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An American National Standard

Standard Guide for Using Scanning Electron Microscopy/Energy Dispersive X-Ray Spectroscopy (SEM/EDS) in Forensic Polymer Examinations[1]

This standard is issued under the fixed designation E2809; 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 guide covers recommended techniques and procedures intended for use by forensic laboratory personnel that perform SEM/EDS analyses on polymer samples.

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

1.3 The theoretical aspects of many of the topics presented can be found in texts such as Scanning Electron Microscopy and X-ray Microanalysis (**1**).[2]

1.4 This guide is intended to be applied within the scope of a broader analytical scheme (for example, Guides E1610, E3260) for the forensic analysis of a polymer sample. An SEM/EDS analysis can provide additional information regarding the potential relationships between the sources of polymeric materials.

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

1.6 The values stated in SI units are to be regarded as standard. Other units of measurement are included in this standard where applicable as a result of common usage (for example, keV).

1.7 *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.*

1.8 *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

E766 Practice for Calibrating the Magnification of a Scanning Electron Microscope

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

E2937 Guide for Using Infrared Spectroscopy in Forensic Paint Examinations

E3085 Guide for Fourier Transform Infrared Spectroscopy in Forensic Tape Examinations

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

3. Terminology

3.1 *Definitions*—For additional terms commonly employed for general forensic examinations, see Terminology E1732.

3.1.1 *aperture, n*—a beam-restricting orifice in an electron optical column; the orifice diameter influences the beam current and depth of focus.

[1] This guide is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and are 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.

3.1.2 *backscattered electron (BE) imaging, n*—a technique that uses high energy electrons that originate from the primary electron beam of the SEM and are elastically reflected by the specimen to create an image of the sample. The probability of backscattering is proportional to atomic number.

3.1.3 *cathodoluminescence, n*—emission of photons in the ultraviolet (UV), visible (Vis), and infrared (IR) regions of the electromagnetic spectrum as a result of electron beam interaction with certain materials.

3.1.4 *charging, n*—negative charge accumulation on either a nonconductive sample or a sample that is not properly grounded.

3.1.4.1 *Discussion*—This effect can interfere with image formation and X-ray analysis because of beam deflection. It can usually be eliminated by the application of a conductive coating or by the use of a low vacuum system.

3.1.5 *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.

3.1.6 *energy dispersive X-ray spectroscopy (EDS, EDXA, EDX), n*—X-ray spectroscopy based on the simultaneous measurement of the energies of X-rays emitted by a sample.

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

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

3.1.8 *exclusionary difference, n*—a difference in a feature or property between compared items that is substantial enough to determine that they did not originate from the same source.

3.1.9 *live time, n*—the time over which the energy dispersive X-ray spectroscopy electronics are available to accept and process incoming X-rays. Live time is often expressed as a percentage of real time.

3.1.10 *microtomy, n*—sample preparation approach that sequentially passes a blade at a shallow depth through a sample resulting in sections of selected thickness as well as a flat block.

3.1.11 *pulse processor time, n*—operator-selected value for the time designated to record a response by the detector.

3.1.11.1 *Discussion*—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.

3.1.12 *raster, n*—the pattern scanned by the electron beam on a sample; the raster dimensions change inversely with magnification.

3.1.13 *sample (representative sample), n*—a representative portion of the specimen selected and prepared for analysis that is expected to exhibit all of the elemental characteristics of the parent specimen.

3.1.14 *scanning electron microscopy (SEM), n*—a type of electron microscope in which a focused electron beam is scanned in a raster on a solid sample surface; the term can also include the analytical technique of energy dispersive X-ray spectroscopy.

3.1.15 *secondary electron (SE) imaging, n*—imaging using low-energy electrons produced from the interaction of beam electrons and conduction band electrons of atoms within the interaction volume, with only those near the surface having sufficient energy to escape.

3.1.16 *spectral artifacts, n*—spectral peaks other than characteristic peaks, produced during the energy dispersive X-ray spectroscopy detection process; examples include escape peaks and sum peaks.

3.1.17 *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).

3.1.18 *sum peak, n*—a peak resulting from the simultaneous detection of two photons; this is manifested as a peak at the combined energy of line(s) for the specific element(s) involved.

3.1.19 *system peaks (stray radiation), n*—peaks that can occur in the X-ray spectrum as a result of interaction of the electron beam or fluorescent radiation with components of the scanning electron microscope itself.

3.1.20 *variable pressure mode, n*—mode that allows some SEMs to operate at varying chamber pressures.

3.1.20.1 *Discussion*—The need for application of a conductive coating is minimized when using variable pressure mode; however, EDS can be complicated because of the electron beam spread experienced at higher operating pressures.

4. Significance and Use

4.1 This guide is intended to advise and assist the analyst in the preparation of polymer samples (for example, paint and tape) for SEM/EDS, the collection of data by SEM/EDS, and the interpretation of images and data resulting from these analyses.

4.2 When polymers are constructed as layered materials, SEM/EDS analysis is conducted on each polymeric layer individually. This analysis can be hindered by a non-discernable layer structure (for example, smear, irregular segregation within the layer system).

4.3 SEM-EDS data can be useful in:

4.3.1 *Layer Elucidation*—SEM images provide insight into the layer structure of a sample.

4.3.2 *Texture Elucidation*—SEM images and elemental maps provide insight into the texture (for example, surface topography, distribution of inclusions).

4.3.3 *Element Identification*—Determination of the elements detected in a sample layer.

4.3.4 *Relative Elemental Abundance Determination*—An EDS spectrum permits the relative abundance of elements in samples to be compared.