Guideline for Using Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy in Forensic Tape Examinations

Scientific Working Group on Materials Analysis (SWGMAT)

Scope │Reference Documents│Definitions │Significance and Use │Sample Handling │Sample Preparation │ Analytical Procedures │Documentation │References

1.0 Scope

This document is part of a series of SWGMAT guidelines relating to the forensic analysis of tape and is an outline of methods for scanning electron microscopy (SEM). This document is a version of SWGMAT's Standard Guide for Using Scanning Electron Microscopy/X-ray Spectrometry in Forensic Paint Examinations, with modifications regarding material-specific information. The methods employed by each examiner and/or laboratory depend on sample size, sample suitability, and laboratory equipment. The term scanning electron microscopy occasionally refers to the entire analytical system, including energy dispersive X-ray spectroscopy (EDS).

This guide is not intended to be an instruction book, nor will it apply in every situation. It is the responsibility of the analyst to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to the use of this technique.

2.0 Reference Documents

ASTM. *Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory*, ASTM E1492-05

Scientific Working Group on Materials Analysis. Guideline for the Forensic Examination of Pressure Sensitive Tapes, *Forensic Science Communications* [Online]. (October 2008). Available: www.fbi.gov/hq/lab/fsc/backissu/oct2008/standards/2008_10_standards02.htm

Scientific Working Group on Materials Analysis. Standard Guide for Using Scanning Electron Microscopy/X-ray Spectrometry in Forensic Paint Examinations, *Forensic Science Communications* [Online]. (October 2002). Available: www.fbi.gov/hq/lab/fsc/backissu/oct2002/bottrell.htm

Scientific Working Group on Materials Analysis. Trace evidence quality assurance guidelines, *Forensic Science Communications* [Online]. (January 2000). Available: www.fbi.gov/hq/lab/fsc/backissu/jan2000/swgmat.htm

Scientific Working Group on Materials Analysis. Trace evidence recovery guidelines, *Forensic Science Communications* [Online]. (October 1999). Available: www.fbi.gov/hq/lab/fsc/backissu/oct1999/trace.htm

3.0 Definitions

The terms defined relate specifically to SEM/EDS analysis as described in this document. General tape definitions can be found in the SWGMAT Guideline for the Forensic Examination of Pressure Sensitive Tapes.

Background X-rays (Bremsstrahlung, braking radiation, continuous spectrum): Nonspecific X-ray radiation with a continuous energy range from zero up to the beam voltage. Background radiation results from the deceleration of beam electrons in the atomic Coulombic field. A typical X-ray spectrum consists of both a continuous background and peaks from characteristic X-rays.

Backscattered electrons (BE): Primary beam electrons that are scattered from the sample after undergoing few inelastic interactions. The probability of backscattering is proportional to the atomic number.

Bulk analysis: A type of scanning electron microscopy analysis that determines the representative elemental composition of a material. The area of analysis is as large as possible and may be achieved by a single large area raster or the summed results from multiple smaller area rasters.

Characteristic X-rays: X-ray emission resulting from de-excitation of an atom following inner shell ionization. The energy of the X-rays is related to the atomic number of the atom, providing the basis for energy dispersive X-ray spectroscopy. A typical X-ray spectrum consists of both a continuous background and peaks from characteristic X-rays.

Charging: Negative charge accumulation on either a nonconductive sample or a sample that is not properly grounded. This effect may 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.

Concentration: For the purpose of this guide, the following ranges shall apply: Major: greater than 10 percent; Minor: 1 to 10 percent; Trace: less than 1 percent.

Energy dispersive X-ray spectroscopy (EDS, EDXA, EDX): X-ray spectroscopy based on the measurement of the energy of X-rays. Energy dispersive X-ray spectroscopy is a complementary spectroscopy to wavelength dispersive spectroscopy.

Escape peak: A peak resulting from incomplete deposition of the energy of an X-ray entering the energy dispersive X-ray spectrometer detector. 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 (1.74KeV). The escape peak intensity is about 1-2 percent of the parent peak.

Extraneous material (contaminant, foreign material): Material originating from a source other than the specimen.

Final aperture: The last beam-restricting orifice in an electron optical column. The orifice diameter influences the beam current and depth of focus.

Interaction volume: The sample volume in which the electron beam loses most of its energy. It is generally thought of as the volume in which detectable X-rays are produced. The actual volume varies depending upon beam voltage, average atomic number, and density of the sample.

KLM reference lines: The energies associated with the transitions of the K, L and M shell electrons. Each element will have a different series of KLM reference lines.

Live time: 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.

Particle analysis: An analytical method intended to determine the elemental composition of a single particle such as a pigment particle in an adhesive. Usually performed with a static (non-scanning) electron beam.

Pulse processor time constant: Operator-selected value for pulse-processing time. 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.

Raster: The rectangular pattern scanned by the electron beam on a sample. The raster dimensions change inversely with magnification.

Sample (representative sample): A representative portion of the specimen selected and prepared for analysis that is believed to exhibit all of the elemental characteristics of the parent specimen.

Scanning electron microscopy (SEM): A type of electron microscope in which a focused electron beam is scanned in a raster on a solid sample surface. The strength of resulting emissions of signals varies according to sample characteristics such as composition or topography. These signals directly modulate the intensity of the display cathode ray tube. The electron beam of the scanning electron microscope and the display cathode ray tube are scanned synchronously, resulting in a two-dimensional image of the sample. By popular usage, the term scanning electron microscopy may also include the analytical techniques energy dispersive X-ray spectroscopy and wavelength dispersive X-ray spectroscopy.

Secondary electrons (SE): Low-energy electrons produced from the interaction of beam electrons and conduction band electrons of atoms within the interaction volume. They are produced throughout the interaction volume, but only those near the surface have enough energy to escape. The secondary electron signal is typically used to form topographic images.

Specimen: Material submitted for examination. Samples are removed from a specimen for analysis.

Spectral artifacts: Spectral peaks other than characteristic peaks, produced during the energy dispersive X-ray spectroscopy detection process. Examples include escape peaks and sum peaks.

Spectral resolution: A measure of the ability to distinguish between adjacent peaks in an X-ray spectrum. It is usually determined by measuring peak width at half the maximum value of the peak height or full-width-half-maximum.

Sum peak: 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.

System dead time: The time during which the energy dispersive X-ray spectrometer is not able to process X-rays. Dead time is typically expressed as a percentage of real time.

System peaks (stray radiation): Peaks that may occur in the X-ray spectrum resulting from interaction of the electron beam or fluorescent radiation with components of the scanning electron microscope itself.

Take-off angle: Angle between the specimen surface and the detector axis.

Thick section: For the purposes of this guide, a sample that is two micrometers or thicker.

Thin section: For the purposes of this guide, a sample with a thickness of less than two micrometers.

4.0 Significance and Use

4.1. The scanning electron microscope is one component of the analytical scheme of the forensic analysis of tape and can be used to define the bulk elemental composition of individual tape components (backing and adhesive) and the elemental composition of individual particulate components within tapes, as well as the surface morphology.

4.2. The methods described in this guide may have some limitations. These may include the inability to detect elements in trace concentrations, the need for a conductive coating of the sample (with a high vacuum SEM), and the discoloration of materials by irradiation.

4.3. Although quantitative and semiquantitative methods are available for energy dispersive X-ray spectroscopy, they are not appropriate for most tape analyses because of the typical condition of the tape.

4.4. The information available from a heterogeneous specimen may diminish as its size is reduced and its condition degrades. The smaller a specimen, the less valuable it may become for an association. As sample size is reduced, it may no longer be representative of the original material. This may also be true of a degraded specimen.

4.5. This guide is intended to advise and assist laboratory analysts in the effective application of SEM to the analysis of tape evidence.

4.6. It is not the intention of this guide to present comprehensive methods of scanning electron microscopy. It is necessary that the analyst have an understanding of scanning electron microscope operation and general concepts of specimen preparation prior to using this guide.

5.0 Sample Handling

5.1. The general collection, handling, and tracking of samples shall meet or exceed the requirements of ASTM 1492-05 as well as the relevant portions of the SWGMAT's Trace Evidence Quality Assurance Guidelines and Trace Evidence Recovery Guidelines.

5.2. The work area and tools used for the preparation of samples should be free of all materials that could transfer to the sample.

5.3. When samples are prepared for scanning electron microscopy, a map identifying sample location on an SEM mount can be constructed to assist in locating the sample when performing the analysis.

6.0Sample Preparation

6.1. Samples should first be examined with a stereomicroscope, noting size, color, structure, and any extraneous material adhering to the sample.

6.2. The choice of a specific method for sample preparation depends on the size, nature, and condition of the specimen, as well as the analytical objective. It may be necessary to use multiple preparation methods in order to analyze all sample characteristics.

6.3. In developing a strategy for analysis, the following should be considered:

- determination of the presence of extraneous materials and a strategy for removal
- method of attachment to a scanning electron microscopy mount
- method(s) for producing a uniform geometry
- the need for a conductive coating on the prepared samples
- determination of the presence of surface features of analytical interest

6.3.1. If the analytical objective is to determine elemental composition, then any possible contribution from extraneous materials should be eliminated or accounted for.

6.3.2. For the accurate comparison of elemental composition and structure, samples should be prepared in the same manner.

6.4. Recognition and removal of extraneous materials

6.4.1 It is not unusual for extraneous materials to be present on the surface of a specimen submitted for analysis, particularly on an adhesive component. Because the scanning electron microscopy method is a surface analysis, the presence of even a small amount of this material can prevent an accurate determination and comparison of composition. Therefore, recognition and removal or abatement of this material should be performed.

6.4.2. Depending on sample size and type, extraneous material may be physically removed with a brush, probe, or fine blade. Debris can also be removed from the backing surface with methanol and a cotton swab or lifted off the backing with office tape. Care should be taken that the adhesive from the office tape does not adhere to the sample surface, which might interfere with any subsequent organic or inorganic analysis. If necessary, a fresh surface may be exposed by scraping or cutting with a fine scalpel blade.

6.4.3. When extraneous materials cannot be removed, these materials should be avoided during analysis

6.5. Methods of attaching samples to SEM mounts

6.5.1 All samples to be analyzed in the scanning electron microscope should be attached to an appropriate SEM mount. Because the presence of a carbon peak in the spectrum does not usually interfere with elemental comparisons, mounts constructed of carbon are preferred.

6.5.1.1. The adhesive should be removed from the backing to ensure no contribution from the backing is in the resulting spectrum. The tape's adhesive is smeared directly onto the surface of the mount.

6.5.1.2 A backing may be attached directly to a mount using the tape's own adhesive. Contribution of the adhesive in the resulting spectrum is typically not a concern during backing analysis. If the backing has been separated from the adhesive or if a cross-section of the backing has been prepared, the backing or cross-section can be attached to the mount with a mounting adhesive. This mounting adhesive may be applied as a liquid or as a double-sided tape. If the tape is known to be multi-layered, both the outer and the adhesive side may be analyzed.

6.5.2. The geometry of each sample, including flatness and take-off angle, should be similar. Often, a backing can be pressed flat with clean glass in order to remove irregularities.

6.5.3. Generally it is necessary to apply a conductive layer to the sample surface to eliminate charging. Carbon is preferred, because the presence of a carbon peak in the spectrum usually does not interfere with elemental comparisons. The use of a variable pressure instrument may also eliminate charging.

7.0 Analytical Procedures

7.1. Instrument calibration

7.1.1. Prior to beginning an analysis, verification of the operational condition of the scanning electron microscope should be established. This may include the presence of system peaks, accuracy of magnification, and determination of spectral energy calibration and resolution.

7.1.2. The presence of system peaks is generally determined upon installation of the scanning electron microscope or following a modification or addition of accessories.

7.1.3. For a determination of accuracy of magnification, a percentage of error of magnification should be calculated. A scanning electron microscope's indicated value of magnification (such as a measurement marker) is compared to a measurement of a certified standard (such as NIST SRM 484D). A calibration check of the primary image output device to the certified standard should be performed periodically and a record kept in a permanent log. Relationships of measurements on display monitors, as well as any other image capture applications to the primary image output device, should also be recorded. Magnification standards for scanning electron microscopes are commercially available, with errors of less than five percent generally achievable.

7.1.4. Energy calibration should be established frequently for the energy dispersive X-ray spectrometer, including zero offset and gain, and a record kept in a permanent log. Energy calibration may be determined directly by measuring the centroid energy of a low- and high-energy peak or determined automatically using software provided by the instrument manufacturer. If automated methods are used, measured spectral energies typically do not deviate more than 10eV from that of actual energies. Automatic methods for calibration are described in documentation from the manufacturer.

7.1.5. Spectral resolution for the energy dispersive X-ray spectrometer may be determined regularly and a record kept in a permanent log. This may be determined automatically or can be determined manually by measuring the width of the Mn K-α peak (or other suitable metal) at half the maximum peak height. Automatic methods for calibration as well as recommended performance limits are often available from the manufacturer.

7.1.6. EDS system performance can also be evaluated by comparison (overlay) of system checks to a suitable reference spectrum.

7.2. Structural imaging

7.2.1 SEM imaging of pressure-sensitive tape backings at moderate magnifications (75-250X) yields structural information complementary to that of traditional light microscopical methods. It can be used to image very small striations, craters, and surface features on the backings of polymer-based tapes, such as black electrical tape and duct tape. It can be used to view the paper fibers in masking tapes, as well as show the cross-sectional structure of each of these tapes.

7.2.2 . A backscattered electron image is useful for defining structures based on the average atomic number of the matrix. Structures containing elements with higher atomic numbers will generally appear brighter than those with lower atomic numbers. This is often useful for evaluating homogeneity and layer structure.

7.2.3. SEM micrographs should include a measuring scale or magnification scale or both. The micrograph should also display which signal (backscattered electron or secondary electron) was used to produce the image.

7.3. Selection of scanning electron microscopy/energy dispersive X-ray spectroscopy operating conditions

7.3.1. The following suggested operating conditions are meant as general guides for starting conditions. As the analyst determines specific analytical needs, actual operating conditions may vary.

7.3.1.1. A beam voltage of 20-30 KeV is an adequate compromise between the need for sufficient over-voltage necessary for efficient X-ray excitation and X-ray spatial resolution. Most of the X-ray lines produced may be displayed with an energy range of 0 to 20KeV. The pulse processor time constant should be set at a midrange value, which is a compromise between maximum count rate and maximum spectral resolution. The beam current should be adjusted to yield an X-ray detector dead time of approximately 30 percent. A live time of 100-200 seconds is usually sufficient to provide reasonable counting statistics for minor peaks.

7.3.1.2. Generally, changes in the suggested initial conditions are required under the following circumstances:

- Beam voltage is increased when higher energy line excitation is required.
- Beam voltage is decreased when greater spatial resolution is required.
- Pulse processor time constant is lengthened when greater spectral resolution is required.
- Pulse processor time constant is shortened when a greater count rate is required (e.g., for trace element analysis or construction of elemental distribution maps).
- Detector to sample distance can be reduced to increase X-ray collection efficiency.
- Spectral energy display scale is expanded when sufficient detail is not evident.
- Beam current is increased when the X-ray count rate is too low. Decreasing the condenser lens current and/or increasing the final aperture size may increase beam current.
- Beam current is decreased when the X-ray count rate is too high. Increasing the condenser lens current and/or decreasing the final aperture size may decrease beam current.

7.4. Bulk spectra collection

7.4.1. Once heterogeneity of the material is evaluated, a spectrum of the average (bulk) elemental composition of the sample is obtained. The raster should include as much area of the sample as possible. Analyzing a single large area or summing the spectra from several smaller areas may achieve this.

7.4.2. When comparing samples, all data and micrographs should be collected in the same manner with the same conditions.

7.5. Qualitative analysis

7.5.1. Once an X-ray spectrum is collected, a qualitative analysis is performed in order to determine the elements present. The process is straightforward for the peaks of elements present in major amounts and those not overlapping. Misidentifications or omissions of minor components are possible unless a systematic approach to elemental identification is used which includes consideration of X-ray line families, spectral artifacts, escape peaks, sum peaks, and overlaps.

7.5.2. Reference lines, or energies, may be obtained from several sources, including energy slide rules, published tables, and computer-generated KLM reference lines that may be superimposed on the spectrum. Additionally, manufacturers often provide an automatic element identification application. These aids often are used in complementary fashion.

7.5.3. Identification begins with high-energy peaks and major peaks. High-energy peaks are generally less likely to overlap than lower energy peaks. If a major peak is present, generally a complete family of peaks can also be identified. Each line within the family is labeled with elemental symbols. Spectral artifacts, including sum peaks and escape peaks associated with major peaks, should be evaluated and labeled.

7.5.4. As spectral interpretation alternates between the identification of major and minor peaks, the vertical (counts) scale should be adjusted to reveal required detail. In addition to the higher energy peaks, the presence of any lower energy families and their expected relative intensities should be noted. Individual asymmetric peaks and inconsistent peak ratios within a family may indicate a peak overlap. Superimposing and scaling KLM reference lines on the spectrum or referencing the actual spectrum of an elemental standard aids elemental identification. The analyst should be familiar with the characteristic pattern and relative intensities of peaks of various atomic numbers. The identification of major elements is usually straightforward.

7.5.5. Following the identification of major elements, lower intensity peaks and overlapped peaks are identified. The limited number of characteristic peaks present for minor elements can limit their identification.

7.5.6. The presence of an element can be considered unequivocal only when a distinctive, unique set of lines is produced or when a single peak occurs at an energy where it cannot be mistaken for another element or spectral artifact. Unequivocal identification may not be possible if an element is present in low concentration or if lines required for confirmation are overlapped with the lines of other elements.

7.5.7. If identification is unequivocal, each individual peak is labeled with the corresponding elemental symbols (and X-ray line if the software permits). If the identification is probable but not absolute, the peak label should so indicate (such as by parenthesizing the elemental symbols).

7.5.8. Spectra should be displayed on a scale that clearly demonstrates the peaks identified. In order to display peaks from elements with significant differences in concentration, the peaks from the elements in low concentration may be viewed by displaying the spectra separately on different display scales.

7.5.9. If an automatic identification application is used, the analyst should confirm the resulting element identifications.

7.5.10. There may be an overlap of peaks in the energy dispersive X-ray spectroscopy spectrum of materials containing several elements. Some commonly occurring overlaps encountered in energy dispersive X-ray spectroscopy are as follows: Ti K β/V K-α, V K-β/Cr K-α, Cr K-β/Mn K-α, Mn K-β/Fe K-α, Fe K-β/Co K-α, Pb M-α/S K- α/Mo L-α, Ba L-α/Ti K-α, K K-β/Ca K-α, Zn L-α/Na K-α, P K-α/Zr L-α, and Al K-α/Br L-α.

In order to resolve these overlaps, several methods may be employed.

- The live time count can be increased.
- The processing time of the pulse processor may be increased to improve spectral resolution.
- Mathematical spectral subtraction (deconvolution) methods supplied by the energy dispersive X-ray spectrometer manufacturer can be employed.
- An alternative method of elemental analysis or X-ray diffraction may be used.

7.6. Individual component analysis

7.6.1. Additional evaluation of composition may be achieved by the spot (nonrastered) analysis of specific particles within layers. Generally, these particles appear bright in the backscattered electron image. Such an analysis may improve the detection limit beyond that achievable by a bulk analysis, as well as serve to associate elements detected by a bulk analysis. For example, the bulk analysis of a tape adhesive may reveal the presence of Al, Si, Mg, and O. Specific particle analysis may associate the elements Si, Mg, and O as being present in one type of particle, and Al, Si, and O in a second type. These associated elemental compositions would then indicate these particles could be talc and kaolinite, respectively. Polarized light microscopy, infrared spectroscopy, or X-ray diffractometry can be used to confirm the presence of some of the compounds.

7.6.2. Because the beam interaction volume may be considerably larger than an individual particle, inclusion of other matrix components may be expected in the spectrum from an individual particle. Lower beam voltages may be used to confine more of the interaction volume to the particle. It should be noted, however, that the use of lower beam voltages may result in the loss of characteristic lines that may be found at higher energies.

7.7. Analysis of a primarily organic matrix

7.7.1. Analysis of a substance that is primarily organic (e.g., duct tape backing, clear electrical tape adhesive) may be useful. Within such a matrix, the interaction volume is significantly larger than that of a substance that is primarily inorganic. This is a result of a lower average atomic number of the matrix. In order to reduce the interaction volume, the beam voltage may be reduced; however, the voltage should be sufficient to produce X-rays from all lines of analytical interest. Charging may also be an issue with such samples. Therefore, precautions may be taken to prevent this from occurring (e.g., sample coating or operation at low vacuum).

7.7.2. Because an organic matrix may contain small amounts of some elements, the counting time should be extended.

7.8. Heterogeneity versus analytical area

7.8.1. In order to compare the average composition of structures, the spectrum used for comparison should come from an area of the structure sufficient to produce representative composition.

7.8.2. The representative nature of a spectrum can be determined by the critical comparison of spectra from adjacent areas. If no differences are evident, the sampled area is homogeneous at that magnification. A representative bulk analysis can be achieved by rastering the beam across as large an area as the sample permits.

7.9. Assessment of results

7.9.1. Generally, comparisons are facilitated by direct spectral comparison.

7.9.2. If spectral differences are not detected, it is likely that the materials are similar in elemental composition.

7.9.3. If spectral differences are detected, it is likely that the materials are not similar in composition; however, several alternative explanations are possible and should be evaluated. Only after the considerations in Sections 7.8.1 through 7.8.2 have been addressed can the analyst confirm that spectral differences are indicative of compositional differences.

7.9.3.1. Differences in background shape may result from dissimilar sample geometry. To correct, see Section 6.5.2.

7.9.3.2. Differences in the composition of major peaks may indicate that the spectra are not representative of the bulk composition of a heterogeneous sample. This could occur as a result of the analysis of a sample too small to be representative or the analysis of a raster area too small to be representative.

7.9.3.3. If there are no differences in major peak ratios, differences in minor/trace components may result from the presence of extraneous materials. If the sample was a fragment or unable to be cleaned, a small amount of foreign material may have been present during the analysis. Consequently, some of the minor elemental peaks in the spectrum may have been produced from elements in the extraneous material. To correct, see Section 6.4.

7.9.3.4. Differences in carbon intensity may result from a contribution of carbon from the mount if the sample is very small. Furthermore, the presence of carbon, oxygen, and nitrogen in the tape matrix limits the usefulness of these elements in direct spectral comparison; therefore, they are typically not evaluated.

7.10. Interpretation of scanning electron microscopy/energy dispersive X-ray spectroscopy data

7.10.1. A conclusion regarding similarity results from the comparison of images and elemental composition of individual layers. Spectra may be critically compared by overlaying them.

7.10.2. If a comparative analysis did not demonstrate significant differences, then no differences were indicated in structure and composition within the limits of the analytical capability of scanning electron microscopy/energy dispersive X-ray spectroscopy.

7.10.3. If ratio differences between peaks exist, it can be concluded that these differences may result from either actual differences in the bulk composition of the materials or from the analysis of a small sample (or area) whose chemistry is not representative of the bulk composition of a heterogeneous sample. The latter should only be concluded following an extensive investigation of the heterogeneity of the known samples and demonstration that the range of variation present in one sample encompasses that observed in the other sample.

7.10.4. If there are no differences in major peak ratios but there are differences in minor/trace peaks, it can be concluded that no differences in major elemental constituents are indicated, although some differences in the bulk composition are evident. For example, if the specimen was a fragment and unable to be adequately cleaned, a small amount of foreign material may have been present during the analysis. Consequently, some of the minor elemental peaks present in the spectrum may have been produced from elements in the foreign material and not from elements in the questioned material. Equally so, the observed differences may be due to actual

differences in the composition of the samples. Therefore, with respect to the elemental composition of these samples, an inconclusive result for this technique is indicated.

7.10.5. If, after taking into consideration Section 7.9.3, a comparative analysis demonstrates significant differences between samples regarding structure and composition, it then can be concluded that the samples are different.

8.0 Documentation

8.1. Case notes should include a copy of all of the instrumental data that was used to reach a conclusion. All hard copies should include a unique sample designation, the operator's name/initials, and the date of analysis.

8.2. Case notes should also include a description of the evidence analyzed by SEM, the method of sample preparation, the analytical instrumentation used, and its operating parameters. The case notes should also include a statement or data confirming system calibration, as detailed in Section 7.1.

8.3. See SWGMAT's Trace Evidence Quality Assurance Guidelines for further requirements.

9.0 References

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