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An American National Standard

# Standard Guide for Using Micro X-Ray Fluorescence (μ-XRF) in Forensic Polymer Examinations[1]

This standard is issued under the fixed designation E3295; 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.

## INTRODUCTION

Micro X-ray fluorescence spectrometry (μ-XRF) is one technique in an analytical scheme that can provide information regarding potential relationships between the sources of polymeric materials.

## 1. Scope

1.1 This guide covers recommended techniques and procedures intended for use by forensic laboratory personnel that perform μ-XRF analysis of polymer samples.

1.2 This guide describes various techniques and procedures used in the μ-XRF analysis of polymers that include sample handling and preparation, instrument operating conditions, and spectral data collection, evaluation and interpretation.

1.3 This guide describes the application of μ-XRF systems equipped with either mono- or poly- capillary optics and an energy dispersive X-ray detector (EDS).

1.4 This guide is intended to be applied within the scope of a broader analytical scheme (for example, Guide E1610, Guide E3260) for the forensic analysis of a polymer sample (**1-6**).[2] A μ-XRF analysis can provide additional information regarding the potential relationships between the sources of polymeric materials.

1.5 The fundamental aspects of the composition and manufacture of polymeric materials or theory of X-ray fluorescence can be found in various texts (**7-18**).

1.6 This standard is intended for use by competent forensic science practitioners with the requisite formal education, discipline-specific training (see Practices E2917, E3233, E3234), and demonstrated proficiency to perform forensic casework.

1.7 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*[3]

E620 Practice for Reporting Opinions of Scientific or Technical Experts

E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory

E1610 Guide for Forensic Paint Analysis and Comparison

E1732 Terminology Relating to Forensic Science

E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Programs

E2926 Test Method for Forensic Comparison of Glass Using Micro X-ray Fluorescence (μ-XRF) Spectrometry

E3233 Practice for Forensic Tape Analysis Training Program

E3234 Practice for Forensic Paint Analysis Training Program

E3260 Guide for Forensic Examination and Comparison of Pressure Sensitive Tapes

2.2 *SWGMAT Documents:*[4]

SWGMAT Trace Evidence Recovery Guidelines

SWGMAT Trace Evidence Quality Assurance Guidelines

[1] This guide is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.15 on Trace.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

[3] 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.

[4] Available from https://www.asteetrace.org/subtrace.

2.3 *ISO Standard:*[5]
ISO/IEC 17025 Laboratory Competence

## 3. Terminology

3.1 *Definitions*—The terms defined relate specifically to μ-XRF as described in this document. For additional terms commonly employed for general forensic examinations, see Terminology E1732.

3.2 *Definitions:*

3.2.1 *background radiation, n*—X-rays resulting from scattered Bremsstrahlung and coherently and incoherently scattered tube target peaks.

3.2.2 *characteristic X-ray, n*—X-ray emission resulting from de-excitation of an atom following inner shell ionization.

3.2.2.1 *Discussion*—The energy of a characteristic X-ray is related to the atomic number of the atom, providing the basis for energy dispersive X-ray spectroscopy.

3.2.3 *coherent (Rayleigh) scatter peaks, n*—spectral artifacts that result from elastic scattering of the tube target characteristic X-rays by the sample.

3.2.3.1 *Discussion*—Because no energy is lost in elastic scattering, coherent scatter peaks occur at the same energies as the tube target characteristic X-rays.

3.2.4 *dead time, n*—the time (expressed as a percentage of real time) during which the energy dispersive X-ray spectrometer is not able to process X-rays.

3.2.5 *diffraction peaks, n*—spectral artifacts that result from preferential diffraction of tube X-rays into the detector as a result of striking a crystalline sample.

3.2.5.1 *Discussion*—Diffraction peaks vary in energy and intensity depending on orientation of the crystalline planes with respect to the beam angle.

3.2.6 *escape peak, n*—a spectral artifact resulting from incomplete deposition of the energy of an X-ray entering the energy dispersive X-ray spectrometer detector.

3.2.6.1 *Discussion*—An escape peak is produced when an incoming X-ray excites a silicon atom within the detector crystal, and the resulting Si Kα fluorescence X-ray exits the detector crystal. It occurs at the energy for the original X-ray minus the energy of the Si Kα fluorescence X-ray (1.74 keV). The escape peak intensity is about 1-2 % of the parent peak.

3.2.7 *exclusionary difference, n*—a difference in one or more characteristics between compared items that is sufficient to determine that the compared items did not originate from the same source, are not the same source, or do not share the same composition or classification.

3.2.8 *incoherent (Compton) scatter peaks, n*—spectral artifacts that result from inelastic scattering of the tube target characteristic X-rays by the sample.

3.2.8.1 *Discussion*—Because energy is lost in inelastic scattering, incoherent scatter peaks occur at a lower energy than the tube target characteristic X-rays.

3.2.9 *KLM reference lines, n*—the energies associated with the transitions of the K, L, and M shell electrons.

3.2.9.1 *Discussion*—Each element has characteristic energies of transitions of electrons between shells.

3.2.10 *live time, n*—the time during which an energy dispersive X-ray spectrometer is available to accept and process incoming X-rays.

3.2.10.1 *Discussion*—Live time is often expressed as a percentage of real time, in seconds.

3.2.11 *pulse processor time constant, n*—operator-selected value for the time designated to record a response by the detector. A higher value (longer time) results in a more accurate determination of the detector amplifier pulse height (better spectral resolution). A lower value results in a higher count rate but with reduced spectral resolution.

3.2.12 *spectral artifacts, n*—spectral peaks other than characteristic peaks from the sample, produced during the energy dispersive detection process. Examples include escape peaks, sum peaks, tube target coherent and incoherent scatter peaks, system peaks, and diffraction peaks.

3.2.13 *spectral resolution, n*—measure of the ability to distinguish between adjacent peaks in a spectrum; it is usually determined by measuring peak width at half the maximum value of the peak height or full-width half-maximum (FWHM).

3.2.13.1 *Discussion*—This value is usually quoted for the FWHM of Mn Kα.

3.2.14 *sum peak, n*—a spectral artifact that results from the simultaneous detection of two X-rays, manifested as a peak at the combined energy of the detected X-rays.

3.2.15 *system peaks, n*—spectral artifacts that result from the production of characteristic X-rays from structural components of the XRF instrument.

## 4. Significance and Use

4.1 μ-XRF is a nondestructive qualitative elemental analysis technique used for polymers. It involves excitation of a sample by an X-ray source resulting in the emission of characteristic X-rays detected using an energy dispersive X-ray detector. Results are displayed simultaneously as a spectrum of intensity as a function of energy for elements of atomic number 11 or greater.

4.2 μ-XRF enables the determination of the elemental composition of a specimen and can be utilized for comparisons of components of polymeric materials (for example, tape backings, tape adhesives, paint layers).

4.3 Comparisons of X-ray spectra acquired from polymer samples are conducted for source discrimination or potential association.

4.4 Quantitative processes for μ-XRF analysis are available but are not used for polymer analyses because of the lack of prepared polymer standard reference samples.

4.5 In general, information available from a heterogeneous specimen diminishes as its size is reduced or its condition degrades, which lessens its likelihood of being representative of the source material.

[5] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.