10.12 Soil Comparison

NOTE: The FSD is not currently performing soil comparisons. If an agency requests this analysis, please have them contact the FBI Laboratory Evidence Control Unit at (703) 632-8360 for instructions. The FBI laboratory will do this analysis at no cost to the agency. The agency will send their evidence items directly to the FBI laboratory.

In addition, soil samples can be examined by FSD Trace examiners for the presence of other possible trace evidence which may lead to a comparative association, such as paint chips, fibers, building materials, etc. Examination for these materials can be considered on a case-by-case basis.

10.12.1 Introduction

Soil is submitted to the laboratory in various types of cases including burglary, sexual assault, homicide, and criminal damage to property.

Soil is defined as a physical, chemical, and biological complex. Soil is a four part system composed of (1) finely divided minerals and amorphous inorganic solid(s), (2) animal, plant and microbiota residues in various stages of decay, (3) a living and metabolizing microbiota, and (4) man-made materials.

Soil formation is affected by six factors: (1) composition of parent material, (2) climate, (3) organisms, (4) topography, (5) time, and (6) vegetation. As a result, soil contains varying amounts of natural organic material, natural minerals, clays, and various contaminants, some of which are man-made. This is the reason that careful and thorough examination of soil samples is important.

Soils within a given area can vary due to the above factors and due to any landscaping. As a result, a series of representative samples need to be obtained and analyzed. Sampling problems can account for differences, but these are out of the control of the analyst. The analyst must work with the samples that have been submitted or request additional samples as necessary.

10.12.2 Safety Considerations

Standard Laboratory Precautions.

Chemical	NFPA
acetone	1-3-0
bromoform	3-0-0
cargille liquids	3-2-2
ethanol, 5%	0-3-0
meltmounts	0-0-0
nitrogen, liquid	3-0-0
permount	3-3-0

Use extreme cold precautions when handling the liquid nitrogen.

10.12.3 Preparations

- stereomicroscope
- ultrasonic bath
- polarized light microscope
- oven
- Munsel color Chart
- microbalance
- sieve set (#35, #60, #80)
- watch glasses
- diSPO culture tubes, 10X75mm
- 5% ethanol soln. (95mlH2O + 5ml EtOH)
- diSPo culture tubes, 13X100mm
- acetone

- diSPO culture tubes, 20X150mm
- liquid nitrogen
- 1.5ml polypropylene microcentrifuge tubes
- bromoform
- 1.680 Meltmount
- small magnet
- 1.538 Meltmount
- slides/coverslips
- Permount
- filter apparatus
- Cargille liquids
- test tube racks
- rubber stopper
- 4mm vials
- silver membrane 25mm filters, 0.45 um
- microcentrifuge
- (FM25-45 Hytax filter 1/800-848-1750)

10.12.4 Minimum Standards & Controls

The minimum amount of soil needed for a soil examination is listed in step 3 of this procedure. No unexplained differences between the questioned and standard samples are allowed for inclusion.

10.12.5 Instrumentation

Stereomicroscope
Polarized light microscope

SEM-EDS

10.12.6 Procedure or Analysis

NOTE: First examine the questioned and known samples with stereoscope for the presence of other trace evidence which may lead to a more conclusive comparative associations, such as paint chips, fibers, building materials, etc.

10.12.6.1 General Separation Scheme

NOTE: When comparing colors, be sure no color difference can be attributed to the amount of sample. Initial screening consists of steps 1-5.

10.12.6.1.1

Step 1: Dry all samples thoroughly by air drying or in a 50-60ºC oven.

10.12.6.1.2

Step 2: Break up the sample using a rubber stopper to remove any clumps. If the sample is stratified, the layers must be removed separately for analysis as individual samples.

10.12.6.1.3

Step 3: Use 500mg of the sample, retaining the rest of the sample for further analysis. If the sample size is less than 500mg, use all the sample. Sieve each sample through the #35 (500um) and #60 (250um) sieves. If the sample in the catch container is less than 25mg, then further no examinations/comparisons can be done due to insufficient sample size. If the sample in the catch is 25mg or greater, then comparison between the questioned and the standard samples is performed.

10.12.6.1.4

Step 4: Compare the physical characteristics of the material on the #35 mesh. Compare the color of the questioned sample to the standard(s) on the #60 mesh and in the catch using the Munsel Color Chart.

Compare the non-mineral particles in the samples. Differences in color between the questioned and standard samples eliminates them from having the same source. Significant differences in non-mineral particles between the samples eliminates them from having the same source. If the samples cannot be discriminated, then proceed to the next step.

10.12.6.1.5

Step 5: Sieve the catch samples through a #80 (177um) sieve. Compare the color and non-mineral particles of the questioned sample to the standard(s) on the #80 mesh and in the catch using the same method as in Step #4. Remove any non-mineral material and retain for later analysis (man-made material, botanical material, insect parts, etc.). Proceed to the next step if the samples cannot be discriminated.

10.12.6.1.6

Step 6: Combine sievings with original sample. Place 300-500mg of sample in a 10 x 75mm test tube and add 5% ethanol solution. Sonicate for three minutes.

10.12.6.1.7

Step 7: Stir and allow to set ten seconds. Remove supernate with a pipette and place in a 13 x 100mm tube.

10.12.6.1.8

Step 8: Add more 5% ethanol solution to the sample, sonicate one minute, stir, and let stand ten seconds. Remove supernate and place with previous supernate.

10.12.6.1.9

Step 9: Repeat the extraction procedure (step 8) until a clear solution is produced and the fine sand/silt granules readily settle out. The material remaining in the tube is called Fraction 1.

10.12.6.1.10

Wash Fraction 1 three times with acetone to dehydrate the fraction. If a color develops on the first washing, save these extracts since oil, grease, asphalt, and similar materials will dissolve in acetone.

10.12.6.1.11

Allow Fraction 1 to dry. Use air or gentle heat (50-60ºC). Compare color.

10.12.6.1.12

Stir the supernate from steps 7, 8, and 9 and let stand ten minutes. Pipette off the supernate and place in 20 x 15mm tube. The supernate is called Fraction 3. The "sludge" is called Fraction 2.

10.12.6.1.13

Wash Fraction 2 with acetone two times, decanting off the liquid each time. Wash it a third time and use the acetone to transfer Fraction 2 to a 10ml beaker. Decant the acetone.

10.12.6.1.14

Allow Fraction 2 to dry. Use air or gentle heat (50-60ºC).

10.12.6.1.15

Allow Fraction 3 to stand overnight. Remove the supernate which is called Fraction 3B. The "sludge" is Fraction 3A.

10.12.6.1.16

Allow Fraction 3A to dry using air or gentle heat.

10.12.6.1.17

Filter Fraction 3B using a silver membrane filter.

10.12.6.1.17.1

If the filtered and dried 3B fractions are consistent in color, continue with the analysis of Fraction 1. (Be sure no differences can be attributed to amount of sample.)

10.12.6.1.17.2

Proceed to Analysis of Fractions 3A and 3B analyses if no differences are observed.

10.12.6.1.18

Analysis of fractions A and B: Both fractions are examined using the following methods.

10.12.6.1.18.1

After the Fractions are filtered, allow to dry.

10.12.6.1.18.2

Compare color using the Munsel Color Chart.

10.12.6.1.18.3

Perform EDS analysis to determine clay types. Can exclude or include at this point. If an exclusion is made, no analysis of Fraction 1 is necessary.

10.12.6.1.18.4

A flow chart of this analysis is given in *Appendix 10.12 A: Analysis of Fractions 2 and 3*.

10.12.6.2 Analysis of Fraction

10.12.6.2.1

The 90-180um sand and silt in Fraction 1 is separated by density using bromoform (specific gravity about 2.89). The mineral particles are transferred into a polypropylene centrifuge tube which is then half filled with bromoform. The heavy minerals will sink, the lighter minerals will float or remain suspended.

10.12.6.2.2

Place the polypropylene tube into a styrofoam cup containing liquid nitrogen to a level covering the heavy minerals. This will freeze the bromoform and heavy minerals into place. The light minerals and some of the liquid bromoform can be washed out of the tube with acetone into a watchglass. Excess bromoform can be washed from the minerals with acetone. Excess acetone can be allowed to evaporate. The dried light minerals are mounted in 1.540 refractive index liquid (or 1.538 Meltmount) for characterization.

10.12.6.2.3

The dried heavy minerals are mounted in 1.680 Meltmount and examined microscopically.

10.12.6.2.4

Count and categorize or identify mineral particles in each of the two mineral sub-fractions. *Refer to Appendix 10.12 B: Polarized Light Microscopy - Soil Mineral Analysis* (McCrone Research Institute Mineral Identification Key).

10.12.6.2.5

The procedure should be applied in exactly the same way to the known and questioned samples of soil.

10.12.6.2.6

Refer to Appendix 10.12 C: Analysis of Fraction 1 for a flow chart of the analysis process.

10.12.6.2.7

X-ray analysis of both light and heavy fractions can be used to assist in identifying mineral content. Crush the sample to less than 25um. If the heavy portion of Fraction 1 is too small, Fraction 3A should give the same results. The sample is then analyzed using an SEM/EDS using the following parameters:

stepwise analysis:

beginning angle 6º

ending angle 70º

step width 0.05º

dwell time 5 sec

EDS should be used until the examiner is proficient in examining the mineral grains with the polarized light microscope.

10.12.6.2.8

Refer to Appendix 10.12 D: Grain-Size Scales

10.12.6.2.9

Refer to Appendix 10.12.E: Comparison Chart for Estimating Percentage of Composition

10.12.6.2.10

Refer to Appendix 10.12 F: Particle Size Conversion Table by Sieve

10.12.6.2.11

Refer to Appendix 10.12 G: Soil Analysis Worksheet

10.12.7 References

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