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Standard Practice for the Forensic Analysis of Geological Materials by Scanning Electron Microscopy and Energy Dispersive X-Ray Spectrometry

Trace Materials Subcommittee
Trace Evidence Scientific Area Committee (SAC)
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OSAC Proposed Standard

OSAC 2024-S-0012
Standard Practice for the Forensic Analysis of Geological Materials by Scanning Electron Microscopy and Energy Dispersive X-Ray Spectrometry

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Standard Practice for the Forensic Analysis of Geological Materials by Scanning Electron Microscopy and Energy Dispersive X-Ray Spectrometry

1. Scope

1.1 This practice covers recommended techniques and procedures for the use of Scanning Electron Microscopy/Energy Dispersive X-ray Spectrometry (SEM/EDS) for the forensic analysis of geological materials to include soils, rocks, sediments, and materials derived from them (for example, concrete).

1.2 The theoretical foundation of SEM/EDS is covered in numerous texts such as Scanning Electron Microscopy and X-ray Microanalysis (**1**). This document describes sample handling and preparation, instrument operating conditions, spectral data collection, evaluation of EDS data quality, interpretation of EDS spectra for the identification of inorganic geological materials, documentation of morphology by SEM imaging, and criteria for sample comparison.

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

1.4 The values stated in SI units are to be regarded as standard. Other units are avoided, but photon energy is commonly reported in units of kilo electron volts (keV), data are collected as counts per second (cps), and data are reported in elemental weight percent (wt. %).

1.5 This standard is not intended for the characterization of building materials by SEM/EDS to assess engineering properties. These are provided in the following (C1723, C295, C856).

1.6 This standard does not purport to address all 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*

C1723 Guide for Examination of Hardened Concrete Using Scanning Electron Microscopy
C295 Guide for Petrographic Examination of Aggregates for Concrete
C856 Practice for Petrographic Examination of Hardened Concrete
E620 Practice for Reporting Opinions of Scientific or Technical Experts
E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory
E1508 Guide for Quantitative Analysis by Energy Dispersive Spectrometry
E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Programs
E1732 Terminology Relating to Forensic Science

E766 Practice for Calibrating the Magnification of a Scanning Electron Microscope

E3272 Guide for Collection of Soils and Other Geological Evidence for Criminal Forensic Applications

E3254 Practice for Use of Color in the Visual Examination and Forensic Comparison of Soil Samples

2.2 *ISO Standards*

ISO/IEC 1702:2017 General requirements for the competence of testing and calibration laboratories.

ISO 22309:2011 Microbeam analysis — Quantitative analysis using energy-dispersive spectrometry (EDS) for elements with an atomic number of 11 (Na) or above.

3. Terminology

3.1 *Definitions* – for additional terms commonly employed for general forensic examinations and SEM/EDS, see Terminology in E1732 and E1508 respectively.

3.1.1 *bulk analysis*, n - analysis of a material either by a large single area or by compiled results of multiple areas, typically intended to capture a representative property of the material.

3.1.2 *compositional domain*, n – compositionally distinct region of a particle.

3.1.3 *morphotype*, n - recognized subdivision of particles that is characterized by distinct morphological characteristics.

3.1.4 *morphometrics*, n - quantitative measurements of morphological features that can be used to characterize particles.

3.1.5 *overscan*, n – an EDS method in which the electron beam is rastered over a region of interest to obtain a bulk elemental analysis.

3.1.5.1 *Discussion*: The area of analysis should be as large as possible and is achieved by a single large area raster or the summed results from multiple smaller rastered areas.

3.1.6 *Spot analysis*, n- an EDS mode in which a spectrum is collected when the electron beam is held static.

3.1.6.1 *Discussion*: The method can also be referred to by the various iterations of spot and point as well as mode and analysis. This type of analysis is the most common EDS method for particle analysis.

3.1.7 *standard*, n – a material with a known composition.

3.1.8 *variable pressure scanning electron microscope*, n - type of SEM that is designed to operate at higher chamber pressure than the conventional SEM.

3.1.8.1 *Discussion*: There are several alternative terms and abbreviations for variable pressure scanning electron microscope including: VP-SEM, ESEM or environmental SEM; low vacuum SEM; CP-SEM or controlled pressure SEM.

4. Significance and Use

4.1 This guide is intended to advise and assist analysts in the effective application of SEM/EDS to the analysis of geological materials. It is intended to be applicable to most modern SEM/EDS systems typically available in the forensic laboratory.

4.2 SEM/EDS can be used to determine the elemental composition of individual component particles with high spatial resolution (sub-micrometer) and the bulk elemental composition of the clay-sized fraction from soils submitted for forensic examination. Some references use SEM/EDX, SEM/EDXA, or SEM/EDAX as synonyms of SEM/EDS.

4.3 SEM/EDS is advantageous because it allows for the simultaneous imaging of individual particles to determine morphology and the measurement of the elemental composition of particles or discrete domains within particles.

4.4 Qualitative or semi-quantitative EDS analysis can be used to confirm provisional mineral identifications made using other techniques (e.g., polarized light microscopy [PLM], Raman spectroscopy).

4.5 Imaging by SEM can provide additional information enabling confirmation of provisional mineral identifications.

4.6 *Limitations to SEM/EDS:*

4.6.1 Detection limits depend on elements and matrices, and are typically around 0.1 weight % concentration for elements with atomic numbers above fluorine. For lighter elements, detection limits are typically around 1 weight % concentration. Alternative analytical techniques, such as inductively coupled plasma mass spectrometry, provide lower detection limits.

4.6.2 Some samples and SEM systems require covering the sample with a conductive coating.

4.6.3 Discoloration of materials can be caused by electron beam irradiation.

4.6.4 EDS provides elemental information, not structural information, preventing identification of polymorphs (*e.g.*, $CaCO_3$, Al_2SiO_5, TiO_2, and $KAlSi_3O_8$).

4.6.5 EDS data obtained from a region (zone) of a particle might not be representative of the whole particle.

4.6.6 Procedures for quantitative methods using EDS are not covered in this guide; they are available elsewhere (ASTM E1508).

4.7 The elemental compositions derived from SEM/EDS can be used to assign provisional mineral identifications.

Provisional mineral identity can be determined by visual comparison of sample EDS data to the following: reference EDS data published in mineralogy textbooks (**2**); empirically collected EDS data from known mineral standards; published mineral elemental composition data; or simulated EDS spectra (DTSA-II (**3**)).

4.8 The goals of a forensic examination of geological evidence include identification of an unknown substance, comparison of two or more items for possible common origin, or estimation of provenance. SEM/EDS analysis is a component of an overall examination scheme and is not typically used on its own (7.1.1).

The presence, absence, and relative abundance of mineral species or morphotypes determined by SEM/EDS in two samples can be used to evaluate whether there are exclusionary differences during a forensic comparison.

5. Sample Preparation

5.1 *General considerations*

5.1.1 Use sample preparation practices that minimize the possibility of cross-contamination. Prepare questioned and known items on separate SEM sample mounts (e.g., stubs).

Note 1 - Approaches for minimizing cross contamination include: preparing questioned and known samples in separate locations, storing prepared samples within covered containers to minimize dust accumulation, and placing questioned and known items within the SEM/EDS instrument at separate times. Exposure of a sticky mount during sample preparation can serve as an air blank. The nature of the samples affects precautionary measures recommended to prevent cross contamination. Samples of limited quantity and small particle size not embedded in mounting material require the greatest measures to mitigate potential cross contamination.

5.1.2 When preparing subsamples of particulate material, use procedures that create representative subsamples.

5.1.2.1 Sample splitters, or cone and quartering (**4**) can be used to create representative subsamples, but both can be impractical for samples of limited quantity.

5.1.2.2 An alternative method of representative sub-sampling appropriate for small quantities of powder, is first to mix the particles, moistening (with an appropriate liquid such as water) to cause particle adhesion, and then selecting one or more sub-samples for analysis.

5.1.3 Document the location of the materials to be analyzed on the SEM mount when more than one sample or subsample is placed on the same mount. Documentation methods can include sketches, photographs, captured video images, or index (fiducial) marks on the mount. Label samples with clear and unique codes/sample numbers.

5.1.4 Sample preparation is commonly carried out with the aid of a stereomicroscope. Once the SEM mounts are prepared, protect them from surface abrasion and atmospheric dust deposition with a protective lid. Storing the prepared samples in a vacuum chamber or desiccator reduces SEM chamber evacuation time.

5.1.5 A conductive coating reduces charging of the sample prior to SEM/EDS analysis. Uncoated samples can be analyzed using variable pressure mode (3.1.11) or using low accelerating voltage.

Note 2 - The width of the spot size often expands when using variable pressure conditions. An increased spot size could result in contributions to the EDS spectrum from the mounting material or other material that is in proximity. The wider spot size could also worsen image quality.

5.1.5.1 Sample coating is commonly accomplished using a vacuum evaporator or sputter coater (**1**).

Note 3 - Metal (e.g., gold, palladium, platinum) coatings can interfere with X-ray peak assignments (e.g., determination of phosphorus and zirconium) and ZAF correction (see E1508). If coating is required, carbon is preferred to metals when EDS analysis is performed.

5.1.6 If the intent of the analysis is the comparison of two or more samples, use the same sample preparation technique for all compared samples.

5.2 *Unpolished particles*

5.2.1 Geological particles can be prepared as unpolished grains to: study their morphology; determine their qualitative elemental composition; or minimize alteration of samples of limited quantity.

5.2.2 The choice of mounting substrate is dependent on the analyses. Conductive carbon tabs are a common mounting substrate, but recovering particles from them can be challenging. Particles adhered to polished beryllium or carbon mounts with organic binders (e.g., collodion) or conductive paint are easily recovered.

Note 4 - Collodion (nitrocellulose) can be dissolved in acetone.

5.2.3 Mineral grains can be mounted either untreated or washed to remove surface coatings or extraneous material prior to analysis. When grains are treated to remove extraneous materials, it is recommended to retain the extraneous material for potential subsequent analysis. Grains can be either individually mounted or applied as a dispersion.

5.2.3.1 Segregation of a sample component for SEM/EDS analysis, usually achieved by hand picking particles, is often used to determine the elemental composition of the component to confirm a provisional mineral identification.

The morphology of individual grains should be considered when orienting them for optimal imaging and X-ray analysis (6.2.3.2).

Note 5 - Back sieving of sand- or silt-sized grains (**4**) onto a sticky SEM stub enables sufficient spacing between the grains to minimize mixing of EDS spectra from distinct grains and minimizes the geometric effect of adjacent grains blocking signal from reaching the detector.

5.3 *Polished samples*

5.3.1 Polished samples can be prepared from bulk samples, lithic fragments, or individual grains to study grain morphology, internal texture, crystal zoning, or inclusions and to improve analysis of elemental composition.

5.3.2 Loose grains can be embedded in a mounting medium, typically epoxy, and polished.

5.3.3 EDS spectra collected from polished samples are more reproducible than EDS spectra from unpolished samples.

5.4 *Clay-sized fraction*

5.4.1 The clay-sized fraction is typically dispersed onto a substrate from a suspension. Dispersion onto a conductive substrate, or application of a conductive coating, will improve SEM imaging and EDS data quality (see 6.2.4). Due to particle size limitations the elemental analysis of clay-sized particles is commonly performed using the overscanning method.

Note 6 - The larger crystal size of diagenetic clay minerals allows identification by SEM/EDS, whereas the small mixed crystals found in soil clay minerals can prevent identification by SEM/EDS.

Note 7 - EDS data from the clay-sized fraction can be used together with other methods (e.g., X-ray diffraction, FT-IR, or staining) for identification of mineral phases present.

5.5 *In situ analysis of particles on items of evidence*

5.5.1 When isolation of particles from their substrate is not feasible due to risk of sample loss, or when the nature of the contact between sample and substrate is of interest, *in situ* analysis can be conducted (e.g., soil embedded in fabrics).

5.5.2 In many situations, it is necessary to excise portions of the substrate for placement in the SEM chamber.

5.5.3 Collect background EDS spectra of the questioned substrate to allow for the differentiation of the EDS signal of the adhering geological materials.

6. Instrumental Procedure

6.1 *SEM Imaging*

6.1.1 SEM calibration

6.1.1.1 Calibrate and conduct performance monitoring of the instrument following a schedule complying with the laboratory operating procedures. Important parameters include, but are not limited to, magnification, stage position and motion, electron source alignment, aperture alignment, backscatter and secondary electron detector signal-to-noise, and vacuum conditions (**5**).

6.1.2 Selection of Imaging Conditions

6.1.2.1 Select the analytical conditions for examination by SEM specific to case and sample. The analyst can choose to conduct specific analysis types (6.1.4.2-6.1.4.5) to meet the goals of the examination.

6.1.2.2 Select an electron imaging detector suitable for observation of the features of interest.

Note 8 - Secondary electron images (SEI) are collected to capture topographic and surface features. Off-axis backscattered electron (BSE) detectors also provide topographic information.

Note 9 - The grayscale intensity value of BSE images is proportional to the specimen average atomic number and can be used to assess compositional information.

6.1.2.3 Adjust the image brightness and contrast conditions to best observe the sample and features of interest.

6.1.2.4 Set the accelerating voltage to achieve performance sufficient for quality imaging.

6.1.2.5 Optimize the magnification, focus, and stigmation to observe the feature of interest.

6.1.2.6 For non-conductive materials, use low kV, variable pressure, a conductive coating, or a combination of these techniques to mitigate charging.

6.1.3 Image Acquisition

6.1.3.1 Collect sufficient images to record the features of interest within the sample.

6.1.4 Assessing morphological characteristics

6.1.4.1 For semi-quantitative morphometrics, confirm the instrument scale calibration.

6.1.4.2 *Particle surface texture examination* - Examine and document the surface features of grains; these features are typically compared to reference atlases (**5-8**) or to known samples.

Note 10 - Individual particles, typically >250 µm in diameter, are selected for surface texture analysis. To improve imaging, these grain mounts are routinely coated with a conductive material (e.g., carbon or gold).

Note 11 - The charging of particles due to limited contact to a conductive substrate can be reduced by using a conductive paint, commonly carbon or silver. However, this option can be supplanted using low vacuum (3.1.9) or low voltage imaging mode in systems with this capability.

Note 12 - The range of characteristics useful for surface texture analysis is beyond the scope of this document and can be found in references **5** and **6**; specific applications of quartz grain surface analysis for forensic purposes can be found in Morgan et al. (**7**).

6.1.4.3 *Particle shape examination* – Describe the morphology of particles based on: presence and nature of crystal faces, roundness (round to angular); and the relative grain dimensions (e.g., equant, elongated, bladed, platy (**9**))

Note 13 - Particle morphometric analysis can use a range of metrics and terminology.

6.1.4.4 *Particle size and sorting* - Determine the particle dimensions and distribution of particle sizes (sorting) by SEM imaging.

Note 14 - If during sample preparation the sample was size-fractionated then assessing particle size and sorting is not appropriate.

6.1.4.5 Document the morphology of microscopic particles of biological origin often found in soil (e.g., pollen, foraminifera, diatoms) by SEM imaging, to enable a level of taxonomic identification.

6.2 *Elemental Analysis*

6.2.1 *EDS Instrument Calibration*

6.2.1.1 Perform verification on a regular, documented schedule following instrument-specific requirements and after instrumental maintenance. This verification includes determination of energy calibration, energy resolution, and visual assessment of the Bremsstrahlung background shape (**10**).

Note 15 - A reference material (often pure metals such as copper or aluminum) can be used to verify peak positions for both low-energy (~1 keV) and high energy (~8 keV) peaks.

6.2.2 *Selection of EDS conditions*

6.2.2.1 Use an accelerating voltage sufficient to generate X-rays of interest. This value is typically 1.5 to 3 times the energy of the X-ray line for an element of interest. An accelerating voltage of 15 - 25 kV is often sufficient for efficient X-ray generation. Lower voltages will reduce the analytical volume and potential for sample damage.

Note 16 - If the SEM/EDS has a beryllium window, detection of light elements (i.e., fluorine and below) is not possible.

6.2.2.2 When L or M X-ray family lines are provisionally identified, increasing the accelerating voltage can confirm the presence of L or K X-ray family lines, thus aiding in element identification.

6.2.2.3 Adjust the beam current, deadtime, and pulse processor time constant to optimize X-ray counts or spectral resolution.

Note 17 - Generally, deadtimes between 10-15 % (for a silicon-drift detector) provide high throughput while minimizing spectral artifacts (e.g., sum and escape peaks).

Note 18 - The pulse processor time constant influences collection speed and spectral resolution. Long time constants achieve greater spectral resolution but result in lower count rates.

6.2.2.4 The working distance for the detector system is defined by the specific instrument configuration. Correctly adjusting the working distance achieves a take-off angle that optimizes X-ray collection by the EDS detector. Adjust the stage to position the specimen at the recommended working distance; refer to manufacturer recommendations.

6.2.2.5 Spectral acquisition times are sample- and task-dependent. Elements present at low concentrations or light elements (e.g., boron, nitrogen, fluorine) typically require longer acquisition times, higher beam current, lower accelerating voltage, or a combination of these parameters to improve detection.

6.2.3 *Acquisition* of elemental composition

6.2.3.1 EDS spectra can be collected for a variety of purposes, such as: characterization of individual particles or compositional domains within particles, overscanning large areas to survey the elements present in a sample, or mapping the distribution of the elements within a defined area.

Note 19 - Deflecting the beam from the center of the field of view can result in non-linear X-ray counts. This effect is greater at low magnification (**10**).

6.2.3.2 Point analysis of a material is achieved by deflecting a static beam to a spot of interest. For grains with significant topography, position the static beam on the top of the grain or on a side facing the detector to prevent the sample from blocking the emitted photon from reaching the detector (see 6.2.4.1).

6.2.3.3 Bulk analysis of a material is achieved by overscanning a selected region of interest. The degree of homogeneity of the material can be assessed by examining a backscattered electron image to select the size and location of the rastered area. BSE imaging will help to ensure analysis of single compositional domains. When analyzing a material at high accelerating voltages, signal contribution can come from phases/ domains at depth.

6.2.3.4 Overscanning (see 3.1.6) large portions of a sample when there are multiple phases/compositional domains present should only be used for qualitative elemental determination.

Note 20 - Overscanning is only recommended for fine-grained clay preparations.

6.2.4 *Quality* assessment of collected EDS spectra

6.2.4.1 During and upon completion of the acquisition of an EDS spectrum, assess the data quality for: the shape, intensity, and continuity of the background; peaks; and spectral artifacts (e.g., sum peaks, escape peaks). Gaps in the background or sudden breaks in the continuum are an indication of a poor-quality spectrum resulting from sample geometry (see 6.2.3.2).

6.2.4.2 Document artifact peak(s) or re-collect a spectrum under conditions that reduce or eliminate them. Unassigned or ambiguous peaks should be noted accordingly.

6.2.4.3 Once a quality spectrum is collected, X-ray peaks can be assigned to elements by comparison to published tables of elemental X-ray energies or with the assistance of software.

Note 21 - Modern instrument manufacturer software packages provide auto-identification of elements; if using auto-identification, confirm all peak assignments (**11**). Confirm the presence of multiple X-ray lines or check for the presence of higher energy X-ray family lines, when applicable. If only a single peak with low counts is assigned to an element, the element identification should be considered provisional. Provisional identifications can be confirmed by collection of spectra with a higher number of counts or by complementary techniques (e.g., X-ray fluorescence, inductively coupled plasma- mass spectrometry, electron microprobe).

Note 22 - Due to the energy resolution limits of EDS detectors (approximately 130 eV), there is the potential for overlap of X-ray peaks from multiple elements. Examples of elements encountered in the analysis of geological materials that have overlapping peaks include Pb-S-Mo (molybdenite-MoS_2, galena-PbS), Ba-Ti (benitoite-$BaTiSi_3O_9$), and Y-P (xenotime-YPO_4). For additional examples of peak overlap, see Goldstein et al. (**1**).

Note 23 - Element detection using EDS depends on several factors including the matrix in which the element of interest resides, accelerating voltage, beam current, count times, or atomic number. At typical operating conditions (15 kV, ~1 nanoampere of current, and 30 seconds live time), elements (> fluorine) can be detected at ~0.1 weight percent.

6.2.4.4 The use of EDS spectral databases or software for the synthesis of spectra is recommended to confirm peak identification.

6.2.5 Element quantitation is beyond the scope of this document. For guidance on quantitation, see Goldstein et al. (**1**).

6.2.6 Automated EDS-based particle categorization or identification

6.2.6.1 The automated detection of particles and their elemental compositions can be used to categorize and count particles. The relative abundance of general groups defined by composition can be used in the forensic characterization of geological materials (**12, 13**).

6.2.6.2 Specialized software for automated provisional mineral identification by SEM/EDS has been shown to be useful in examinations of geological material (**14-17**).

6.2.6.3 Automated particle categorization or identification provides both quantitative modal abundance information as well as morphological assessment of mineral grains.

6.2.6.4 Procedures for the use of automated mineral identification are beyond the scope of this document.

7. Interpretation

7.1 Reports derived from the forensic analysis of geological materials typically address identification of material (7.2), restriction of the possible geographic source area (7.5 provenance), and the comparison of two or more materials to determine if they could share a common source (7.4). SEM/EDS analysis of any portion of a sample of geological evidence can be included in a report to aid in these three goals.

7.1.1 SEM/EDS analysis is usually a component of an overall forensic examination of geological evidence and is not typically used on its own. Use of additional orthogonal methods for forensic examinations is highly recommended. For example, SEM/EDS analysis is commonly conducted following color evaluation (E3254) and detailed polarized light microscopy (PLM) examination in the scheme of forensic soil comparisons.

7.2 *Material identification*

7.2.1 In forensic applications, mineral identifications based solely on qualitative EDS analysis are typically considered provisional.

7.2.2 Provisional mineral identification based on qualitative EDS analysis is performed by visual inspection for the presence, absence, and relative peak area in a spectrum. The identification of a mineral group or species should be supported with the use of mineralogy reference materials, mineral database spectra, or synthesized EDS spectra (e.g., DTSA-II (**3**)).

Note 24 - EDS-based mineral identification and quantification are well established methods in widespread use in the Earth Sciences (**2, 17-21**). Validation of EDS-based mineral identification is instrument and sample specific. Studies have shown strong agreement between XRD and EDS based mineral identification for natural (**18**) and synthetic (**19**) mineral mixtures.

Note 25 - Take caution in provisional identification of minerals within particular groups that display significant solid-solution (some examples include: members of the feldspar, garnet, pyroxene, amphibole, and tourmaline groups).

Note 26 - Some compounds occur as polymorphs (minerals having the same chemical composition but different crystal structures). The identification of a particular polymorph (e.g., SiO_2, $CaCO_3$, Al_2SiO_5, TiO_2, and $KAlSi_3O_8$) is not possible by EDS alone. Differentiation of polymorphs can be achieved using PLM, X-ray diffraction (XRD), or Raman spectroscopy.

Note 27 - Some mineral varieties can be confidently identified by their EDS spectra alone (e.g., zircon, $ZrSiO_4$), but for other minerals EDS analysis alone is limited to provisional identification.

7.2.3 Element abundances calculated using standardless quantitation algorithms common in instrument software packages should be considered estimates and might not be accurate.

7.2.4 Mineral identifications can be confirmed with the use of an orthogonal instrumental technique (e.g., PLM, Raman spectroscopy, XRD, quantitative elemental analysis, or by distinctive morphology).

7.3 *Interpreting Morphology*

7.3.1 Morphological features including particle surface textures, grain coatings, grain shapes, particle size and sorting, the presence of crystal faces or cleavage, or the identification of microscopic biologically-derived particles (e.g., phytoliths, diatoms, foraminifera, and pollen) can be used in the interpretation of the geological history of a sample, as a basis of sample comparison (**5**,**6**,**9**), to confirm a provisional mineral identification (7.2.4), or to aid in provenance interpretations (7.5).

7.4 *Sample comparisons*

7.4.1 The goal of forensic comparisons is to determine whether two samples have exclusionary differences indicating distinct sources. Alternatively, the lack of exclusionary differences supports the proposition that two or more samples could share a common origin.

7.4.2 Samples with minimal differences - Two samples of geological material consisting of similar components in similar relative abundances could have been derived from a common source. If analysis by SEM/EDS supports the conclusion that two samples share similar components (morphological characteristics, provisional mineral identifications, and mineral elemental compositions) in similar relative abundances, then no exclusionary differences are detected.

7.4.2.1 EDS spectra of particles in two or more samples can be compared even if the identity of the particles is not known. For particles with similar sample preparation and data acquisition, spectral overlay can be used for comparisons, where the presence or absence of peaks, peak shapes, and relative intensities are all considered in the evaluation as to whether exclusionary differences exist between compared samples.

7.4.2.2 The identification of uncommon features, including minerals, morphologies, mineral compositions, or bulk elemental profiles within both compared samples, increases the probative value of the evidence. For example, soil mineral occurrence data can be used to substantiate the rarity of a given mineral, in general, or specifically within an area of interest.

Note 28 - Minerals that are rare, in general, might be common in the relevant areas of interest to the case. The local characteristics can be confirmed with more expansive known exemplars, consulting published data, or contacting experts with localized knowledge (e.g., regional geological surveys, industry, academic literature, and persons with local expertise).

7.4.3 Samples with differences - If SEM/EDS analysis indicates that there are significant differences with respect to relative abundance, variation, or the omission/addition of particle types (e.g., mineral types, grain morphologies, or chemical variants of the same mineral), or bulk elemental composition of the fine fraction, the analyst shall evaluate possible explanations for these differences.

7.4.3.1 Samples with explainable differences - Explainable differences can include: contamination or alteration of one of the samples due to the effects of fire, stomach acid, mixing or dissolution of soluble mineral phases (e.g., anhydrite/gypsum); task-relevant case information; transfer and persistence processes; sample size limitations; and the representativeness of the known exemplars with respect to time of collection or location. If there is no scientifically supported or logical explanation for the observed differences, then these would be considered exclusionary differences. Document the justification for the explainable differences.

7.4.3.2 Samples with exclusionary differences - Samples with differences detected by SEM/EDS that are not explainable are considered exclusionary differences and indicate that these geological materials were derived from distinct sources.

7.5 Provenance:

7.5.1 The mineralogy, mineral elemental composition, or morphology determined by SEM/EDS can be used to interpret likely and unlikely sources of the material. The methods of this interpretation are beyond the scope of this document. See Pirrie et al. (**14, 16**) for a description and methods.

8. Documentation

8.1 Documentation of geological materials examinations by SEM/EDS should include:

8.1.1 Sample preparation procedures (e.g., sieving, washing to remove grain coatings, density separation, conductive coating, embedding and polishing).

8.1.2 Instrumental conditions used: chamber vacuum conditions, working distance, magnification, beam current/ spot size, and accelerating voltage.

8.1.2.1 For imaging: detector, and a scale bar or field-of-view (FOV) dimension.

8.1.2.2 For EDS data: deadtime, time constant, and analytical collection interval (count time or defined total counts).

8.1.3 Information supporting the interpretations of mineral or material identification, provisional mineral or material identification, and references or databases used in identification (E620).

8.2 For comparisons, document rationale for determining differences that are explainable or exclusionary.

8.3 Documentation should allow a second analyst to understand and evaluate all the work performed, and independently interpret the data.

8.4 Refer to E1492, E620, and ISO 17025 for further guidance.

9. Keywords

9.1 Energy dispersive X-ray spectrometry; EDS; scanning electron microscopy; SEM; particle analysis; mineral identification; geological material; elemental composition; soil.

10. References

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