in suitable increments. The current increments should be no greater than 5 mA between 5 and 40 mA and no greater than 10 mA between 40 and 80 mA. Adjustment of both X-ray tube voltage and current should be either manual or automatic by means of the hardware and software programs at the user's option. The kilovolt and milliamp adjustments should be interlocked such that if the maximum safe power of the X-ray tube or the power supply is exceeded the generator will either shut off automatically or will limit itself to the

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

[3] General references for XRF include the following:
Bertin, Eugene P., *Principles and Practices of X-ray Spectrometric Analysis*, Second Edition, Plenum Press, New York and London, 1975.
Jenkins, Ron, *An Introduction to X-ray Spectrometry*, Hayden and Sons Limited, London, New York, Rhine, 1974.
Jenkins, Ron, Gould, R. W., and Gedke, Dale, *Quantitative X-ray Spectrometry*, Marcel Dekker, Inc., New York and Basel.

[4] NBS Handbook 111, *Radiation Safety for X-Ray Diffraction and X-Ray Fluorescence Analysis Equipment*, National Institute of Standards and Technology, Washington, DC.

maximum safe power. Stability and reproducibility of high voltage and tube current should be at least 0.05 % or better for a ±10 % change in line voltage and for ambient temperature variation of 15°C.

5.2 *Cooling System*— The system should be equipped with a recirculating cooling unit for cooling the X-ray power supply and the X-ray tube. The cooling water unit should have sufficient volume and cooling capacity to supply the recommended flow of cooling water at the optimum temperature and pressure recommended by the manufacturer of the X-ray analyzer system. The cooling unit and the X-ray power supply should be interconnected so that they are switched on and off simultaneously. An automatic interlock should be included to shut off both the X-ray power supply and the cooling water unit when either the maximum cooling water temperature is exceeded, or the cooling water flow is interrupted, or, in the event of a breakdown, in the cooling water circuit.

5.3 *X-Ray Tube*—An X-ray tube having a power rating of at least 3000 W and continuously operable at the voltages and currents specified in 5.1 should be included. The particular tube target (Terminology E 135) to be used will depend on the elements of interest. Normal choices for nuclear-related materials include chromium, molybdenum, gold, and rhodium.

6. Spectrometer

6.1 *Specimen Changer*— A sample changer that will accept solid or liquid samples or both of any size up to 50 mm in diameter and up to at least 25 mm in thickness should be included. Multiposition sample changers are available for most instruments, and the choice of such would depend on the user's application.

6.2 *Vacuum and Helium Flush Systems*—The X-ray analyzer system should be equipped with both vacuum and helium flush systems. A vacuum pump, vacuum gage, helium regulator, and all necessary tubing, valves, fitting, flow meters, and controls for vacuum, air, and helium X-ray analysis should be included. The inclusion of such systems will allow the analysis of solids, liquids, or powders for elements for fluorine (atomic number 9) to uranium (atomic number 92).

6.3 *Detectors*:

6.3.1 A flow proportional counter using argon/methane (P-10) gas and mounted inside the evacuable crystal chamber should be included. The flow counter should have a scanning range of at least 40 to 147° two-theta (2θ). The gas for the flow counter should be pressure and temperature stabilized to eliminate the influence of ambient pressure and temperature on the counter.

6.3.2 A scintillation detector should be supplied and have a scanning range of at least 5 to 88° two-theta (2θ). The detectors should be mounted in such a way that they may be used separately or in tandem. Alternately, a third sealed gas detector may be included for use over the analytical range in which tandem counting with the scintillation and flow-proportional counters would be done.

NOTE 1—If the quality of P-10 gas is not well known or controlled, a gas dryer may be required. The gas used for any flow-proportional counter should be moisture free.

6.4 *Crystal Chamber and Analyzing Crystals*:

6.4.1 An evacuable crystal chamber containing the crystal changer and the gas counter(s) should be supplied. The crystal changer should accommodate at least four (4) analyzing crystals. The crystal chamber should be temperature stabilized.

6.4.2 The choice of analyzing crystals will be dependent on the user's application. A complete list of available crystals is outside the scope of this guide.[3] Normal usage within the nuclear field would include LiF_{220}, LiF_{200}, and PET_{002}. All crystals should be mounted in metal boats and individually prealigned.

6.4.3 Layered synthetic microstructures (LMS) and other crystals may be desired for light element analysis.[5]

6.5 *Collimating System*—A collimating system that allows selection of coarse and fine collimation of the secondary X-ray beam should be provided. It should be possible to insert a limiting aperture between the sample and the collimators so as to eliminate spurious lines arising from sample holders or masks from reaching the detectors.

6.6 *Goniometer*—A scanning goniometer (see Terminology E 135) with a range of at least 5 to 147° two-theta (2θ) should be included. The goniometer control should have a selection of continuous scanning speeds of at least ¼, ½, 1, 2, and 4° two-theta (2θ) per minute. The goniometer should have a slewing speed of at least 400° two-theta (2θ) per minute in both directions. The reading accuracy of the goniometer setting should be 0.01° or better, and the reproducibility of the goniometer setting should be 0.001° or better.

6.7 *Automatic Pulse Height Control*—A sine amplifier, or some other mechanism such as programmatic control of detector voltage, which will provide functional amplification such that the average pulse height distribution is kept constant for all analyzing crystals over the entire range of the goniometer described in 6.6, should be supplied.

7. Electronic Measuring Units

7.1 *Linear Amplifier*— The linear amplifier should have amplification of up to at least 128 X. Gain controls should have amplification factors of at least 8, 16, 32, 64, and 128. Linearity should be 1 % or better over the pulse height analyzer window placement range.

7.2 *Pulse Height Analyzer*—The pulse height analyzer should have selectable lower level (baseline) plus window (channel width) and lower level only discrimination (differential and integral modes).

7.3 *Ratemeter*—The ratemeter should have both linear and logarithmic ranges. The log section should have a range of at least 10 to 10^5 and an accuracy of 10 % or better. The linear range should have selectable scaling to produce full-scale deflection at 1, 2, and 4 times 10^2, 10^3, 10^4, and 10^5 counts per second.

7.4 *Pulse Counter and Timer*:

7.4.1 The pulse counter should have a resolution (counting speed) of at least 10 MHz. It should have a decimal readout of at least 9 999 999. The counter should have preset count settings of at least 1, 2, and 4 times 10^2, 10^3, 10^4, 10^5, 10^6, and 10^7.

[5] Appendix 10 of Berlin's *Principles and Practices of X-ray Spectrometric Analysis* (see Footnote 3) has a list of crystals and multilayer films.

7.4.2 A timer with a crystal oscillator reference should be included. The timer should have controls for presetting times of at least the following: 1, 10, 100, and 1000 s with multipliers of 1, 2, and 4.

7.5 *High Voltage Supplies*—High voltage supplies (one for each detector) should be included. The scintillation detector supply should have an output voltage range of at least 500 to 1000 V, and the flow counter supply should have a range of at least 1000 to 2500 V.