Geologically-Derived Materials Examinations

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1 INTRODUCTION

This document provides guidance for selecting/organizing an analytical scheme for identifying and comparing geologic materials (e.g., soil, rocks, minerals, gemstones), geologically-derived materials (e.g., bricks, concrete blocks, ceiling tile), and unknown materials of suspected geologic origin. Geologic materials, geologically-derived materials, and unknown materials of suspected geologic origin will be collectively referred to in this document as "geologically-derived materials." These materials lend themselves to identification and comparison utilizing standard geologic/mineralogic techniques and instrumentation. The quantity and condition of the items of evidence, as well as the information requested will influence the selected analytical scheme and the order in which examinations are performed.

Forensic examinations of geologically-derived materials are conducted for investigative and intelligence purposes to:

- determine if two or more geologically-derived materials originated from different sources;
- identify an unknown material;
- determine the origin/end use of geologically-derived materials; and
- determine the significance of finding two or more geologically-derived materials indistinguishable.

A comparison involves the recognition and evaluation of class characteristics that associate materials to a group but never to a single source with the exception of a fracture fit. Only when two or more geologically-derived materials physically fit together can it be said that they were once part of the same broken object (e.g., brick pieces). When a difference is found between compared items, the examination may be immediately discontinued. Refer to *Geoforensics* by Ruffell and McKinley (2008) for further discussion.

This technical procedure is implementing through incorporation by reference the ASTM International E3272, Standard Guide for the Collection of Soils and Other Geological Evidence for Criminal Forensic Applications, E3392, Standard Guide for Forensic Physical Fit Examination. E3254, Standard Practice for Use of Color in the Visual Examination and Forensic Comparison of Soil Samples. ASTM E3272, E3392, E3254 are on the Organization of Standard Area Committees (OSAC) Registry of Approved Standards.

2 SCOPE

This document applies to Geologist-Forensic Examiners and qualified analysts within the Trace Evidence Unit (TEU).

3 EQUIPMENT

- 1 and 1½ gauge cover slips
- 10% Hydrochloric Acid (HCl) (reagent grade or higher)
- Balance capable of measuring in grams to the second decimal place

- Calcite, Ward's Scientific or equivalent supplier of geologic material
- Cameras
- Cargille liquids
- Centrifuge
- Detergent
- Ethyl Alcohol (practical grade or higher)
- Glass microscope slides
- Heat lamp
- Hot plate
- Light box capable of at least providing daylight equivalent light, and equipped with a tungsten lamp
- Magnifying glass
- Microwave oven
- Mortar and pestle
- Muffle furnace
- Munsell Soil Color Charts
- Negative pressure fume hood equipped with HEPA filtration
- Personal protective equipment as needed
- Petrographic microscope with minimum 4x objective, 7.5x oculars
- Probes (e.g., metal, wooden.)
- Rock hammer or equivalent tool
- Sediment texture standard card
- Sieves
- Stereobinocular microscope with minimum magnification of 4 diameters
- Tweezers
- Ultrasonic bath

4 STANDARDS AND CONTROLS

A. Spot test sample of calcite reference material with cold 10% HCl by dripping a drop of acid on a clean section of calcite to verify reactivity of acid. If the standard effervesces, the acid is acceptable.
B. Munsell Soil-Color Charts with genuine Munsell® color chips, Munsell Color x-rite Productions, Michigan, 2015 or later.

5 SAMPLING

The collection and processing of geologically-derived materials from submitted items of evidence can be reviewed in the TRACE-200: Evidence Handling and Processing.

6 PROCEDURE

A. Geologically-derived materials may be received in many conditions. Assess the sample of evidence and handle appropriately to minimize deleterious change.
    1. Centrifuge samples received in liquids (e.g., pond water, stomach contents) to aid in recovery of solid materials, when appropriate.

i. Place sample in a centrifuge tube.
ii. Place tube in a centrifuge.
iii. Spin sample at 3000 RPM for 5 minutes. Repeat if necessary.
iv. Decant supernatant liquid sample. Solid material is retained in the bottom of the tube.

2. When wet samples are received, dry them to ambient conditions. The samples may be air dried, gently heated with a heat lamp, or exposed to microwaves.
    i. When air-drying samples, place open containers in a fume hood and allow to dry.
    ii. For more rapid drying, place samples in an open heat-resistant container under a heat lamp and allow to dry.
    iii. When using a microwave oven, place samples in an open heat-resistant container in the microwave oven, and heat on the highest setting for five-minute increments. Check samples for dryness after each five-minute increment, adding more increments as needed until the samples are dry.
3. Recovered geologically-derived materials (questioned materials) will be evaluated to identify characteristics suitable for comparison prior to comparison to exemplar geologic materials samples. Characteristics not present or expressed in the recovered geologically-derived materials need not be assessed in the exemplar geologically-derived materials. Similarly, characteristics not present or expressed in the exemplar geologically-derived materials need not be assessed in the recovered geologically-derived materials.

B. If the questioned material is cohesive and of sufficient size, and the examination is requested or useful, perform fracture analysis with known exemplars. Refer to the GEO 510: Fractography of Brittle Materials. If the materials physically fit together and show sufficient correspondence between their macro- and microscopic characteristics to indicate they once comprised a single object, and insufficient disagreement between their macro-and microscopic characteristics to conclude that they originated from different objects, no further testing may be necessary.

C. Examine the sample using a low power magnification (e.g., magnifying glass, stereobinocular microscope), and process to remove materials of interest as needed.
    1. For intact or partially intact samples, record approximate dimensions, as appropriate, prior to altering.
    2. If necessary, mechanically separate or subdivide components using probes, tweezers, mortar and pestle, rock hammer, or other tools as appropriate.
    3. The sample may be ashed to remove organic material (e.g., stomach contents, leaf litter).
        i. Place the sample or a portion of the sample in a crucible.
        ii. Place the crucible in a muffle furnace.

iii. Set the temperature of the muffle furnace to between 450ºC and 550ºC, and heat for at least 8 hours.

D. Assess the dried sample(s) for gross color, texture, quantity, general appearance, and initial identification of the item's components to include anthropogenic and organic materials. At the discretion of the Geologist-Forensic Examiner, the sample(s) may be photographed.

E. Using natural light or a light box under simulated daylight conditions, observe the color of each sample noting color variation and/or mottling, the color of multiple geologically-derived materials if present, and any differences in color between unconsolidated soil and aggregates if present.
    1. The color of the samples may be measured by comparison with the Munsell Soil Color Charts.
    2. Metamerism may be observed using the tungsten lamp.
    3. Note any mottling, staining, or variation in color from exterior to interior of a sample. If only weathered surfaces are apparent, the sample may need to be further broken until a fresh surface is obtained.
    4. If the analysis is a comparison, compare the color of the samples to each other. Comparison of like-sized samples side-by-side will reveal slight differences in color. Samples are considered distinguishable if the observed hue, tone, or saturation of color is different.

F. Examine each sample for texture visually, using stereobinocular and petrographic microscopes, or other magnification as necessary. Textural features include grain and aggregate morphology, particle size distribution, and weathering. In addition to light microscopy, scanning electron microscopy (SEM) or Raman spectroscopy can be useful in characterizing particle morphology. Particle size can be determined by a variety of techniques including sieving, sediment texture standard cards, Raman spectroscopy, and microscopy (stereobinocular, petrographic, and electron).
    1. If the analysis is a comparison, compare the texture of the samples to each other**.** Samples are considered distinguishable if any of the observed textural features between samples are different, with the exception of grain size distribution in unconsolidated materials.
        i. Perform comparisons between similar grain-sized fractions. Differences in grain size distribution between questioned and exemplar samples in unconsolidated materials may be attributed to transfer or may reflect genuine differences in sources. The samples are considered distinguishable when other textural features are different.

G. If it is beneficial for the analysis and there is sufficient material to sub-sample, remove a sub-sample of the material for washing into a crucible or other appropriate container. Sub-samples can be washed to facilitate component identification by petrographic microscopy or Raman spectroscopy. If warranted for additional analyses (e.g., clay mineralogy), weigh the sample prior to washing.
    1. Mix enough detergent in water to form suds when agitated.

2. Place sample in dilute detergent and water solution and ultrasonicate for 10 to 15 minutes.
3. Decant the supernatant liquid with suspended clays and organic matter into a beaker.
4. Repeat steps 5.7.1 through 5.7.3 until supernatant liquid is clear after ultrasonication.
5. For comparison examinations, visually compare supernatant liquid side by side, and document any differences noted.
6. After removal of the supernatant liquid, the material left behind in the crucible (washed particles) is triple rinsed with water then triple rinsed with ethyl alcohol. The rinsate is recovered into the beaker containing the supernatant liquid.
7. The washed portion and supernatant liquid may be air dried or gently warmed by a heat lamp, hot plate, or microwave oven to hasten drying.

H. At the discretion of the Geologist-Forensic Examiner, perform a heavy mineral separation on the washed particles according to the GEO-530: Sample Preparation: Gravity Separation Using Heavy Liquids.

I. Identify the components present and their relative proportions using stereobinocular and petrographic microscopes, or other methods, as needed.
    1. Materials examined using a petrographic microscope may be mounted on glass slides in various mounting media (e.g., water, Cargille liquids) to facilitate identification.
    2. If the analysis is a comparison, compare composition between samples. Samples are considered distinguishable if their components are different or if the components are present in different relative proportions. The significance of finding two or more items distinguishable will vary depending upon the rarity of the materials involved.
        i. If the volume of either the known or questioned sample is small, minerals present in trace amounts may not be present/observed in the sample. The absence of a phase present in trace amounts in a small sample does not by itself constitute a difference between samples; however, conclusions using these samples can be more limited.

J. If Portland cement is identified as a phase in a sample of unknown materials for building material by PLM and/or microchemical tests, confirm its presence by x-ray diffraction (XRD).

K. In samples containing Portland cement, the Portland cement may be removed to examine the aggregate.
    1. Digest a portion of the sample in 10% HCl.
    2. Decant supernatant liquid.
    3. Rinse solid material with water.
    4. Dry solid material in air or with gentle heat. It should be noted that this method will also dissolve any calcite, dolomite, or gypsum contained in the aggregate.

L. If glass fiber is identified, determine whether it is continuous, single strand, matted, woven, etc.
M. Conduct additional analyses at the discretion of the Geologist-Forensic Examiner, depending on the needs of the examination. Some examples are listed below, although other techniques may be used as necessary.
    1. X-ray diffraction may be useful for identifying minerals below the resolution of a petrographic microscope.
        i. At the discretion of the Geologist-Forensic Examiner, prepare samples according to GEO 515: Sample Preparation: X-ray Powder Diffraction.
        ii. At the discretion of the Geologist-Forensic Examiner, analyze samples according to GEO 538: X-ray Powder Diffractometry Using the Empyrean X-ray Diffractometer.
    2. Pollen identification may be useful in provenance examinations.
    3. X-ray fluorescence spectrometry may be used to assess chemical composition.
    4. Scanning electron microscopy can be used to image materials below the resolving power of a petrographic microscope.
N. Record all pertinent observations, measurements and instrument data in case notes.

6.1 Verification

A. Geologically-derived materials associations will be verified by a second, qualified Geologist-Forensic Examiner.
B. A geologically-derived materials association is defined as follows:

    1. When two or more pieces of a similar geologically-derived material (e.g., block) physically fit together, the compared items were once part of the same object.

    or

    2. When the color, texture and composition are indistinguishable, the possibility that the compared item(s) originated from the same source as the known exemplar cannot be eliminated.

C. All other geologically-derived materials examinations results may be verified by a second, qualified Geologist-Forensic Examiner.
D. Verifications will be recorded in Forensic Advantage (FA).

7 LIMITATIONS

A. The amount and condition (e.g., adulteration, composition) of an item(s) of evidence can limit the examination(s) and resulting conclusion(s) (See, for example, Section 6.F.1.i).
        i. Soil colors with low chroma and low value may require additional characterization by other methods.

B. Possible selective transfer and persistence of different geological components should also be considered. Generally, the composition of the questioned soil sample can change during transfer or persistence from the original source contact location. Therefore, similar size fractions should be compared in any analysis.
C. It is usually not possible to predict the total number of items in a class of materials produced with the same characteristics. In materials with greater compositional and morphological variability (e.g., bricks), a smaller percentage of the total number of items in a class will be indistinguishable than in materials with very little variability (e.g., type E glass fiber).
D. Soil properties vary both across the land and below the land surface as a function of parent material, climate, biological activity, geography, and time, yielding soil that is different from location to location and with depth below the surface. These differences can occur abruptly or gradually. Therefore, the exemplar soils from a specific location must be interpreted to represent only that location and may not be representative of all soils in the area or soil that may have been present in the past.
E. Due to the possible variations in soil, the boundaries of a homogeneous soil cannot be predicted with absolute certainty. Soil and geologic studies and maps of an area may assist in defining the approximate extent of a homogeneous soil.

8 SAFETY

A. Some mounting liquids are classified as hazardous and are handled and disposed of in accordance with manufacturer's recommendation or the FBI Laboratory Safety Manual. Avoid contact with skin. Acids can cause burns, throat and eye irritation. Avoid contact with skin and use in a well-ventilated area. SDS sheets for mounting liquids are maintained with the mounting liquids and should be consulted for specific precautions for these materials.
B. Eye protection and gloves will be used when crushing specimens of building materials.
C. Building materials such as floor and ceiling tiles, wallboard, skim coats, acoustical plasters, mastic, insulation, and fireproofing may contain asbestos fibers. If a material is suspected to contain asbestos, it must be examined in a negative pressure fume hood equipped with HEPA filtration. Specimens and waste material must be contained in airtight containers when outside the fume hood. These precautions must be followed until it is determined that the material does not contain asbestos. Asbestos containing waste material must be disposed of as a hazardous waste.

9 REFERENCES

E3272, Standard Guide for the Collection of Soils and Other Geological Evidence for Criminal Forensic Applications.

E3392, Standard Guide for Forensic Physical Fit Examination.

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

Ruffell, Alastair, and McKinley, Jennifer, *Geoforensics*, 2008. Wiley and Sons, Ltd

Munsell Soil Color Charts (current version)

FBI Laboratory Safety Manual (current version)

GEO-530: Sample Preparation: Gravity Separation Using Heavy Liquids (current version)

10 REVISION HISTORY

Revision	Issued	Changes
06	09/01/2021	Added "Additional equipment..." to Section 2 Equipment list. Changed "ped/clod" to "aggregate" throughout. Added "Raman spectroscopy" to sections 5.6. and 5.7. Changed "polarized light" to "petrographic" in Section 5.7. Expanded abbreviation to complete name in section 5.10. Changed "Mineralogy" to "Geology" in Approval Section. Changed TL in Approval Section.
07	01/28/2022	Reformatted entire document including updated references. Added 'qualified analyst' to Scope.
08	01/02/2025	Added ASTM references. Clarified 6M1i and 7A.