



**SAN DIEGO POLICE DEPARTMENT
CRIME LABORATORY
TRACE EVIDENCE UNIT MANUAL**



Trace Unit Manual

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1.1 UNIT OVERVIEW

UNIT DESCRIPTION

The Trace Evidence Unit is located on the 6th floor of the San Diego Police Department Headquarters building. The general hours of operation are Monday through Friday, 0600 hours until 1630 hours.

The unit is composed of one Supervising Criminalist, one Criminalist III technical lead, and a staff of Criminalists.

The unit adheres to the requirements of the Laboratory Quality Assurance Manual. Additional requirements specific to the Trace Evidence unit are listed in this manual.

UNIT FUNCTIONS

The criminalists in the Trace Evidence Unit perform several different types of analyses. These include analyses in the following areas:

- Evidence Screening
- Fire Debris and Ignitable Liquids
- Fiber
- Hair
- Impression
- Explosive
- Paint
- Physical Match
- Gunshot Residue

Additional duties may include:

- Crime Scene Reconstruction
- Projects assigned by the unit supervisor

FACILITY

The Trace Evidence unit is comprised of the main unit, an instrument room, and an SEM room. The unit doors will remain closed and locked during business hours. The unit is considered a secure evidence storage room.

EVIDENCE HANDLING

Unit personnel will maintain control of their evidence by ensuring that case evidence is not in an area where the evidence can be lost, compromised or altered. They will wrap or cover the evidence, as needed, to ensure evidence integrity.

The analyst must ensure that tools, examination area, laboratory coat, and gloves are clean. Each item of evidence is examined on a clean surface. Based on the types of analyses, the clean surface can be new butcher paper, clean glassware, or counters cleaned with water.

The examination of questioned and known reference items of evidence are separated either by location or by the examination of items at different times.

Victim and suspect evidence will be examined in different areas within the unit.

To prevent GSR contamination of the work areas within the unit, situations where contamination might occur and the appropriate precautions to take are outlined below:

- An analyst is exposed to primer residue during a firearms examination.
 - The analyst will not examine firearms in the same area that SEM/GSR analysis is conducted.
 - A separation in time and location is desired for firearms examination by trace examiners conducting GSR analysis.
 - The area to be used for the firearms exam will be cleaned before and after any firearms examination and a single use lab coat will be worn.
- Test firing
 - The analyst will wear a single use lab coat. This will either be a disposable lab coat, or the lab coat will be washed after one use.
 - The analyst will wash his hands and face after test firing or contact with firearms.
 - The analyst will not perform any GSR/SEM analysis on the same day that he or she test fires or has contact with a firearm.
- Firearm qualifications/shooting range practice
 - The analyst will schedule range qualification and practices in the afternoon and avoid the trace evidence area after qualifications or range practice.

2.1 GENERAL ANALYSIS

Materials and Equipment

Reagents and Supplies

- Permunt
- Xylene
- Refractive index liquids
- Glass slides
- Coverslips
- Forceps
- KBr
- NaCl plates
- Standard Materials
- Sample mounting stubs for SEM
- Methanol for GCMS
- Carbon Disulfide

Equipment

- Stereomicroscope
- Polarizing microscope
- FTIR microscope
- Scanning electron microscope with EDS
- FID-GC
- GC/MS

Procedures

General Guidelines

- Items submitted for general analysis include a wide variety of materials. The Supervising Criminalist shall assign a Criminalist to each case based on the type of examination requested and the instrumentation and expertise required to perform the analysis.

Evidence Handling

- Preliminary Considerations
 - Prior to the analysis of evidentiary material, an evaluation of the relevant elements of each case should be obtained through communication with the submitting detective and/or prosecutor. This evaluation should include an assessment of the evidence and its relevance.
- Special Considerations
 - Trace evidence can be contaminated during laboratory examination.
 - The analyst must ensure that tools, examination area, laboratory coat, and gloves are clean.
 - Examine each item of evidence on a clean piece of butcher paper.
 - Separate the examination of questioned and known reference items either by location or by examination of the items at different times.

Goals of Examination

Identification: Liquid, powder, or granular material can be examined utilizing a variety of instrumental, microscopic, and microchemical techniques. A variety of evidence could fall under the category of general unknowns (e.g. caustics, bleach, tear gas, glass, plastics, wax, organic compounds, metals, wood, chalk, diatomaceous earth, fire extinguishers, match heads, safe insulation, etc.).

Physical Matches and Comparisons: A variety of materials can be examined for physical characteristics to determine if two items could share a common source or can be physically matched back to the original source. Examples of physical matching can include fractures in metal, wood, plastics, tears in tape, paper, fabric, etc. When a physical match is not possible, then the questioned and known samples can be physically and chemically characterized to determine if they share a common source.

General Approach

The analyst will evaluate the exhibit and attempt to characterize the type or class of evidence. The analyst can choose microscopic, microchemical, or instrumental methods of examination. Appropriate controls, standards and laboratory procedures will be employed. Appropriate safety precautions will be exercised on unknown exhibits. The analyst will consider the possibility of toxic substances or reactions. For example, cyanide or tear gas exhibits would require personal protective gear and/or analysis in the hood. The analyst will evaluate the type of evidence, the amount of evidence, and the potential for interpretation in making the decision to use specific types of instrumentation. In most cases, destructive testing such as pyrolysis or solubility is to be avoided. See the general policy for evidence consumption.

The analyst will also be aware of other types of evidence such as fingerprints, blood, semen and narcotics. The analyst will safeguard the evidence by sequencing the types of examinations to minimize any loss of evidence through sampling. The potential for individualizing must be considered paramount. If fingerprints or DNA evidence could be compromised, the analyst will consult with the appropriate sections to determine the sequence of examinations.

Standards of Performance

Focus: The aim of this type of analysis is to identify an item chemically and/or physically to the exclusion of all other classes of items or to determine if two items are similar and could have come from the same source. The analyst will evaluate the evidence and decide the best analytical approach. The type and amount of evidence will dictate the analytical approach. Each case will be evaluated on its own merits. It is recognized that no single analytical approach can cover all types of examinations. This is only meant to provide a guide or framework.

The analyst will document the condition of the evidence as received, the date the evidence was received, and will provide a written description of the items received. The analytical notes will reflect the examinations conducted and the results and/or conclusions reached and will include appropriate sketches, photographs, and analytical printouts. The notes should

enable the technical reviewer to reconstruct the analytical steps taken and compare any spectra or data.

Infrared examination with high quality spectra and standards is a conclusive identification. GCMS with high quality spectra and standards also constitutes an identification. Polarized light microscopy with microchemical or other confirmatory tests can be a conclusive identification. An energy dispersive spectrum on the SEM can qualitatively describe the elemental composition of a sample. It is highly desirable to have two independent forms of examination applied to a sample for an analytical conclusion.

If the evidence falls under a method described elsewhere, those standards (under that method) will be applied.

Methods of analysis will be validated and scientifically justified. The methods will be reproducible. Another analyst should be able to derive the same information from the same evidence or data. The analyst will avoid consuming all of the evidence. Some of the evidence must be maintained for defense analysis.

Conclusions

If an item has been identified chemically and/or physically the analyst can report precisely that a particular compound was found in the submitted item. The analyst needs to be specific in explaining why certain examinations were conducted and the significance of these examinations. Not detecting a substance can mean that the levels present are below the sensitivity of the method or that the substance is not present.

When a physical match has been demonstrated, the analyst can state that the two pieces (items) were at one time, one piece. This is a unique association.

References

Walter C. McCrone, Lucy B. McCrone, and John G. Delly, "Polarized Light Microscopy", Michigan: Ann Arbor Science Publishers, 1978.

Frederick Cunliffe and Peter Piazza, "Criminalistics and Scientific Investigation", New Jersey: Prentice-Hall Inc., 1980.

Operation manuals for the GC, GCMS, FTIR, FTIR microscope, and the SEM.

2.2 FIRE DEBRIS ANALYSIS

MATERIALS AND EQUIPMENT

Reagents and Supplies

- CS₂ (Use this material in a hood, with proper personal protection equipment)
- Activated charcoal strips
- Injection syringes – gas and liquid
- Dental floss
- Forceps
- Glass sample vials with Teflon septa
- Standard accelerants
- Resolution test mixture (Restek)
- Glass wool/cotton
- Glass Pasteur pipettes

Equipment

- GC-FID
- GC/MS
- Oven (up to 100 ° C)

PROCEDURES

Evidence Handling/Documentation

- Preliminary Considerations
 - Solid debris: Solid debris evidence suspected of containing a flammable liquid is collected at the scene in 1 gallon or smaller paint cans with crimp type lids firmly sealed. These cans are preferably epoxy coated and should be tested on a lot-by-lot basis for the presence of petroleum products prior to being used for evidence collection. The laboratory tests every lot submitted by the investigators. The laboratory does not provide these cans to the investigators. Other suitable material for larger items is Kapak. This material is available in pre-made bags, or in rolls for very large items. Each lot of the Kapak material should be tested prior to use.
 - Comparison samples: The investigator should submit comparison samples with each case. These may include unburned and uncontaminated carpeting, padding, upholstery padding, wood, etc. The comparison samples are analyzed using the same procedures as those utilized for analysis of the unknown samples.

- Liquid evidence: Liquid evidence is collected from the scene and placed in a tightly capped glass vial. If liquid is found in its original container, collect the container.
- Documentation
 - The form in which the evidence is received should be described, and the integrity of the container noted. If solid debris is submitted in a paint can, the can should be opened carefully. Quickly make note of the contents and any odors, then reseal tightly. If a liquid sample is submitted, make note of the color, viscosity, clarity, the presence of layers, and any odors. For safety reasons, do not purposefully smell or inhale the odors from an evidence container.

Sample Preparation

- Passive adsorption/elution – solid sample
 1. Suspend a carbon strip in the interior of the can using unwaxed dental floss. The carbon strip should not come into direct contact with any of the contents if possible. Contact with materials can overload the strip. This can be avoided by splitting up the sample between two containers. If the strip does come in prolonged contact with the material in the can, keep this in mind when evaluating the chromatogram for apparent overloading of the strip.
 2. Reseal the can tightly. Heat for 1 – 2 hours at 80°C or leave can at room temperature overnight. If a Class 4 or 5 accelerant is indicated after room temperature adsorption period, insert a new carbon strip and repeat the procedure heating for 1–2 hours at 80°C.
 3. Remove strip and place in small glass vial with 20 – 50 drops of CS₂. Cap tightly.
 4. Prepare a blank charcoal strip similar to samples and analyze with each set of samples.
 5. Prepare CS₂ blank to be analyzed with each set of samples.
- Liquid Extraction – liquid sample
 1. Test flammability.
 2. If necessary, filter liquid through Pasteur pipette containing glass wool to remove particulates.
 3. Dilute sample appropriately with CS₂.
 4. Prepare CS₂ blank to be analyzed with each set of samples.
- Liquid Extraction – Solid Sample (This procedure is a destructive procedure and should only be utilized when other extraction methods have failed to yield results.)
 1. Transfer a representative portion of the sample to suitable container (beaker, test tube, Kapak bag, etc.) of the appropriate size.
 2. Add enough solvent (CS₂, pentane, etc.) to just cover the material.
 3. Agitate by stirring, shaking, or vortexing for a few minutes.
 4. Decant or pipet off solvent. Filter the solvent if particulates are present using filter paper, a Pasteur pipet with glass wool or cotton, or other suitable filter apparatus in to a smaller beaker.
 5. Evaporate the extract down to a suitable volume using an air stream without heat.

6. Transfer the extract to an appropriately labeled autosampler vial.
7. Follow procedure for liquid analysis.
8. Prepare a blank of the solvent used for extraction (CS₂, pentane, etc.).

Instrumental Analysis

- Once a suitable sample is prepared, it is injected into a gas chromatograph. The GC must be equipped with a non-polar capillary column at least 12 meters in length, a temperature programmable oven capable of producing temperatures in the range of 40 – 300°C, and an FID. Any column and temperature conditions can be used provided a test mixture consisting of normal alkanes ranging from n-hexane through n-eicosane can be resolved into its component peaks. The test mixture should also include the following aromatic compounds: toluene, p-xylene, o-ethyltoluene, m-ethyltoluene, and 1,2,4-trimethylbenzene. Analyze samples using the following steps:
 - 1 Blank - Inject 1µl CS₂ prior to sample analysis and between samples
 - 2 Blank - Extract charcoal strip with CS₂. Inject 1µl CS₂ extract prior to sample analysis (if required)
 - 3 Standard- Resolution test mixture (Restek), inject 1µl or less
 - 4 Samples - Inject 1µl or less of sample
 - 5 References - run fresh standard accelerants (from library) under the same conditions as the sample (if required). Inject 1µl or less
 - 6 Print chromatograms - Print standard, sample, and control chromatograms using the same x (time) scale. Ensure that all peaks are present. The resulting chromatogram peaks should be well resolved and the largest peak excluding the solvent is 50 – 100% of full scale. Manipulation of x and y axes may be necessary to detect, demonstrate, or compare special features. Each chromatogram must be labeled with:
 - Examiner's handwritten name or initials
 - Case number and date
 - Item number
- A commercially available resolution test mixture (Restek) that includes n- alkanes will be run every time there is significant maintenance on the instrument that could alter retention times, and after the yearly preventative maintenance service.

GCMS Analysis

- Samples are prepared and run on the GC/MS in a similar fashion to those analyzed on the GC-FID.

Interpretation

- Classification Scheme (See ASTM E 1387-95): Based on GC-FID chromatographic patterns alone, virtually all commercial, volatile, petroleum products can be assigned to one of six classes. Five of these six classes represent petroleum distillates whose predominant feature is boiling point range as reflected in the peak spread of the chromatogram. Another important feature includes an evenly

spaced series of normal alkane peaks. Aromatic and branched-chain compounds may also be present. The sixth class includes single- component compounds or synthetic mixtures consisting of only a few compounds whose chromatograms do not display a range of boiling points or a symmetrical distribution.

- Interpreting the Chromatogram (See ASTM E 1387-95): In order for an extract to be identified as containing residues belonging to a particular class of petroleum distillate, the pattern of the chromatogram must be comparable in the number, position, and relative peak heights to a standard chromatograph. Special care should be taken with pyrolysis products that are present. These products may interfere with the identification of an accelerant. The chromatograph from the comparison sample can be useful in differentiating the peaks contributed by pyrolysis products from those of the accelerant.

TABLE 1

Class 1: Light Petroleum Distillates

Majority of the pattern occurs in the C₄ to C₉ range of normal alkanes. No major peaks above C₁₁.

Class 2: Gasolines

Peaks present in the range of C₄ (n-propane) to C₁₂ (n-dodecane). The methyltoluene/pseudocumene 5-peak group must be present. This group occupies the range between C₉ and C₁₀ and is still present in gasolines which have lost as much as 90% of their initial weight by evaporation or combustion. Other peak groupings characteristic of gasoline include C₄ alkyl benzenes and various aliphatic compounds must be present.

Class 3: Medium Petroleum Distillates (MPD)

Peaks present in the alkane range of C₈ to C₁₃. No major peaks associated with ignitable liquid below C₇ or above C₁₄.

Class 4: Kerosenes

Peaks present in the range of C₉ to C₁₆. At least five consecutive n-alkane peaks between C₁₂ and C₁₇ must be present.

Class 5: Heavy Petroleum Distillates (HPD)

Peaks in the alkane range C₉ to C₂₃. Pattern starts above C₉ and at least five consecutive n-alkane peaks between C₁₇ and C₂₂ must be present.

Class 0: Unclassified

Variable peak spread. One to several peaks present. Excellent agreement with laboratory reference specimen and exclusion of background interferences are required before characterization.

Note Packet

- Include how samples were extracted for analysis
- When a sample chromatogram displays all the features that the analyst feels necessary for a particular class identification, the report will state that a petroleum product of that class was detected. It will also name examples of the class, except in the case of a class 2 (gasoline). Gasoline is sufficiently characteristic that it can be reported as a single item rather than as a class.
- Include in final notes the chromatograms of all standards, samples, and blanks produced during the analysis.

Conclusions

- Suggested wording:
 - Identification (solid debris) – A class () petroleum distillate was detected. Alternately, the terms Light, Medium, Heavy (LPD, MPD, HPD) may be used instead of the class. Examples of these include, but are not limited to:
 - Identification (liquid) – The liquid was identified as a class () petroleum distillate. Examples of these include, but are not limited to:
 - Non-Identification – No identifiable ignitable liquids were detected. Instances of non-identification could include: analyst did not find evidence of an accelerant, analyst was unable to identify accelerant, or analyst was unable to exclude pyrolysis products or substrate as source of accelerant.

References

Richard Saferstein, "Forensic Science Handbook", New Jersey: Prentice Hall Regents, 1982.

John O'Conner, "Practical Fire and Arson Investigation", New York: Elsevier Science Publishing Company Inc., 1987.

John DeHaan, "Kirk's Fire Investigation", New Jersey: Prentice-Hall Inc., 1991.

2.3 FIBER ANALYSIS

MATERIALS AND EQUIPMENT

Reagents and Supplies

- Permunt
- Xylene
- Refractive index liquids
- Glass slides
- Cover glass
- Forceps
- Tape lifts
- Razor blades
- BaF₂ cells
- Micro sample press
- Pipette tips
- Acrylic yarn (white and blue)
- TLC plates
- Chloroform
- Methanol
- Acetic Acid
- Pyridine
- Diamond compression cell
- Collodion

Equipment

- Stereomicroscope
- Polarized light microscope
- FTIR microscope
- Comparison microscope
- Hot glue gun
- Hardy microtome
- Hardy thin cross-sectioning device
- UV light box
- ALS
- Mettler hot stage

PROCEDURES

Evidence Handling

Preliminary Considerations

- Trace evidence examinations can employ chemicals and reagents that are known carcinogens and/or hazardous substances. Therefore, the examiner should read the guidelines published by NIOSH and the Material Safety Data Sheets for all reagents and chemicals that are used.

Special Considerations

- Trace evidence can be contaminated during laboratory examination.
- The analyst must ensure that tools, examination area, laboratory coat, and gloves are clean.
- Examine each item of evidence on a clean piece of butcher paper.
- Separate the examination of questioned and known reference items either by location or by examination of the items at different times.
- The questioned items will be examined first and documented before the known items are examined.

Collection of Fiber Evidence

- Collection of questioned fibers - Items examined for fiber evidence are generally viewed visually for any apparent or significant fibers. These are documented, removed, and packaged separately. The following are appropriate collection techniques.
 1. Forceps - Fibers of interest may be located through a visual examination. If the fibers are large enough, collect the fibers with forceps and place in a paper bundle or on a tape lift for storage.
 2. Tape lifts - If the fibers are small, tape lifting is the recommended method for fiber recovery. A tape lift can be patted over the surfaces of the clothes to collect the fibers. The tape lift can then be used for storage of the fibers and for future examinations of the fibers.
 3. Alternate techniques
 - a. Viewing with oblique lighting or with an ALS may enable the examiner to better visualize the fibers prior to collection.
 - b. Scraping to collect fibers and debris from very dirty items where a tape lift would not be effective.

- Collection of standards/known samples – It is important when collecting a fiber sample to obtain an appropriate sample. Ensure that all shades and types of fibers from your sample are represented.
- Evidence storage and disposition – The fibers should be stored in paper bindles or on tape lifts. Evidence should be packaged to prevent cross-contamination.

Fiber Analysis

- Use non-destructive techniques first for both fiber identifications and comparisons. Evaluate any destructive testing for its potential value prior to proceeding. Record with sketches or photography the location of the fibers when this has special significance.
- Macroscopic analysis – Fibers are examined macroscopically for characteristics such as color, length, contour, thickness, and adhering debris. If fabric is submitted, characteristics such as weave pattern are noted. This examination is done with the fibers in an unmounted condition using low power magnification. The analyst may be able to differentiate between known and questioned fibers at this point, however further analyses must be done to identify fiber type.
- Microscopic analysis – Mount the fibers in an appropriate mounting media. Xylene can be used for a temporary mount and Permount can be used for a more stable mount. Each slide must be properly labeled for identification with the barcode number and case number or item number and property tag, analyst's initials, date, or other unique identifier. This examination is done utilizing a polarized light microscope with a calibrated eyepiece micrometer. The levels of magnification range from 40x to 1000x. Examine the fibers for the following properties:
 - Physical properties
 - Color
 - Diameter: approximate range in μm with a eyepiece micrometer. Also note the regularity of the diameter
 - Cross-section
 - Delusterants: Note as to type, size, and amount.
 - Voids, spherulite formation, or any other unusual inclusions should also be noted
 - Twisting, marks, nodes etc.
 - Optical properties
 - Refractive index: n_{\parallel} and n_{\perp} are determined using the Becke line technique. The fiber is immersed in a series of Cargille liquids of known refractive indices. Using uncrossed polars, n_{\parallel} is measured with the fiber parallel to the polarizer's vibrational direction. n_{\perp} is measured with the fiber perpendicular to the polarizer's vibrational direction. The Becke lines moves the media of higher refractive index when the object is moved away from the objective.
 - Birefringence: Can be determined with the use of a Michel-Levy chart or by measuring n_{\parallel} and n_{\perp} .

$$n_{\parallel} - n_{\perp} = \frac{R \text{ (retardation in nm)}}{\text{(diameter in mm x 1000)}} = \text{birefringence } D$$

- Sign of elongation: Orient fiber with its long axis parallel to the slow ray on the $\frac{1}{4}$ wavelength compensator plate. Under crossed polars, insert the compensator plate and observe the change in interference colors. If additive retardation occurs, the sign is positive. If subtractive retardation occurs, the sign is negative. Alternately, determine the refractive indices in the two vibrational directions. If $n_{\parallel} > n_{\perp}$ the fiber is positive, if $n_{\parallel} < n_{\perp}$ the fiber is negative.
- Extinction: Rotate the specimen 360° between crossed polars. Determine if the fiber has parallel extinction.
- Pleochroism: Rotate the specimen 90° and note any color change in the fiber.

At the conclusion of this examination, the analyst may be able to differentiate between the questioned and known fibers based upon the optical and physical properties. This examination will also enable the analyst to identify most common types of fibers. At this time, if the fibers appear similar, a side-by-side comparison of the physical properties of the known and questioned fibers is done using a comparison microscope. See Appendix B and C for relevant information regarding fiber identification. In addition, a fiber reference collection is available containing both natural and synthetic fibers. A set of mounted reference fiber slides from McCrone is available in the laboratory. A set of unmounted reference fibers purchased from McCrone is also available in the laboratory.

- Cross-sectioning (if required)
 - There are many techniques available for cross-sectioning. This analysis should not be performed on the questioned fiber(s) if destruction of the evidence is an issue. The following three techniques are utilized in this laboratory:
 - Hardy Microtome
 1. Place the fiber in a small bundle of acrylic yarn fibers of a contrasting color.
 2. Place yarn bundle and questioned fiber into the slot in the microtome, close microtome to anchor the fibers in place.
 3. Cut off the excess fibers protruding on either side of the microtome with an unused razor blade.
 4. Place the microtome onto the microscope with the light directed through the fiber bundle.
 5. The cross-section of the fiber can then be observed and photographed.
 - Hardy Thin Cross-sectioning Device
 1. To use this method, a strand of fibers at least 1mm in diameter is necessary.
 2. Separate the parts of the cross-sectioning device.
 3. Release the lock and turn the swiveled bracket to a position transverse to the frame.
 4. Insert the fibers into the slot.
 5. Insert plates and press together.
 6. Cut off the surplus fibers that project from each side of the device.
 7. The fibers should now be flush with the plate.

8. Swing the bracket back into position and lock with the taper pin.
 9. Screw down micrometer screw until the fibers may be seen projecting slightly from the reverse side of the plate.
 10. Coat these projecting fibers with a thin solution of collodion and allow fibers to dry.
 11. Slice off dried film of collodion containing the fiber cross- sections and discard.
 12. Turn the screw head according to the thickness of the cross-section desired (usually 1/3 of a single gradation on the top of the screw).
 13. Apply a thin coating again to the exposed face of the fibers and allow to dry.
 14. Slice off the dried film of collodion containing the cross- sections and mount on a microscope slide using an appropriate mounting media.
 15. Place slide on microscope, observe and document cross- sections.
- Glue gun/pipette tip technique
 1. Place fiber in the open end of a pipette tip.
 2. Place a drop of hot glue into the opening, surrounding the fiber.
 3. Allow glue to dry and harden.
 4. Slice thin cross-sections of the fiber in the pipette tip with a sharp razor blade/scalpel blade.
 5. Mount cross-sections on a slide using an appropriate mounting media.
 6. Place slide on microscope, observe and document cross- sections.
 - By hand
 1. Place fiber on slide and place another slide or cover slip on top.
 2. Move the fiber to allow a small area to be sticking out from the top slide
 3. Using preferred cutting tool, cut the fiber as thin as possible
 4. Mount the cross-sections on a slide using an appropriate mounting media.
 5. Place slide on microscope, observe and document cross-sections.
- FTIR microscope
 - The infrared analysis of the questioned fiber can enable the analyst to identify the type of fiber based upon the molecular structure. In addition, this technique will enable the analyst to compare the structure of the known and questioned fibers to each other. This technique is particularly useful when attempting to differentiate between to similarly colored fibers of the same class.
 1. Prepare sample by flattening as much as possible.
 2. Place the fiber between two diamond compression cells.
 3. Run the sample on the FTIR according the manufacturer's suggestions.
 4. Search library for best match.

- Print all scans used in analysis and label appropriately for notes.
- Fiber dye analysis
 - This procedure is partially destructive in that it removes the dye from the fiber. It should only be utilized when destruction of the fiber is not an issue. Several types of extractants and eluent systems may be utilized. Occasionally a different system is needed due to fiber type or dye components. If an alternate method is required, this procedure can be found in the references listed at the end of this method. The following is the system used in this laboratory for most extractions:
 1. Known and questioned fibers must be extracted at the same time in the same conditions.
 2. Single fibers can be extracted in a short length of a fine capillary tube (internal diameter of about 1.5mm) that is sealed at one end.
 3. Place the fiber into the tube using a fine wire to push the fiber down the tube.
 4. Place approximately 10 µl of extractant (pyridine H₂O 4:3) into the tube using a syringe. Extractant should cover fiber.
 5. Heat seal top of capillary tube.
 6. Allow fiber to sit in extractant for 30 minutes at 100°C.
 7. Prepare a solution consisting of chloroform/methanol/acetic acid in the ratio of 70:20:10
 8. Set up a TLC tank with this eluent.
 9. Spot all samples on a TLC plate and place plates into tank.
 10. Run solvent front a minimum of 5cm. Run longer if time permits.
 11. Remove plate and mark solvent front.
 12. Dry plate and examine visually and with a UV light.
 13. Record results.

CONCLUSIONS

- Comparison
 - Association – The questioned and known fibers could share a common source.
 - Conditions – There are no significant differences in any of the macroscopic, microscopic, or chemical characteristics.
 - Elimination – The questioned and known fibers do not share a common source.
 - Conditions – Questioned and known fibers exhibit significant differences in their macroscopic, microscopic, or chemical characteristics.
 - Inconclusive – The questioned and known fibers show both similarities and differences such that no clear conclusion can be drawn.
 - Conditions – Analyst is unable to identify a fiber or do a meaningful or extensive comparison due to the small sample size, possible environmental decomposition, or other uncontrollable factors.
- Identification
 - Identification:
 - Synthetics – fiber is identified as an acetate, acrylic, nylon, polyester, etc.
 - Animal – fiber is identified as wool, angora, etc.
 - Vegetable – fiber is identified as cotton, linen, etc.

- Mineral – fiber is identified as fiber glass, glass wool, etc.
- Inconclusive – The analyst is unable to identify the fiber due to the small sample size, possible environmental decomposition, or other uncontrollable factors.
- Note: When an identification of fiber type is made AND reported, the technical reviewer will examine the fiber to the degree necessary to determine the generic fiber type.

References

The Textile Institute, "Identification of Textile Materials", seventh edition, New Jersey: Textile Book Service, 1975.

James Robertson, "Forensic Examination of Fibers", Lightning Power Co., Oregon: 1992.

Kathryn Hatch, "Textile Science", West Publishing Co., New York: 1993.

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F. Haphey, "Applied Fiber Science, Volume 1", Academic Press, New York: 1978.

F. Haphey, "Applied Fiber Science, Volume 2", Academic Press, New York: 1979.

F. Haphey, "Applied Fiber Science, Volume 3", Academic Press, New York: 1979.

OMNIC FTIR Software User's Guide, Version 3.0, Nicolet: 1996.

(Appendices Follow)

APPENDICES

Appendix A (see reference 1)

Refractive Indices of Fibers

<i>Fiber</i>	n_{\parallel}	n_{\perp}	<i>Birefringence</i>
ACETATE			
Diacetate	1.476	1.473	0.003
Triacetate	1.469	1.469	0
ACRYLIC			
Acrilan 36	1.511	1.514	-
Courtelle	1.511	1.514	-
Orlon 42	1.511	1.515	-
ARAMID			
Kevlar	>2.00		
ASBESTOS			
Chrysotile	1.50 - 1.56	-	varies
Amosite	1.64 - 1.69	-	varies
Crocidolite	1.68 - 1.71	-	varies
CHLOROFIBER			
Fibravyl	1.541	1.536	0.005
CUPRO			
Cuprammonium rayon	1.553	1.519	0.034
GLASS			
A-glass	1.542	-	-
E-glass	1.550	-	-
S-glass	1.523	-	-
C-glass	1.541	-	-
MODACRYLIC			

Dynell	1.535	1.533	0.002
Teklan	1.520	1.516	0.004
NYLON			
Nylon 11	1.553	1.507	0.046
Nylon 6	1.575	1.526	0.049
Nylon 6, 6	1.578	1.522	0.056
POLYESTER			
Terylene	1.706	1.546	0.160
POLYOLEFIN			
Polypropylene	1.530	1.496	0.034
Polyethylene	1.574	1.522	0.052
VISCOSE			
Normal tenacity viscose	1.542	1.520	0.022
High tenacity viscose	1.544	1.505	0.03
High wet modulus viscose	1.551	1.513	0.038
WOOL	1.557	1.529	0.010
COTTON	1.577	1.529	0.04
SILK			
Degummed	1.591	1.538	1.538
FLAX	1.58 - 1.60	1.52 - 1.53	0.06

Appendix B

Fiber Identification

Manmade: uniform continuous cross-section Natural:
shorter with discontinuities along length

Birefringence

Low - < 0.01

Medium - $0.01 - 0.05$

High - > 0.05

Isotropic Fibers

Glass Wool



Uniform diameter
Straight Colorless
n's ~ 1.52

Mineral Wool Exotic
shapes Irregular
diameters
May be colorless, gray, or brown
n's ~ 1.52 - 1.70

Triacetate (Arnel)



Not truly
isotropic n's ~
1.469
BR - 0.0001

Anisotropic Fibers

LOW

Acetate (+)

n_{\parallel} ~1.478
 n_{\perp} ~1.473



Acrylics (-)

n_{\parallel} ~1.511
 n_{\perp} ~1.515



Modacrylics

Dynel (+)
 n_s ~1.53



Scales
BR = 0.009

MEDIUM

Coniferous Wood

Flat fibers with 1
– 2 rows of pits

Viscose

n_{\parallel} ~1.55
 n_{\perp} ~1.52



Non-Coniferous Wood

Flat cells, usually
without pits; baggy
cells with many rows
of pits

Asbestos
Very fine
fibers

Straw (Nylon)

Lignified, serrated
cells, baggy cells

Mineral

Crysotile
 n_{\parallel} ~1.55
 n_{\perp} ~1.54

Jute

Rounded polygonal,
nodes lumen varies in
diameter

n_{\parallel} ~1.58
 n_{\perp} ~1.53

Cotton

Twists, no
extinction n ~1.58
 n_{\perp} ~1.53

Olefins

Polyethylene
 n_{\parallel} ~1.57
 n_{\perp} ~1.52
Polypropylene
 n_{\parallel} ~1.53
 n_{\perp} ~1.496

HIGH

Rayon, Silk

Rounded
trilobal cross
–over marks
 n_{\parallel} ~1.59
 n_{\perp} ~1.54

Flax (linen)

Rounded
polygonal
nodes
 n_{\parallel} ~1.59
 n_{\perp} ~1.525

Ramie

Long coarse
fibers, rounded
cross-section
nodes
 n_{\parallel} ~1.60
 n_{\perp} ~1.53

Hemp

Rounded
polygonal,
nodes
 n_{\parallel} ~1.59
 n_{\perp} ~1.53
Polyester
Any shape
 n_{\parallel} ~1.71
 n_{\perp} ~1.54

Polyamide (Nomex)

Cylindrical
 n_{\parallel} ~1.75 – 1.80+
 n_{\perp} ~1.67

Aramid

Cylindrical
 n_{\parallel} ~2.35
 n_{\perp} ~1.64

Polyamide

Any shape
 n_{\parallel} ~1.58
 n_{\perp} ~1.52



2.4 HAIR ANALYSIS

MATERIALS AND EQUIPMENT

Reagents and Supplies

- Permunt
- Xylene
- Glass slides
- Coverslips
- Forceps

Equipment

- Stereomicroscope
- Polarized light microscope
- Comparison microscope
- Sonicator

PROCEDURES

Evidence Handling Preliminary

Considerations

- Prior to analysis of evidentiary material, an evaluation of the important elements of each case should be obtained through communication with the submitting investigator and/or prosecutors. This evaluation should include an assessment of the evidence and its relevance.
- Special Considerations
 - Trace evidence can be contaminated during laboratory examination.
 - The analyst must ensure that tools, examination area, laboratory coat, and gloves are clean.
 - Examine each item of evidence on a clean piece of butcher paper.
 - The questioned items must be examined prior to the known items
 - Separate the examination of questioned and known reference items either by location or by examination of the items at different times.

Collection of Hair Evidence

- Collection of questioned hairs – Items examined for hair evidence are generally viewed visually for any apparent hairs. The following are appropriate collection techniques.
 - Forceps – Hairs may be located through a visual examination. If the hairs are large enough, collect them with forceps and place in a paper bindle or on a tape lift for storage.
 - Tape lifts – If the hairs are small or difficult to see, tape lifting is the recommended method for recovery. A tape lift can be patted over the surfaces of the clothes to collect the hairs. The hairs may be collected from the tape lift for examination at a later time.
- Collection of hair standards
 - Head: A known head hair sample consists of at least 25 hairs, preferable 100 hairs, from each of five different areas of the scalp (front, center, back, and both sides). The hairs will be obtained by pulling and finger combing.
 - Pubic: A known pubic hair sample consists of at least 20 hairs from all different areas of the pubic region. The hairs will be obtained by pulling and finger combing.
 - For collection of hair standards of deceased individuals at the autopsy, the hairs can be collected after the body has been washed.
 - Guidelines: these requirements are subject to the condition of the subject's hair. In some cases it may not be necessary or even possible to collect the suggested number of hairs. It also may be necessary to collect the hairs before the body is washed. Each situation will dictate whether trace evidence that might be present in the head hair is significant or not. Laboratory personnel can use their discretion as appropriate.
- Evidence Storage and Disposition
 - Evidence should be packaged to prevent cross contamination. If a hair is determined to be suitable for DNA analysis, store the hair in the freezer. If no DNA testing appears warranted, paper bindles placed in coin envelopes at room temperature is the preferred method for long-term storage.

Macroscopic Examination

- Examine the questioned hair macroscopically using low power magnification. The following characteristics should be noted if appropriate:
 - animal or human origin
 - condition of tip and root
 - length
 - somatic origin
 - disease
 - color
 - chemical alterations
 - contour
 - debris
 - cross-section
 - It is important to note the presence of any cells on the root in order to determine suitability of hair for nuclear DNA analysis.
- A similar examination is performed on the standard hairs. At this point in the analysis, the analyst makes a determination on similarity between the questioned

- hair and the standard hairs. In order to conclude a questioned hair is similar to a known hair standard, there must be no significant differences in the characteristics outlined above. If the questioned hair is similar to the standard hairs and has a root with visible cellular material, the hair is reported as similar to the standard hairs and recommended suitable for DNA analysis. If the questioned hair is similar to the standard hairs but does not have visible cellular material on the root, the hair is reported as macroscopically similar to the standard hairs but not appropriate for DNA analysis. If the questioned hair is not similar to the standard hair, the hair is reported as not similar to the submitted standards.
- A microscopic examination of the hair is only done if nuclear DNA analysis is not possible (no root present) and a more thorough examination of the hair is requested by the detective or district attorney. This request can be verbal or written and can occur either before or after the macroscopic examination has been completed. An analyst can also fill out a DNA request form or ask the DNA supervisor to assign the analysis, when the hair examiner thinks it is appropriate due to limited time or evidence.

Microscopic Examination

- Cleaning - Remove visible debris from the hair. If necessary, the hair(s) can be sonicated in deionized water to remove any additional debris.
- Mounting - Xylene can be used for a temporary mount and Permount can be used for a more stable mount. Each slide must be properly labeled for identification with the barcode number and case number or property tag and item number, analyst's initials, date, or other unique identifier.
- Observations - Examine the proximal, medial, and distal regions of the hair for the following characteristics:
 - Diameter
 - cortical texture
 - pigment size
 - ovoid bodies
 - pigment location
 - cortical fusi
 - pigment texture
 - cuticle
 - pigment amount
 - buckling
 - pigment color
 - diameter variation
 - medulla
 - condition of tip
 - condition of root
- Comparison
 - In order to conclude that a questioned hair is similar to a known hair standard there must be no significant differences in the macroscopic and microscopic characteristics outlined above. To do this type of comparison, place questioned and known hairs under the comparison microscope and compare side-by-side from root to tip. Document similarities and differences. Note:

Both the questioned and known hairs must be mounted in the same mounting media in order to make a comparison.

CONCLUSIONS

- Association – The questioned hair is similar in (color, length, contour, etc.) to the hairs in the submitted standard.
Conditions – The macroscopic/microscopic characteristics of the questioned hair can all be found within the range of characteristics of the hair standard.
- Non-association – Significant differences were noted in the (color, length, contour, etc.) between the questioned hair and the hairs in the submitted standards.
Therefore, these hairs are not similar.
Conditions – The questioned and known hairs show significant, unexplainable macroscopic/microscopic differences.
- Inconclusive – The questioned and known hairs show both similarities and differences such that no clear conclusion can be drawn.
- Suitable for nuclear DNA – The questioned hair contains cellular material and is suitable for nuclear DNA.
- Non-suitable for nuclear DNA – The questioned hair does not contain cellular and is not suitable for nuclear DNA.
 - Note: If the questioned hair is a head or pubic hair it is best to write appears to not be suitable for nuclear DNA.

References

John W. Hicks, "Microscopy of Hair", FBI, Washington D.C.: 1977.

Richard Saferstein, "Forensic Science Handbook", Prentice-Hall inc., New Jersey: 1982.

Walter C. McCrone and John G. Delly, "The Particle Atlas, Edition Two" Ann Arbor Science Publishers, Michigan: 1973.

APPENDIX A

Somatic Origin (**reference 2**)

Scalp: head hair; 100 - 1000 mm long, 25 - 125 mm diameter; 0.4 mm per day growth; small root; tapered tip, little diameter variation; various medullation; often with cut tips; may be artificially treated.

Pubic: pudental; 10 - 60 mm long; coarse diameter and prominent diameter variation and buckling; broad medulla; follicular tags common; asymmetrical cross-section twisted and constricted; may be straight, curved, or spirally tufted.

Vulvar: secondary pubic hair; finer and shorter than pubic hair; may be abraded.

Chest: pectoral; moderate to considerable diameter variation; long fine arch-like tip; usually longer than pubic hair.

Beard: facial hair, very coarse; 50 - 300 mm long; large root, irregular structure; often triangular cross-section; complex medullation; blunted or razor cut tip; grows 0.40 mm per day.

Axillary: arm pit; 10-50 mm long, grows 0.30 mm per day; coarse, blunt tip, abraded or frayed; usually straighter than pubic hair; many cortical fusi, sometimes yellow and bleached.

Eye brow: superciliary; 1cm long, 0.16mm per day growth; curved; relatively coarse for length; smooth curve with puntate tip and large medulla.

Eyelash: ciliary; less than 1cm long; short curved pointed hair.

Limb: leg and arm hair; 3-6 mm long, fine tips, irregularly medullated; often indistinctly and slightly pigmented

Ear: tragi, pinnae; downy.

Buttocks: anal hair; short blunted and abraded

Nose: similar to facial hair

APPENDIX B

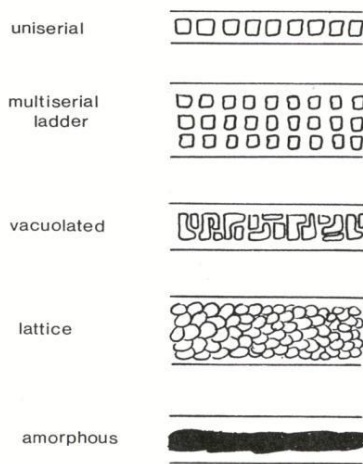
Animal vs. Human Hair Determination (reference 1)

Animal Hair

1. Guard hair and wool hair
2. Color banding
3. Tapered shaft
4. Coarse, structured medulla

Human Hair

1. Uniform hair sample
2. Even coloration
3. Even diameter
4. Thin, thready medulla

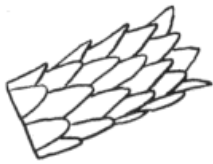


fragmentary
discontinuous
continuous

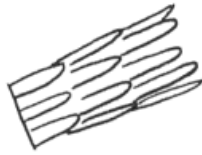


5. Pigment in or near medulla
6. Coarse, irregular granules
7. Prominent cuticle scales

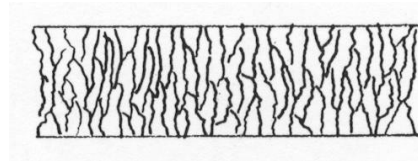
5. Pigment away from the medulla
6. Fine, regular granules
7. Smooth, flattened cuticle scales



CORONAL



SPINOUS



IMBRICATE

8. Root shape

9. Tapering tip

8. Small, regular root

9. Tips usually cut or blunt

ARCHIVED

2.5 IMPRESSION EVIDENCE EXAMINATIONS

MATERIALS AND EQUIPMENT

Materials

- Large plastic dishpan
- Soft bristle brush
- Methanol
- Hydrochloric acid
- Potassium ferrocyanide
- Aerosol sprayers
- Fingerprint powder (black and magnetic)
- Clear adhesive covers
- Identicator Kit – Special sensitized sheets, coater
- Ink
- Fingerprint roller
- Transparency film for plain paper copiers
- Roller transport film
- Silicone lubricant

Equipment

- Scanner
- Light source for oblique lighting
- Appropriate camera equipment
- ALS

PROCEDURES

Special Considerations

- The questioned impressions will be examined prior to examining the known footwear or tires.

Evidence Handling

- Preliminary Considerations – Footwear enhancement techniques can employ chemicals and reagents that are known carcinogens and/or hazardous substances. Therefore, the examiner should read the guidelines published by NIOSH and the Material Safety Data Sheets for all reagents and chemicals that are used.

Collection of Impression Evidence – Impression evidence can be received into the laboratory in many forms. These may include photographs, casts, electrostatic lifts, adhesive lifts, or the item of evidence with the impression on it. The following are guidelines for each of these general types of evidence:

- Photographs – If the impression evidence is in the form of printed photographs or digital images, enlargements of the images to actual size are printed for comparative analysis.
 - The photographs must have been taken from a 90° angle (a tripod is highly recommended) to avoid any size distortion, and a ruler must have been included in the photograph, in the same plane as the impression. The analyst must ensure that the 1:1 enlargement is the correct size by measuring

the ruler in the image against a similar ruler.

- Die Stone Casts – Fill large dishpan with water. Place cast in water and allow loose debris to fall off. Additional cleaning may be accomplished by using a soft bristle brush. Care should be taken not to remove any debris in the sole pattern or alter the pattern in anyway.
- Electrostatic Lifts – If the impression has been collected using an electrostatic lifter, the impression must be photographed immediately. Varying photographic techniques can be utilized to enhance the image, and photographs may be handled extensively while an electrostatic lift should not be.
- Adhesive Lifts – Lifts may be used for comparison purposes as is, or may be enhanced using various image enhancement techniques.
- Item of Evidence – If the evidence with the impression on it is submitted for examination, the impression should be photographed immediately. After photo documentation, various enhancement techniques may be utilized depending upon the type of impression.

Enhancement Techniques – Impressions may be left by a deposition of material (e.g. blood, dust, grease, etc.) or by a removal of material. Depending on the substrate the impression was left on and the material deposited or removed, a number of enhancement techniques can be used. These techniques include:

- Photography – Enhancement through specialized lighting and various photographic techniques is a non-destructive method and therefore, whenever possible, should be attempted first. The use of filters, UV light, infrared light, oblique light etc., are all options for image enhancement. This lab is staged with professional photographers to assist in this type of enhancement.
- Image Enhancement – Images can be enhanced using a scanner equipped with an image enhancement program. By varying the contrast, resolution, and color of the scanned impression, the analyst may be able to enhance the print. In addition, the image may be viewed as a positive or a negative impression.
- Chemical Enhancement – These techniques are destructive and should only be used in the event that all non-destructive techniques have been exhausted.
 1. Impressions made by dust – The enhancement is based on the reaction of $K_4Fe(CN)_6 \cdot 3H_2O$ with iron found in the dust, which gives a blue color. This procedure must be done in a fume hood. Safety glasses, gloves, and a lab coat must be worn.
 - a. Prepare a test impression using a non-evidence shoe with a dusty sole by stepping on a sheet of paper.
 - b. Mount test print on piece of stiff cardboard with tape or pushpins to prevent curling.
 - c. Prepare reagents.
 - d. Prepare a 1:1 mixture of $HCl:CH_3OH$ by slowly adding 50 mLs HCl to 50 mLs CH_3OH .
 - e. Prepare a 5% $K_4Fe(CN)_6 \cdot 3H_2O$ solution by adding 5.0 grams $K_4Fe(CN)_6 \cdot 3H_2O$ to 100 mLs distilled water. Stir until dissolved.
 - f. Using a separate aerosol sprayer for each reagent, spray the $HCl:CH_3OH$ lightly over surface of paper. Allow CH_3OH to evaporate.
 - g. Lightly spray surface of paper with ferrocyanide reagent until no further enhancement can be seen.
 - h. Dry with warm air.

- i. Repeat above steps with evidence impression.
- j. Photograph immediately using high contrast photography with a 1:1 scale.

Note 1: Luminol, amido black, and comassie blue can be used for enhancement of impressions in blood. The procedures can be found in the Crime Scene Reconstruction and Crime Scene Unit Manuals.

Note 2: Chemical enhancement procedures for other materials may be found in the literature and can be used after in-house validation studies.

Collection of Standards – There are a variety of techniques that can be used to create test impressions. It is important to try and duplicate the type of movement you suspect created the questioned print when making a test print. The shoe should be placed on the analyst's foot and a standing, walking, jumping, running, etc. print can be created. Make test prints of shoes utilizing one of the following techniques:

- Shoes
 - Adhesive lift with powder
 1. Make print using dust or fingerprint powder.
 2. Ensure that shoe sole is completely dry.
 3. Evenly dust with fingerprint powder.
 4. Shake off loose powder.
 5. Remove protective cover from clear adhesive material and press sole of shoe carefully against adhesive surface.
 6. Press adhesive sheet onto all areas of shoe sole.
 7. Peel adhesive away from shoe and place on protective sheet.
 8. If enough dust is initially present on the shoe sole, fingerprint powder need not be used.
 - Identicator Kit
 1. Wash and dry sole of shoe.
 2. Place coater on the floor with a sensitized sheet next to it, treated side up.
 3. Press shoe firmly on coater and coat entire sole.
 4. Press shoe firmly on sensitized paper.
 - Ink and Paper
 1. Using fingerprint roller, deposit ink evenly over sole of shoe.
 2. Make impression on white paper.
 - Roller Transport Film
 1. Ensure that shoe sole is completely dry.
 2. Evenly dust with fingerprint powder.
 3. Shake off loose powder.
 4. Dip sponge into water; wipe sponge over surface of roller transport film to wet.
 5. Use squeegee to remove any excess water from film.
 6. Walk sole of shoe across film, leaving a powder print.
 7. Allow to dry. This technique is best used in conjunction with procedure
 - a. Dust sole of shoe, walk across adhesive surface, then walk

immediately across the damp film surface.

- Tires – Tires should remain mounted on the original vehicle with the original air pressure if possible.
 - Ink and white paper stock (exam paper is too thin and will wrinkle as the tire moves over it).
 1. The tire can be rolled or sprayed with ink.
 2. The tire should be evenly coated with ink.
 3. Drive/roll the entire circumference of the tire using a reference point marked on the side of the tire onto white paper.
 4. Label impression with direction tire was traveling, location of tire on vehicle, initials, case number, and date.
 - Finger print powder and petroleum jelly
 1. Spray or rub tire with silicone lubricant or petroleum jelly
 2. The entire circumference should be sampled using a reference point marked on the side of the tire.
 3. Drive/roll the entire circumference of the tire over clear colorless polyester sheeting attached to poster board or similar surface.
 4. Powder impression with magnetic powder
 5. Remove excess powder
 6. Spray impression with hair spray or any clear acrylic spray, and allow it to dry
 7. Label impression with direction tire was traveling, location of tire on vehicle, initials, case number, and date.
 8. Take care not to reverse the overlay. Roll the polyester sheet with the impression between exam paper to protect the impression from damage.
 9. Inked tires can be rolled onto clear wet media film. See reference “Tire Tread and Tire Track Evidence.”

EVALUATION AND COMPARISON

- Comparison – Using the test impressions and overlays from the suspect shoes and the photographs, die stone casts, or impressions from the scene, the following comparisons should be made.
 - Size, shape, and sole or tread pattern.
 - Wear characteristics and manufacturing defects.
 - Randomly acquired characteristics (cuts, gouges, scratches, etc.).
- Identification of Tire Type – Occasionally, the analyst will be requested to identify the tire or tire types that could have made a questioned impression. At this time we do not possess any current *Tread Design Guide* books to evaluate the brand of tire.

CONCLUSIONS

- Identification – the known shoe/tire made the questioned impression.
Condition – If the class characteristics (size, pattern, and shape) and wear characteristics are similar, and randomly acquired characteristics found on the shoe/tire are reflected in the questioned impression, then an identification can be made.
- Association – The questioned impression is similar in sole/tread pattern, shape, size, and wear characteristics to the known shoe/tire. The known shoe/tire or a shoe/tire of similar size, shape, and pattern could of have made the impression.
Condition – If class characteristics and/or wear characteristics are similar on the shoe/tire and are reflected in the question impression, however no randomly acquired characteristics are found, an association can be made.
 - Note: If only a few of the class characteristics can be compared and are similar then, the analyst can determine that a limited association exists between the questioned impression and the known shoes/tire.
- Exclusion – The known shoe/tire is excluded from making the questioned impression.
Condition – If the class characteristics and/or wear characteristics are dissimilar on the shoe/tire to the class characteristics and/or wear of question impression, an exclusion can be made.
 - Note: Analysts must be careful when considering wear characteristics, the time of collection of the known shoes relative to the time of incident is important and should be known prior to analysis.
- No Conclusion – The questioned impression lacked sufficient details; no conclusion can be made
Condition – If insufficient details or lack of a proper scale are present with the question impression then no conclusion can be made.
- Inconclusive – The questioned impression is similar in sole/tread pattern to the known shoe/tire. Due to no scale being present, no conclusions can be made on shape, size, and wear. The comparison of the question impression and known shoe/tire is inconclusive. The known shoe/tire cannot be included or excluded from making the questioned impression.
Condition – If some class characteristics and/or wear are similar and other characteristics are dissimilar or can't be determined then an inconclusive conclusion can be made.

References

William J. Bodziak, "Footwear Impression Evidence", New York: CRC Press, 2000. Peter

McDonald, "Tire Imprint Evidence", Ann Arbor: CRC Press, 1993.

John R. Abbott, "Footwear Evidence", Springfield, Illinois: Charles C. Thomas, 1964. "Tread Design Guide", (several editions) Boca Raton, Florida: Tire Guides Inc.

"The Tread Assistant, 1999 edition", Tire Guides Inc, The Pearl Group Inc.

William Bodziak, "Tire Tread and Tire Track Evidence," New York: CRC Press, 2008.

MATERIALS AND EQUIPMENT

Reagents and Materials

- Glass Slides
- Cover slips
- Refractive index liquids
- TLC plates (silica gel)
- Developing tank with lid
- Acetone
- Chloroform
- Ether
- Methylene chloride
- Methanol
- Ethanol
- Barium chloride
- Acetic acid (conc.)
- Hydrochloric acid (conc.)
- Diphenylamine
- Silver nitrate
- Ammonium hydroxide
- Sodium hydroxide
- Griess reagent
- Sulfanilic acid (conc.)
- Sulfuric acid (conc.)
- Zinc (powdered)
- Nitron reagent
- Methylene blue
- Zinc sulfate
- Nitric acid
- Potassium nitrate
- Sodium azide
- Nessler reagent
- Potassium test paper
- Lead acetate paper
- Iodine
- Pyridine
- Spot plates
- Tungsten needles
- Injection syringes
- Naphthol
- Capillary tubes
- Sieves
- Beakers
- Carver press
- Pellet die
- Spatula

- Potassium bromide
- Test tubes
- Explosive standards
- Distilled or deionized water

Equipment

- Stereo microscope
- Polarized light microscope
- FTIR microscope
- GC-MS
- SEM-EDS
- UV light source
- Hot plate

PROCEDURES

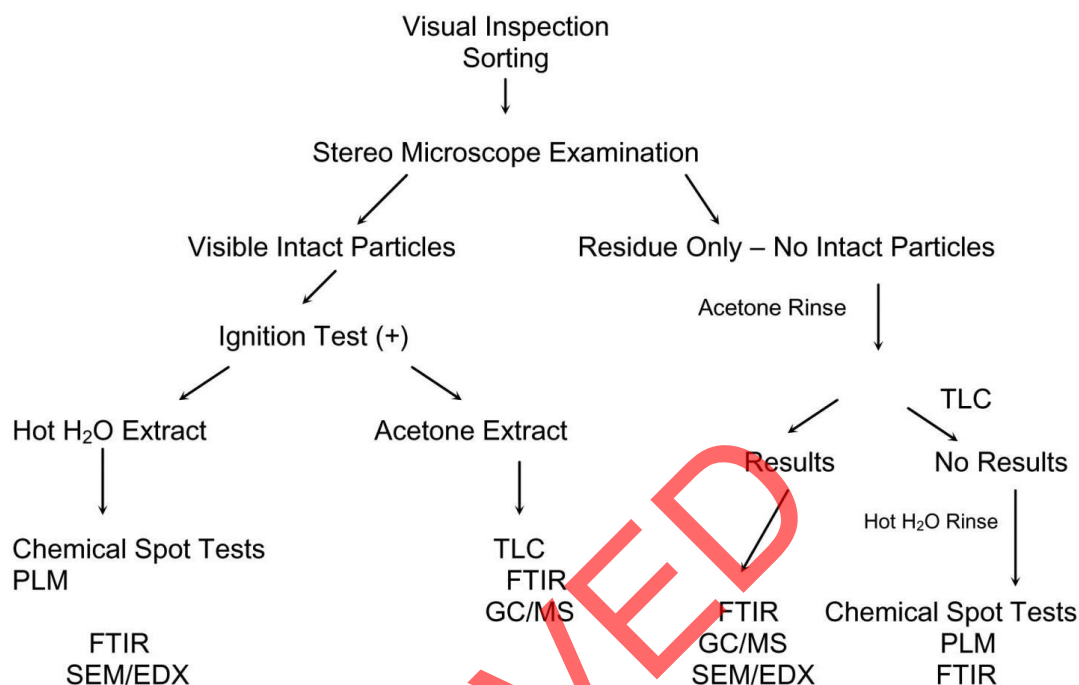
Evidence Handling/Safety – Preliminary considerations

- The type of explosive suspected and the condition of the evidence will determine the analytical scheme. This procedure is meant to be used as a guideline for explosive examinations but the analyst must determine the best analytical approach on a case- by-case basis. Other solvents, TLC systems, and extraction techniques may be utilized to optimize results.
- Trace evidence examinations for explosives can employ chemicals and reagents that are hazardous substances. Therefore, the examiner should read the guidelines published by NIOSH and the Material Safety Data Sheets for all reagents and chemicals that are used. Use appropriate personal protective gear and a fume hood when handling hazardous substances.
- In addition to the chemicals and reagents used in the analysis, the explosives themselves should be handled with care. If working with unexploded material, always use the smallest amount of material possible. Do not subject the material to violent shock, heat, fire, other explosives, or accelerants. Do not work on any explosive device before it has been dismantled or disarmed by the MAST unit.
- Due to safety considerations, no intact device will be brought into the lab.

Evidence Examination

- Visually examine the evidence with the stereomicroscope and attempt to determine the type of explosive, i.e. smokeless powder, black powder, high explosive, by its physical appearance.
 - Note the general appearance, color, morphology, and presence of metal, crystalline inclusions, homogeneity, and ignition characteristics.
 - Document by photographs, drawings, or written descriptions any device or parts of a device.
 - Remove any suspected explosive particles. This may require the use of sieves if the majority of the sample is soil or sand. Picking out particles of similar types using the stereomicroscope and forceps can also be used to isolate the particles for further testing. Sieves can also be used to assist in the search for device parts. An ignition test on the particles may be performed if a large number of particles are present. Use a tungsten needle or a glass slide and heat with an open flame.
 - If no particles are found, a wash using acetone or chloroform, and a subsequent wash using hot water can be used to collect any residues of smokeless powder, high or primary explosives, black powders, or flash powders.

Explosive examination flow chart:



Analytical Techniques

- Polarized Light Microscopy – Isolated Particle Examination Particles removed from a sample, using the above techniques, can be examined by mounting in an appropriate medium. Note the optical characteristics and any other observations.
- Polarized Light Microscopy – Recrystallization
 - Particle removed from the sample:
 - Place a small drop of distilled water 5-7mm in diameter in the center of a clean microscope slide.
 - Add the particle to be investigated into the droplet.
 - Crush the crystal in the drop with a tungsten needle or drawn glass rod and stir the drop.
 - Push any crusts that form back into the center.
 - Continue as above until well formed crystals begin to grow.
 - Characterize the optical properties of the crystals observed.
 - The refractive indices can be determined using refractive index liquids on any isolated crystals or original particles.
 - Water extracts:

- Evaporate to a small volume.
- Transfer to a microscope slide and proceed with step 4 above.
- Thin Layer Chromatography
 - The acetone extract of any suspected smokeless powder particles or high explosives or the acetone wash of a device is spotted on a silica gel plate.
 - Known standards and an acetone blank should be run on each plate.
 - CHCl_3 can be used as the developing reagent. Allow to develop up to 10 cm. Note or mark the solvent front. Allow to air dry.
 - Visualize with 5% DPA solution in ethanol (5 grams DPA in 100 mLs ethanol) followed by UV irradiation 5 to 15 minutes. More information can be obtained by spraying with concentrated H_2SO_4 following UV irradiation. Determine R_f values for standards and unknowns. Compare to literature values.
- Chemical Spot Tests
 - The hot water extracts of any suspected black powder or pyrotechnic particles of the hot water wash of a device can be tested for the presence of anions and cations
 - Known standards and blanks must be run with every spot test. The results will be recorded in the case notes.
 - Anion and Cation tests: (Note: Many of these reactions can be done directly on the microscope slide.) Add a few crystals of the dry chemical (standard) to the slide and a microdrop of the liquid used to dissolve the chemical adjacent to the dry chemical. Bring them together with the tip of a probe. Watch the reaction under the microscope. Record the results. Add your unknown (either wet or dry sample) to the same slide in an area adjacent to the test reagent. Bring the two together using a probe. Watch the reaction under the microscope. Record the results. If a known sample is provided complete the test on the same slide as well.
 - Carbonates – BaCl_2 / Acetic acid
 - BaCl_2 / Acetic acid – 1-2 drops of unknown solution are placed in a test tube or black spot plate. One drop of BaCl_2 is added. A white precipitate that redissolves in concentrated acetic acid indicates the presence of carbonates.
 - Chlorates – Diphenylamine (DPA)
 - Diphenylamine (DPA) – 1-2 drops of unknown solution are placed in a white spot plate. Add 1-2 drops of DPA solution. An immediate deep blue color indicates the presence of an oxidizer. Note: Many oxidizing ions will color the solution blue, including nitrates, nitrites, chlorates, and ferric ions.
 - Chlorides – AgNO_3 / NH_4OH
 - AgNO_3 / NH_4OH – A few drops of AgNO_3 are added to 1-2 drops of unknown solution in a black spot plate. A white, curdy precipitate that is soluble in NH_4OH indicates the presence of chlorides. Note: Sulfates may form a white precipitate. As sulfates are insoluble in basic solutions, if the white precipitate redissolves in concentrated NH_4OH , it confirms the presence of chloride. Other

halides react similarly, although their precipitates vary from off-white to yellow. Basic solutions form a brown interference, another aliquot of the test solution can be acidified with dilute HNO_3 and retested.

- Nitrates – Modified Griess / Zn; Nitron; DPA
 - Griess – 1-2 drops of unknown solution are placed in a white spot plate. Add 2 drops of reagent I and 2 drops of reagent II, add a small amount of powdered zinc. A pink/red color after the addition of zinc indicates the presence of nitrates.
 - DPA – 1-2 drops of unknown solution are placed in a white spot plate. Add 1-2 drops of DPA solution. An immediate deep blue color indicates the presence of an oxidizer.
 - Nitron – 1-2 drops of unknown solution are placed in a black spot plate. A drop of Nitron reagent is added. The immediate formation of thin, needle-like crystals indicate the presence of nitrate. Chlorates and perchlorates also form crystals, which are wider blade-like crystals. Chlorate crystals take the longer to form.
- Perchlorates – Nitron; methylene blue
 - Methylene blue – 1-2 drops of the unknown solution are placed in a white spot plate. Add 2 drops of drops ZnSO_4 , 2 drops KNO_3 , and one drop of methylene blue solution. The formation of a purple color indicates the presence of perchlorates.
 - Nitron – See Nitrates section.
- Sulfates – BaCl_2 / Acetic acid
 - BaCl_2 – 1-2 drops of the unknown solution are placed in a test tube or black spot plate. One drop of BaCl_2 is added. A white precipitate that does not redissolve in concentrated acetic acid indicates the presence of sulfates.
- Sulfides – Sodium azide; Lead acetate paper
 - Sodium azide – 1-2 drops of unknown solution are placed in a white spot plate. A drop of the brown sodium azide solution is added. Decoloration with the evolution of gas indicates the presence of sulfides. Note: Thiosulfates and thiocyanates react similarly. For dilute solutions, look for the evolution of gas bubbles at the base of the depression. Basic solutions may decolorize the azide solution, but they do not cause the evolution of gas. The solution has a long shelf life. Use extreme caution disposing of bulk azide solutions since they may be explosive. DO NOT WASH DOWN DRAINS AS HAZARDOUS COPPER AZIDE OR OTHER METAL AZIDES MAY FORM.
 - Lead Acetate Paper – Add a drop of the unknown solution to lead acetate test paper. A brown-black stain that forms instantly, indicated the presence of sulfide ions.
- Ammonium – Nessler
 - Nessler – 1- 2 drops of unknown solution are placed in a white spot plate. 1-2 drops of Nessler reagent are added. An orange-brown precipitate indicates the presence of the ammonium ion. Note: Many organic solvents, including methanol, acetone, and

ethanol interfere with the Nessler test. Organic solvents should be completely evaporated before this test is performed. Ag, Pb, and Hg reportedly interfere.

- Potassium – Potassium test paper
 - Potassium test paper – A drop of unknown solution is placed on the test paper. Add a drop of dilute nitric acid (6% or 1:10) to the paper where the unknown spot is, or dip the paper in the acid. The paper remains orange if potassium is present but turns lemon-yellow if potassium is absent.
- Sulfur – Pyridine/ 2N NaOH
 - Pyridine / 2N NaOH – A small piece of suspected sulfur is placed in a test tube with several mLs of pyridine and heated until dissolved. Remove from heat and add a few drops of NaOH. A brown-yellow or green-blue liquid interface indicates the presence of sulfur.
- Sugar – Naphthol
 - Naphthol – 1-2 drops of unknown solution are placed in a white spot plate. One drop of naphthol reagent and 2 drops of concentrated H₂SO₄ are added. A blue, purple-blue, or pink color indicates the presence of sugar. Note: Fructose forms a similar purple-blue color. Glucose and maltose form pink colors at the sulfuric acid interface. Nitrates form a yellow-green color that obscured the color test. Keep the naphthol reagent from light.
- FTIR
 - Microscope
 - Soak a suspected smokeless powder particle overnight in approximately 20µL of methanol. Other solvents may also be used, such as CH₂Cl₂, ether, or acetone. Place a few microliters of the resultant solution on a copper or gold slide or other IR reflective surface and allow to evaporate.
 - Analyze on the FTIR microscope set on reflectance.
 - Prepare and analyze known standards in the same way
 - Extract plastic explosives with appropriate solvents and compare plasticizers using the FTIR.
 - Bench
 - KBr pellets or PTFE cards can be used to analyze solvent (methanol, acetone, methylene chloride) extracts of organic explosive compounds in relatively high concentrations.
 - The solvent extract is place directly on the PTFE card and allowed to evaporate.
 - A KBr pellet is prepared by adding the solvent extract to the KBr prior to grinding. Grind the KBr and press into a pellet. Micro pellets or regular 13mm pellets can be made. Pellets can also be made with isolated crystal particles of whole unexploded particles of black powder or with the dried residue from the water extracts.
 - Analyze cards or pellets using the IR bench set on transmission.

- Prepare and analyze appropriate standards in the same way as the samples.
- GC/MS
 - After extracting smokeless powder or high explosive particles in methanol, the extracts can be analyzed direction using GC/MS.
 - Inject approximately 1µl of methanol extract into the injection port.
 - Analyze known standards and blanks.
 - Use the following or other conditions:
 - Initial temp. – 80°C ramped to 230°C at 12°C/min.
 - Final temp. – 230°C
 - Injector temp. – 220°C
- SEM/EDS
 - Analysis can be done on unknown powders, individual particles, or water washes after evaporation.
 - Prepare a stub with the unknown sample.
 - Scan sample and identify elements in all types of particles present on the stub.
 - Prepare known samples if possible in the same way and analyze.

INTERPRETING RESULTS/REPORTING

- The following components can be reported if the unknowns are morphologically and chemically similar to standard preparations of the compounds listed below:
 - Black Powder – contains KNO₃, S, C
 - (Post blast black powder – K₂CO₃, K₂SO₄, K₂S, K₂S₂O₃, KHSO₄, KHCO₃, KSCN, KNO₃, KNO₂, NH₄CO₃, S, C)
 - Pyrodex – contains KClO₄, KNO₃, S, C, cyanoguanidine, and sodium benzoate
 - (Post blast pyrodex – K₂CO₃, K₂SO₄, KCl, S, KHSO, KHSO₃, K₂S, cyanoguanidine, sodium benzoate, KNO₃, KNO₂, KClO₄, KHCO₃, K₂S₂O₃, C)
 - Golden Powder – contains KClO₄, KNO₃, ascorbic acid, erythorbic acid
 - Flash powder – contains KClO₄ or KClO₃, S, Al
 - (Post blast flash powder – KCl, Al, K₂SO₄, KClO₄ or KClO₃, Al₂O₃, KHSO₄, KHSO₃, AlCl₃, Al₂SO₄, KAl(SO₄)₂)
 - Road flares – KClO₄, Sr(NO₃)₂, S, wood chips
 - Match heads – P, KClO₃
 - Smokeless powder:
 - Single base – nitrocellulose
 - Double base – nitrocellulose, nitroglycerine, ethyl centralite, rosin,
 - Polyester
 - Triple base – nitrocellulose, nitroglycerine, and nitroguanidine.
 - PETN – pentaerythritol tetranitrate C(CH₂ONO₂)₄

- RDX – cyclotrimethylene trinitramine $(\text{CH}_2)_3\text{N}_3(\text{NO}_2)_3$
- EGDN – ethylene glycol dinitrate $\text{C}_2\text{H}_4\text{N}_2\text{O}_6$
- SEMTEX – RDX/PETN
- ANFO – NH_4NO_3 , fuel oil
- C-4 – RDX
- Dynamite – typically NG, EGDN, S, NaNO_3 , $\pm\text{NH}_4\text{OH}$; synthetics such as gel explosives, NCN, MMAN, etc.
- TNT – 2,4,6-trinitrotoluene, $\text{C}_7\text{H}_5(\text{NO}_2)_3$
- DNT – 2,4-dinitrotoluene $\text{C}_7\text{H}_6(\text{NO}_2)_2$
- Tetryl – tetryl-2,4,5-trinitrophenylmethylnitramine
- Lead azide – $\text{Pb}(\text{N}_3)_2$
- Lead styphnate – $\text{PbO}_2\text{C}_6\text{H}(\text{NO}_2)_3$
- Mercury fulminate – $\text{Hg}(\text{ONC})_2$
- Solidox – NaClO_3 , sand, fiberglass, Fe
- Ammonium nitrate fertilizer – NH_4NO_3
- Sugar/chlorate mixtures – NaClO_4 , sucrose
- Detasheet / Flex explosives – PETN or RDX
- Primacord – PETN
- In addition to reporting the chemical compounds that were detected, the analyst can give investigative information regarding the device itself. A description of the pipe and end caps, electrical components, fusing, etc. should be included in the report.

CONCLUSIONS

- Identification – The sample contains PETN. PETN was detected.
Condition – Sample morphology and chemical composition is consistent with double base smokeless powder.
- Pyrotechnics – The sample is morphologically and chemically similar to a pyrotechnic mixture, which typically contains, _____, and _____.
Condition – Sample is similar chemically and morphologically to a known pyrotechnic mixture
- Improvised explosive mixtures – The sample contains X, Y, and Z and is consistent with an improvised explosive mixture
Condition – sample contains material similar with knowns improvised explosive mixtures
- Inconclusive – The fragment in Item contained traces of potassium carbonate. No unconsumed explosive particles were detected on the fragments, however potassium carbonate is a reaction product of black commercial powder.
Condition – a reaction product was found, but no explosive product was found
- Negative results – No explosives or explosive residues were detected
Condition – no explosive or explosive residues were detected

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(Appendices follow)

APPENDICES

TLC Rf values for CHCl₃ and other systems

COLOR DEVELOPMENT*

Compound	Stage 1, DPA	Stage 2, UV Light	Stage 3, H ₂ SO ₄	Rf in CHCl ₃
2,4-DNT	yellow	Yellow	color fades ^a	0.8
2,6-DNT	yellow	Yellow	color fades ^{ab}	0.85
EGDN	NCD	gray to gray-green	blue-gray	0.8
NC	NCD	gray to gray-green	blue to blue-gray	0
NG	NCD	gray to gray-green	blue-gray	0.7
NS	NCD	gray to gray-green	blue to blue-gray	0
RDX	NCD	gray to gray-green	blue-gray	0.15
PETN	NCD	gray to gray-green	blue to gray-green	0.7
Tetryl	brown	Brown	yellow to blue-gray ^c	0.5
TNT	orange-brown	orange-brown	color fades ^a	0.75

* Table from Parker, et.al., "Analysis of Explosives...", Part 2:..." (See References)

NCD = no color development

^a Dark spot visible under UV light.

^b Turns pink in center after re-exposure to UV light.

^c Yellow in center with blue-gray edge.

Rf Values in Various Solvent Systems*

Compounds

Solvent Systems	2,4-DNT	2,6-DNT	EGDN	NC	NG	NS	RDX	PETN	Tetryl	TNT
CHCl ₃	0.8	0.85	0.8	0	0.7	0	0.15	0.7	0.5	0.75
CCl ₄ /Dichloroethane (4:1)	0.5	0.5	0.5 0.6 ^a	0	0.35 0.4 ^a	0	0.02	0.3	0.1	0.45
Benzene/Hexane (1:1)	0.5	0.55	0.5 0.6 ^a	0	0.35 0.4 ^a	0	0.02	0.3	0.1	0.45

Xylene/Hexane (3:2)	0.4	0.45	0.5 0.6 ^a	0	0.35 0.4 ^a	0	0.03	0.3	0.15	0.4
Benzene	0.7	0.75	0.75 0.9 ^a	0	0.65, 0.8 ^a , 0.3	0	0.1	0.65	0.45	0.75
Hexane/Acetone (4:1)	0.4 0.4 ^b	.45	0.4 0.5 ^a	0	0.4 0.5 ^a	0	0.2	0.4	0.3	0.4 0.44 ^b
CHCl ₃ /Acetone (1:1)	0.95	0.9	0.9	0	0.9	.0 ^c	0.7 0.47 ^b	0.95 0.69 ^b	0.9 0.66 ^b	0.9

^a Hoffman and Byall (See References) ^b Jenkins and Yallop (See References) ^c Continuous streaking from origin.

* Table from Parker, et.al., "Analysis of Explosives.., Part 2:.." (See References)

B. Spot Test Reagent Preparations

- BaCl₂ - 5 grams of BaCl₂ in 100 mls H₂O.
- Diphenylamine (DPA) - 1 mg of diphenylamine is dissolved in 10 mls concentrated H₂SO₄. Store away from light. The solution is highly corrosive.
- AgNO₃ - Commercially available in a 10% solution. Alternately, a 5% solution can be prepared with 5 grams AgNO₃ in 100 mls H₂O. Store away from light.
- Modified Griess -
- *Reagent I* - 1 gram sulfanilic acid dissolved with warming in 100 mls of 30% acetic acid.
- *Reagent II* - 1 gram N-1-naphthylethylenediamine dihydrochloride is dissolved in 100 mls of 30% acetic acid/70% ethanol.
- Zinc dust - commercially available (flammable when exposed to air - dispose of the dust following appropriate safety precautions.).
- Nitron - 1 gram Nitron (diphenylenedianilohydrotriazole) is dissolved in 20 mls 88% formic acid.
- Methylene blue -
- Dissolve 0.03 grams methylene blue in 100 mls H₂O. Prepare a saturated solution of zinc sulfate. Dissolve 20 grams of KNO₃ in 100 mls H₂O.

- Sodium azide – 3 grams of sodium azide and 1.3 grams of iodine are dissolved in 100 mls H₂O. DO NOT WASH DOWN DRAINS AS HAZARDOUS COPPER AZIDE OR OTHER METAL AZIDES MAY FORM.
- Nessler – Commercially available. Alternately, dissolve 10 grams mercuric iodide and 5 grams potassium iodide in 50 mls of H₂O. Add a solution of 20 grams KOH in 50 mls H₂O. Let the solution stand for several days. Decant and store the supernatant liquid in a brown bottle.
- Potassium test paper – Commercially available.
- Lead Acetate paper – Commercially available.
- Naphthol – 15 grams of 1-naphthol is dissolved in 100 mls ethanol.
- Sodium Hydroxide (2N) – 4 grams of NaOH is dissolved in 50 mls of distilled water.

C. Compositions – pyrotechnics (fireworks)

- Oxidizers:
 - KNO₃
 - KClO₃
 - KClO₄
 - NH₄ClO₄
 - Ba(NO₃)₂
 - Ba(ClO₃)₂
 - Sr(NO₃)₂
- Fuels:
 - Al
 - Mg
 - Ti
 - Charcoal
 - S
 - Sb₂S₃
 - Dextrin
 - Polyvinyl chloride
 - Red gum
- Binders:
 - Dextrin
 - Red gum
 - Synthetic Polymers

- Special Effects:
 - Red flame: $\text{Sr}(\text{NO}_3)_2$, $\text{Sr}(\text{CO}_3)_2$
 - Green Flame: $\text{Ba}(\text{NO}_3)_2$, $\text{Ba}(\text{ClO}_3)_2$
 - Blue flame: CuCO_3 , CuSO_4 , CuO
 - Yellow Flame: $\text{Na}_2\text{C}_2\text{O}_4$, Na_3AlF_6 (cryolite)
 - White flame: Mg , Al
 - Gold sparks: Fe filings, charcoal
 - White sparks: Al , Mg , Al-Mg alloy, Ti
 - White effect: $\text{KC}_7\text{H}_5\text{O}_2$, $\text{NaC}_7\text{H}_5\text{O}_3$
 - White smoke: KNO_3/S mixture
 - Colored smoke: $\text{KClO}_4/\text{S}/\text{organic dye}$ mixture
- Compositions:
 - Red Star: KClO_3 , $\text{Sr}(\text{CO}_3)_2$, Red gum, dextrin
 - Blue Star: KClO_3 , NH_4ClO_4 , CuCO_3 , Red gum, dextrin
 - Green Fire: NH_4ClO_4 , $\text{Ba}(\text{NO}_3)_2$, fine sawdust, shellac
 - Cone Fountain: KNO_3 , Fe , S , C , Al , Stearic acid

D. Refractive Indices of some water-soluble explosive compounds.

- Isotropic
 - $\text{Ba}(\text{NO}_3)_2$ 1.571 KCl 1.4904
 - $\text{Pb}(\text{NO}_3)_2$ 1.781 NaCl 1.5443
 - $\text{Sr}(\text{NO}_3)_2$ 1.587 $\pm .001$ NaCN 1.452
 - NaClO_3 1.518 KCN 1.410 $\pm .003$
1.515
- Anisotropic
 - Low Birefringence
 - NH_4ClO_4 1.4818 K_2SO_4 1.4935
1.4833 1.4947
1.4881 1.4973
 - KClO_4 1.4731 SrSO_4 1.6215
1.4738 1.6232
1.4769 1.6305
 - Medium Birefringence
 - NaClO_4 1.4730 K_2CO_3 1.426 $\pm .004$
1.4606 1.531 $\pm .002$
1.4617 1.541 $\pm .002$
 - KClO_3 1.415 CaCl_4 1.600
1.517 1.605
1.523 1.613

○ High Birefringence			
▪ NaNO ₃	1.5874	SrCO ₃	1.5199
	1.3361		1.6666
			1.6685
▪ KNO ₃	1.335	NaCNS	1.545 ± .005
	1.5056		1.625 ± .005
	1.5064		1.695 ± .005
▪ NH ₄ NO ₃	1.413	KCNS	1.532 ± .003
	1.611		1.660 ± .005
	1.637		1.730 ± .005

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2.7 PAINT ANALYSIS

MATERIALS AND EQUIPMENT

Reagents and Supplies

- Permunt
- Xylene
- Acetone
- Chloroform
- DI water
- Diphenylamine reagent
- Forceps
- Tape lifts
- Scraping tool (dull-bladed knife, putty knife/scrapper, or similar tool)
- Butcher paper
- Petri dishes
- Glass slides
- Coverslips
- Tungsten needles
- Scalpel
- Razor blades
- Diamond compression cells
- KBr
- Graphite sampling mounts for SEM-EDS analysis

Equipment

- Stereomicroscope
- Polarized light microscope
- FTIR microscope and accessories
- SEM/EDS

PROCEDURES

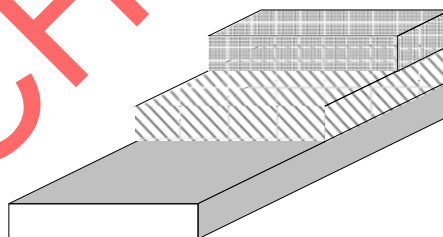
- Evidence Handling/Safety
 - Special Considerations - The analyst must be aware that trace evidence is susceptible to contamination during laboratory examination. The analyst must ensure that the tools, examination area, laboratory coat, and gloves are clean. Each item of should be examined on a new piece of paper.
 - Questioned evidence is examined prior to any known or comparison samples.
 - Separate the examination of questioned and known reference items with by location or by examination of the items at different times.
- Safety
 - Some of the methods described employ dangerous chemicals, temperatures, and radiation. It is the responsibility of the analyst to follow appropriate health and safety practices.

- Evidence Examination
 - General Considerations – Laboratory analysis currently consists of identification and comparison of paint samples. No possible make/model information is provided at this time.
 - It is the goal of this procedure to provide a reasonable approach to paint comparison. The methods referenced in this procedure need to be validated by each analyst. Paint should not be approached with a rigid analytical scheme as each method has strengths and limitations. It is up to the analyst to evaluate both the samples and the analytical technique applied.
 - This method is specific to the analysis of automotive paint. Architectural paint requires different analytical techniques and requires a difference approach. For information regarding the analysis of architectural paints see appendix A.
 - The paint samples should initially be evaluated for the possibility of a physical match. This is the most conclusive association. Edges and surface striae having unique characteristics must be documented using appropriate means.
 - Paint samples are compared by their physical and chemical characteristics. The physical characteristics include color, layer sequence and thickness, and surface and layer features (striations). The chemical characteristics include pigments, binders, and additives.
 - The evaluation and documentation of the differences between known and questioned samples is the focus of paint comparison. The existence of a range of variation in samples of a known source needs to be recognized. The examiner's goal is to assess the significance of any observed differences. The absence of significant differences implies a common origin. The strength of association determination is dependent on the type and number of corresponding features.
- Collection/Documentation of Evidence
 - Sketch and/or photograph the item of evidence, note any damage and the location of obvious paint transfer.
 - Questioned sample – Obtain all loose or transferred paint materials. Sources can include tools, floors, walls, and glass transfers on vehicles or individuals. The following collection techniques should be utilized.
 - Stereoscopic examination and particle picking – Search for paint chips on clothing near compressed fabric (impact areas). Often the paint evidence from hit and run cases will be melted or smeared onto fabric. Smeared transfers should be collected with the underlying layers or object. It is preferable to recover intact paint chips. Use forceps to recover visible chips.
 - Scraping – This is a good recovery technique for paint evidence that was not collected using a stereoscope and forceps. Place a clean piece of butcher paper under the article to be scraped. Using a dull bladed knife, wall scraper, or similar tool, scrape the surface of the item of evidence (clothing) onto the paper. Transfer scraping into a petri dish and examine using a stereoscope.
 - Tape lifts – Every effort should be made to collect the paint evidence

manually before the application of tape lifts. The adhesive could cause contamination of the paint. After the above-mentioned techniques have been utilized, the analyst may choose to collect any remaining trace evidence utilizing tape lifts. These tape lifts may then be examined using a stereomicroscope.

- Known sample
 - The analyst should obtain samples from areas as close as possible to the damaged area (outside the actual transfer area). The possibility of two-way transfers (between two coated surfaces) should be considered. Samples of both surfaces should be collected. Collect samples by prying/scraping particles from the surface into a paper bindle using a scalpel or razor blade.
- Analytical Techniques
 - Prior to the instrumental analysis of a paint sample, the analyst must determine if the unknown paint particles are consistent with automotive paints. The following observations may be helpful in determining what type of paint you have.
 - Automotive Paints
 - High gloss
 - Hard but not brittle in texture
 - Metallic flake in base (color) coat
 - Multiple layer coating systems
 - Body fillers and spot putties
 - Architectural Paints
 - Latex Finishes
 - Often medium to low gloss.
 - Rougher surfaces, even in gloss finishes
 - Less homogeneity in the pigment distribution
 - Flexible texture which tears with tensile force
 - Multiple layers are usually not coating systems
 - Oil Based Finishes
 - Often hard thin single layered fragments.
 - Often high gloss with rougher surfaces.
 - The most conclusive statement regarding the relationship between questioned and known samples is that of a fracture match. Examine questioned and known chips for a possible physical match before any other tests are performed.
- Microscopy
 - Examine the size, color, gloss, texture, number of layers, and layer sequence of the questioned and known paint samples under a stereoscope. Identify the layers by viewing the sample edges with an optical microscope at magnifications between 5x and 100x. Top illumination without mounting will reveal the layers. To identify layers and perform instrumental analysis, the sample must be prepared as follows:
 - Paint Sample Preparation
 1. Work with a new scalpel blade

2. Place chips on a microscope slide, or hold chips in place using a post-it note
3. Sample each layer using repetitive very thin peels working your way through each layer. Start with the topcoat facing down, peel from the primer side.
4. Make thin peels by holding chip down with forceps and use a scalpel blade to provide a shaving action toward the tweezers.
5. Use middle, ring, and little fingers to rest on the counter top providing steady support for the scalpel. Guide the blade with the index finger and thumb.
6. Always take thin peels to avoid pressure on the chip's layer interfaces.
7. For small chips that do not permit placement of forceps on top of the chip, use a blocking technique with dull forceps.
8. On extremely difficult samples, try a cross section peel.
9. Save any peels generated for other examinations microchemical, IR, etc.
10. Resulting chip should have a stair step shape. This chip can be utilized for SEM analysis of the individual layers.

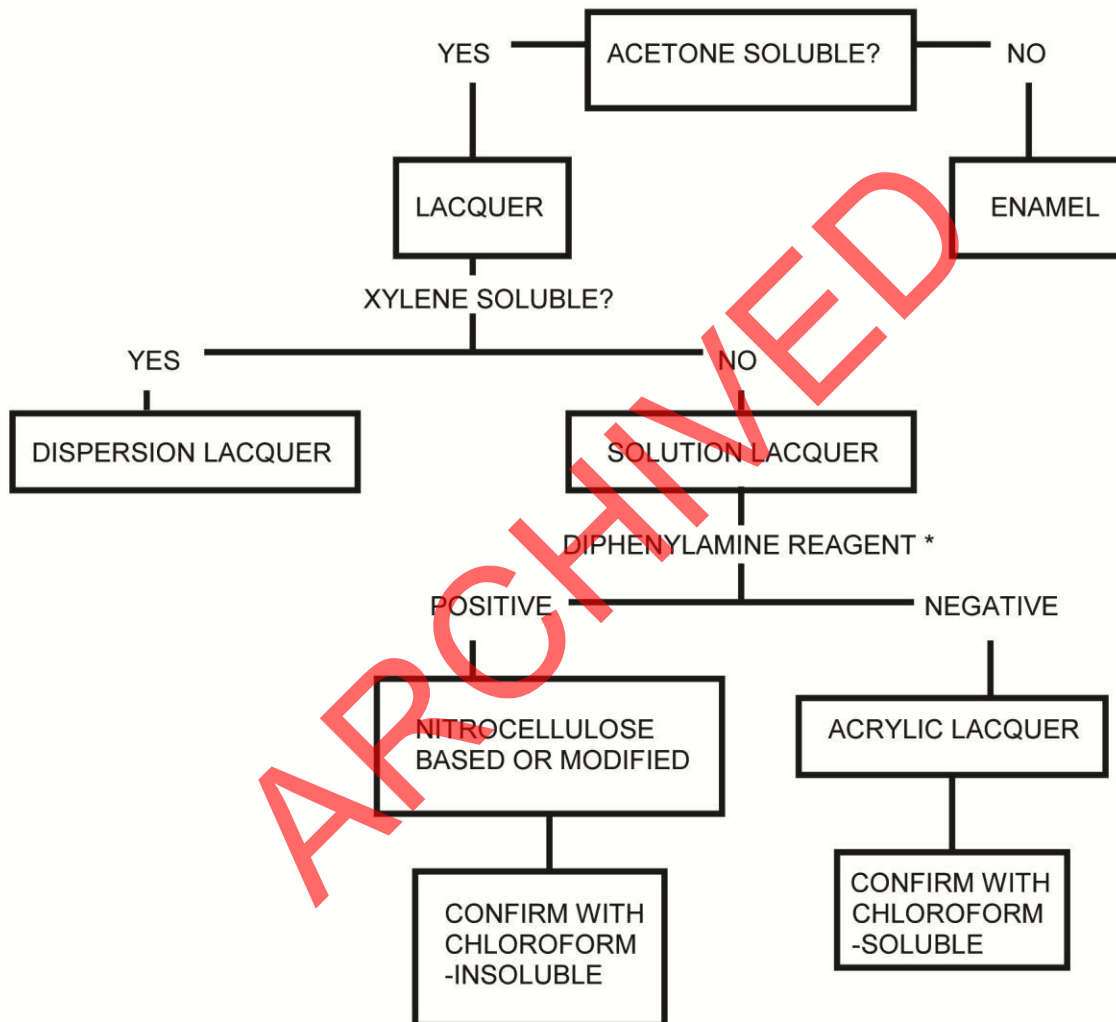


- Use a microscope to perform side by side comparisons of known and questioned sample layers. Both samples need to be positioned side by side in the same field. This is necessary due to illumination difficulties encountered with a comparison scope.
- Use polarized light microscope to characterize the inorganic pigments based on their morphology and optical properties. The analysis can examine thin peels, pyrolysis residues, sublimation condensates (see D. Crown's book) and dispersed particles. Tease the paint with a tungsten needle and observe the pigment granules. The size, shape, transparency, homogeneity, pleochroism, and relative indices can be observed with plane polarized light. Isotropy, anisotropy, retardation, birefringence, extinction, sign of elongation, and extinction colors can be views with crossed polars (see

McCrone's Particle Atlas).

- Chemical/Solubility Tests
 - If sufficient paint is available, destructive chemical testing may be performed. Automotive paint chips (individual layers) can be easily classified as lacquers or enamels. Lacquers can be differentiated into dispersion or solution lacquers. Thornton's solubility scheme provided below may assist in determination and classification of original versus refinish automotive paints.

Basic Microchemical Scheme for Automotive Paints



*Note: To perform DPA microchemical test, take a particle of paint, place on a microscope slide, and add one drop of acetone and allow to dry. Paint pigments will tend to remain towards the center of the dried drop, while the binder will have traveled away from the center along with the acetone. Add one drop of the DPA reagent. An immediate blue color indicates the presence of nitrocellulose.

DPA Reagent: Dissolve 0.3 grams of diphenylamine in 20 mls concentrated H_2SO_4 and 10mls of glacial acetic acid.

- FTIR
 - Classification of paint chip binders is achieved using Micro- FTIR. Each layer should be analyzed separately to provide the most information. The proper aperture must be applied to minimize stray light and to provide the greatest sensitivity for analysis. The FTIR microscope can be used in the transmittance mode or by utilizing an ATR objective. For the most accurate results, run multiple analyses on each layer to achieve reproducible spectra. Automotive finish coats can be classified using the “Automotive Paint Binder Infrared Classification Flow Chart” authored by Tillman. Extender and color pigments may contribute to the paint spectrum.
 - *Typically Encountered Coating Binders*
 - Alkyds
 - Polyesters
 - Acrylics
 - Urethanes
 - Epoxies
 - Cellulosics
 - General Approach to Binder Classification By IR
 - Determine if paint chip is lacquer or enamel by microchemical solvent examination.
 - If paint is a lacquer, determine the type by microchemical examination.
 - Acrylic Solution Lacquer
 - acetone soluble
 - xylene insoluble
 - DPA negative
 - Acrylic Dispersion Lacquer
 - acetone soluble
 - xylene soluble
 - DPA negative
 - Nitrocellulose Lacquer
 - acetone soluble
 - xylene soluble
 - DPA positive
 - If paint is an enamel, determine the type by IR.
 - Look for melamine cross-linking (1550 cm^{-1} and 815 cm^{-1}) found in original finishes.
 - Look for urea cross-linking (1650 cm^{-1} , 1540 cm^{-1} , and 770 cm^{-1}). Typically found in primers, not in finish coats.
 - Look for epoxy cross-linking (1510 cm^{-1} and 830 cm^{-1}). Found mostly in primers.
 - Look for urethane modification ($1530 - 1520\text{ cm}^{-1}$ and possible “stair step” on carbonyl 1730 cm^{-1} at 1690 cm^{-1} and 1640 cm^{-1} . “Newer generation” paints have doublet at 1730 cm^{-1} and 1690 cm^{-1} with intense band at 1460 cm^{-1}).

- Look for MAJOR broad band in the 1300 cm^{-1} to the 1000 cm^{-1} region.
 - Acrylic – 1150 cm^{-1} to 1180 cm^{-1}
 - Alkyd – 1260 cm^{-1} to 1280 cm^{-1}
 - Polyester – 1235 cm^{-1} to 1255 cm^{-1}
- Always check peak tables of respective binders to confirm other absorption bands.

(Classification Chart follows)

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PAINT BINDER INFRARED CLASSIFICATION FLOW CHART

1540 cm⁻¹ or 1550 and 815 cm⁻¹

NO

YES

1530-1520 cm⁻¹

1510 cm⁻¹

1540, 1650 and 770 cm⁻¹

UREA

1510 and 930 cm⁻¹

EPOXY

Is 1280-1260 cm⁻¹ dominant if present?

NO

YES

Is 1240-1230 cm⁻¹ dominant, if present?

NO

YES

1250-1235 cm⁻¹ (be careful here!)

NO

YES

1270 cm⁻¹

1120 cm⁻¹

1070 cm⁻¹

also look for

ALKYD-MELAMIN

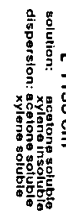
Are 1690, 1450-1470

NO

YES

LIBETHANE

ACRYL



- SEM/EDS
 - The SEM/EDS can be used to analyze the pigment portion of the sample. The surface can be examined by mounting the chip onto a carbon stub. In addition, the SEM can be used to examine surface topography. Comparison of X-ray spectra is generally a non-quantitative method. Multiple areas or large areas will give representative spectra. The chip can be edge mounted in a vertical orientation. As an alternative, the chip can be beveled to reveal the layers. Individual layer analysis is preferred.
 - Commonly Encountered Extender Pigments
 - Silicates

• Quartz	Si
• Diatomaceous earth	Si
• Synthetic Silica	Si
• Kaolin (China Clay)	Al, Si
• Bentonite	Al, Si
• Talc	Mg, Si
• Asbestine	Mg, Si, K,
• Mica (Muscovite)	Al, Si
• Mica (Phlogopite)	K, Mg, Al, Si
• Wollastonite	Ca, Si
• Synthetic Calcium Silicate	Ca, Si
 - Sulfates

• Barytes	Ba, S
• Blanc Fixe	Ba, S
• Gypsum	Ca, S
• Precipitated Calcium Sulfate	Ca, S
• Calcium Sulfate Anhydrite	Ca, S
 - Carbonates

• Precipitated Calcium Carbonate	Ca
• Calcite	Ca
• Limestone	Ca
• Aragonite	Ca
• Dolomite	Ca, Mg
 - Commonly Encountered Coloring Pigments
 - Inorganics

▪ Ferric Oxide	Fe
▪ Lead Oxide	Pb
▪ Yellow Iron Oxide	Fe
▪ Chrome Yellow	Pb, Cr, S
▪ Titanium Dioxide	Ti
▪ Lead Chromate	Pb, Cr
▪ Zinc Iron Ferrite	Zn, Fe, Ti
▪ Molybdate Orange	Mo, Pb, S, Cr
▪ Chromium Oxide	Cr
▪ Zinc Oxide	Zn
 - Organics

▪ Phthalocyanines	Cu and Cl or Br
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- Architectural Paints

- General Considerations

- Architectural paints occur infrequently in forensic casework. These paints are examined using a slightly different analytical scheme. Overall collection and instrumental techniques remain the same. Areas of differing techniques are outlined below.

- Macroscopic Examination

- Latex Finishes

- Often medium to low gloss.
 - Rougher surfaces, even in gloss finishes.
 - Less homogeneity in the pigment distribution.
 - Flexible texture which tears with tensile force.
 - Multiple layers are usually not coating systems.

- Oil Based Finishes

- Often hard thin single layered fragments.
 - Often high gloss with rougher surfaces.

- Microchemical Tests

- Add one drop of chloroform to an unknown paint chip on a microscopic slide. If the paint chip becomes tacky and sticks easily to your forceps that is indicative of a latex paint.

- FTIR

- Typically Encountered Coating Binders

- Latex Systems

- Polyvinyl Acetate-acrylic

- 1735 cm^{-1} , 1370 cm^{-1} , 1240 cm^{-1} , 1175 cm^{-1} , $1135 - 1020\text{ cm}^{-1}$, 945 cm^{-1} , 605 cm^{-1}

- Acrylic

- 1730 cm^{-1} , 1370 cm^{-1} , 1240 cm^{-1} , $1170 - 1150\text{ cm}^{-1}$ (dominant)

- Styrene-Butadiene

- 1450 cm^{-1} , 1495 cm^{-1} , 1600 cm^{-1} , 1730 cm^{-1} , 760 cm^{-1} (very strong), 700 cm^{-1} (very strong)

- Oil-Based Systems

- Alkyd Enamels (ortho)

- 1730 cm^{-1} , 1270 cm^{-1} , 1120 cm^{-1} , 1070 cm^{-1} , 645 cm^{-1} , $710 - 705\text{ cm}^{-1}$

- Alkyd Enamels (iso)

- 1730 cm^{-1} , 1235 cm^{-1} , 1300 cm^{-1} , 730 cm^{-1} (strong), 1605 cm^{-1}

- Urethane Enamels

- 1730 cm^{-1} , 1530 cm^{-1} , and 1240 cm^{-1}

- Nitrocellulose and Acrylic Lacquers

- (see references)

- Acrylic Enamels

- (see references)

SEM/EDS – see list of extender pigments in Automotive section.

CONCLUSIONS

- Identification/Physical Match- The questioned sample was once joined with the known piece of paint
Conditions- The paint from a questioned sample physically matches the standard paint sample.
- Association – The questioned sample and known or standard paint could share a common source
Conditions – all microscopic, microchemical, and instrumental comparisons revealed the questioned sample indistinguishable from the known or standard paint.
 - Note: Consider factors such as sampling, loss of layers, different paint system on different parts of the sample vehicle, and limited samples such as monolayer smears. The analyst can determine the nature of the sample and significance of the association.
- Elimination – The questioned sample and known samples do not share a common source
Conditions – microscopic, microchemical, and instrumental examination reveal dissimilarities in layer structure, layer colors, and/or chemical composition.
- Inconclusive – The questioned and known samples show both similarities and difference such that no clear conclusion can be draw.
Conditions – The analyst is unable to identify a sample or do a meaningful or extensive comparison due to the small sample size, possible environmental decomposition, or other uncontrollable factors.
- Investigative on make and model
 - After examination using microscopic, microchemical, and instrumental analysis, the red debris on the victim's clothing was found to be consistent with red colored, multi-layered automotive paint. This laboratory can provide no possible make/model information at this time.

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2.8 PHYSICAL MATCH ANALYSIS

MATERIALS AND EQUIPMENT

- Camera
- Stereoscope
- Casting media
- Tape, putty, or other adhesive
- SEM/EDS

PROCEDURE

General Guidelines

- Items submitted for physical match analysis include a wide variety of materials. The Supervising Criminalist shall assign a Criminalist to each physical match case based on the type of examination requested and the instrumentation and expertise required to perform the analysis.
- When pieces can be fit together, the Criminalist can say with certainty that the matching pieces were at one time a single unit. No further analysis is required.
- For items in which a physical match is not found, other examinations or comparisons may be performed.
- The questioned items will be examined prior to examination of the known item.

Examination

- Visually examined each questioned item of evidence to determine its class characteristics (e.g., size, color, pattern, dimension, composition, etc.) and compare the questioned items for similarities.
 - Orient the pieces and determined if they have broken/fractured edges that physically fit together.
 - Fabric matching involves examining the general size and shape, weave/knit type, fiber type and twist, colors and patterns, long versus short threads, unusual stretching or contours, stains, damaged area, and stitched edges or selvages.
 - When matching flexible materials (e.g., fabric, tape, and some plastics), care must be taken to account for edge rolling, stretching, and twisting.
 - Matching of rigid materials involves examining the general size and shape, colors and patterns, edges and contours, cracks, breaks, and other damaged areas.
 - If the edges on the pieces physically fit together, observe all orientations of the physical match for specific, individual, characteristics (e.g., scratches, striations, inclusions, and stains, defects, hackle marks, etc.) that traverse the broken, cut, or torn edges.
 - If comparisons at the microscopic level are necessary, a stereomicroscope, comparison microscope, and/or SEM shall be used.
 - Castings of samples may aid in the comparison. Any suitable casting media may be used, such as mikrosil.
 - Photographs shall be taken of all physical matches.
- If sufficient individual characteristics are present, it can be concluded that the items physically match. All reported identifications shall be verified by a second qualified criminalist who will conduct a verification from the original evidence. If the initial authorized Criminalist has reconstructed the material for the fracture match, it may be left assembled for the verifying authorized

Criminalist.

CONCLUSIONS

- Identification – The questioned item was once joined with the known item to form a single item
Conditions – Both class and individual characteristics match and the pieces fit together.
- Inconclusive – The physical match analysis of the items is inconclusive
Conditions – The class characteristics are similar, but there is very limited detail in the break/fracture area.
- Negative – The questioned item was excluded as being at one time joined with the known item.
Conditions – The class characteristics are dissimilar between items
- Association of class characteristics, but no physical match – The questioned item is similar in class characteristics to the known item, but no physical match was found.
Conditions – The class characteristics are similar between items, but the edges of the items do not match or could not be analyzed. The items could go on for further analysis and comparisons.

SAFETY

Broken edges can be sharp. Care shall be exercised during this technical procedure.

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2.9 GSR ANALYSIS

PREPARATION OF GUNSHOT RESIDUE COLLECTION KITS

- Material and Equipment
 - Supplies
 - Specimen holders
 - Aluminum stubs: 1/2" x 1/8" pin
 - Carbon conductive tabs 12 mm diameter
 - Disposable gloves
 - SDPD evidence seals
 - Disposable "Handiwipes" on "Wet Naps"
 - SDPD Gunshot Residue Evidence kit printed envelopes
 - Numbered labels 3M #465 double sided tape
 - Kraft Paper
 - Methanol
 - The carbon conductive tabs are applied to the top of the 1/2" aluminum stubs. The stubs are inserted into the specimen holders. This completes the sample disc vials. The vials are labeled with left and right numbers corresponding to the kit number.

Principle

- The gunshot residue (GSR) collection kits are used to remove potential GSR particles from hands or other surfaces. Adhesive stubs are pressed against the collection surface and then submitted to the Lab for analysis.

Procedure—Gunshot Residue Kit Preparation

- Place the following items into each envelope to complete the kit:
 - One evidence seal
 - One towelette
 - One pair disposable gloves
 - A folded piece of clean kraft paper (measured out 14 3/4")
 - The two sample disc vials with the corresponding 'L' and 'R' numbers on them. (Make sure the numbers on the disc vials matches the number on the GSR kit envelope.)
 - Double check the contents of each envelope then seal with a small piece of transparent tape, and date the envelope.

EXAMINATION OF PREPARED SAMPLE USING THE SCANNING ELECTRON MICROSCOPE/ENERGY DISPERSIVE X-RAY SYSTEM

- Materials and Equipment
 - Supplies
 - Standard Loop Filaments (Tungsten)
 - Equipment
 - JEOL 6360 SEM with OXFORD energy dispersive X-ray detection system and Inca software
- Principle
 - The SEM/EDAX system is used to scan the prepared sample stub for gunshot residue. An automated search uses the feature software program on the INCA X-ray system's computer.
 - The GSR program is used to identify particles of gunshot residue while scanning in a defined sequence over the specimen under test.
 - A backscattered image of the specimen is acquired, with the threshold set so that heavy elements such as lead, antimony and barium are visible. As the SEM scans the specimen it will stop each time a signal is detected above the BSD threshold and then acquire x-ray information from the particle.
 - The INCA data system printer will record the classification, indicating the coordinates for later assessment. After completing its search, a summary is printed which will specify the coordinates of the 3- component and 2- component particles found and the total number of particles found in the pre- programmed classifications.
 - When the stub has been searched the operator can rapidly recall any of the identified particles for a confirmatory assessment of the results. This is achieved by double clicking the cursor on the particles in the classification table.
- Procedures
 - Sample loading and removal; the sample requires no preparation. Samples are examined with a stereomicroscope prior to SEM analysis. If smokeless powder particles are found and confirmed by FTIR analysis, no further examinations are necessary. (See the Trace Evidence Explosives Procedure for FTIR analysis.)

- To load or remove any sample you must open the sample chamber. To do this, the filament must be off and the chamber must be vented. To do so, follow the steps below:
 - Insure that the instrument is "ON". This means that the JEOL 6360 column has power and either the "vent" or "evac" light is illuminated.
 - The JEOL 6360 control program should also be running on the computer. Press the "vent" button on the column or on windows control screen.
 - When the chamber door "cracks" open, the chamber door can be pulled open.
 - Specimen mounts can be inserted in or removed from the holder by gently pushing them in or pulling them out.
 - Before closing the chamber, blow out any loose dust and debris with a spray gun (but be careful not to blow across or on your sample).
 - To close the chamber, push the door closed. Then press the "evac" button (or windows icon).
 - After the chamber door sucks closed, wait until the "evac" light stops flashing.
- Obtaining an image (secondary electron)
 - To obtain an image several conditions must be met.
 - First, the filament must be receiving excitation voltage.
 - Second, the emission current must be balanced with the filament voltage.
 - Third, accelerating potential must be applied.
 - Fourth, the filament must be centered.
 - Fifth, brightness and contrast must be set and sixth the focus must be adjusted.
 - After a filament has been installed and centered, most of the above conditions will already be set and not require additional adjustment. Most of the time, to obtain an image it will only be necessary to power up the filament, check the and adjust the brightness, contrast and focus.
 - Generally the following steps will obtain an image:
 - The HV READY icon must be lit before the filament can be powered up.
 - Once the vacuum is at the appropriate level click on the "HV READY" icon. The icon will change to HT on.
 - Standard settings include a "spot size" setting of "Approximately 50" and "KV" of 20 (in secondary image).

- Obtaining X-ray data
 - Once you have obtained an adequate image, you may acquire Energy Dispersive X-ray (EDX) data. Anytime you command the INCA system to acquire x-ray data it will acquire x-ray data of whatever is being displayed on the entire SEM screen. If you want to limit the area of analysis you must limit what is being scanned (i.e., what is shown on the CRT). You can limit the scanned area one of two ways. You can use a combination of magnification, zoom and stage controls.
 - Once you have an image of the area you want analyzed, you can generate x-ray data by going through the following steps:
 - Before starting, insure that the Oxford detector Dewar has an adequate supply of liquid nitrogen.
 - Be sure that the "KV" setting is at 20.
 - Starting at the ANALYZER screen of the INCA system, select the "acquisition" function.
 - The following hints may help in gathering the best x-ray data:
 - Keep the dead time under 60% (you can do this by controlling the size of the area analyzed or by decreasing the spot size).
 - Use the backscatter image to help sample areas of uniform composition.
 - Choose areas that are not "shadowed" from the detector (left side of screen) by their topography, and reasonable acquisition time, process time of 5, acquisition rate >2000.
- Positioning GSR Samples for auto searching
 - The specimen holder of the SEM is designed to accept up to four samples. The size of the stub used limits the number of samples to two in the specimen holder.
- Running auto search program
 - Once the sample stub(s) have been positioned for auto searching the following settings and the following procedures should be followed in order to auto search:
 - Initial SEM settings
 - OL Aperture - third position, largest (Gives better compromise of count rate and depth of focus).
 - Working Distance - 10 mm
 - Resolution - Spot size as needed

- High Voltage - 20 KV
- BIAS/Emission Current - Approx. 80 Micro amp
(to extend filament life)
- Magnification - 300
- Stage Tilt - 0 degrees
- BSD Setting - See appendix
- Initial INCA feature settings
 - Begin by naming a "Project", saving the project. Then choose a sample name, specify the appropriate Quant Optimization. Next go to Recipe and delete the database, then create a database and save.
 - Use the GSR defaults for Quant setup and Class setup. Use Feature detection to setup contrast and brightness setting for the Backscatter detector. Use Spectrum setup to specify ED analysis: 2 passes, process time of 5, spectrum range of 0-20 Kv and 1000 channels. Detection setup includes field 512 x 384, BSE signal, no gray image processing and no Binary image processing.
 - Set MAGNIFICATION to 300
 - Select Run
- Particle verification
 - To determine the presence of particles unique to gunshot residue, the significant particles detected in the Feature auto search program must be reviewed. The particles are located using the previously described particle review method.
 - Significant unique particles to be reviewed from the classification table are of the following designation:

Pb, Sb and Ba	Pb = Lead
Pb, Sn, Ba and Sb	Ba = Barium Sb = Antimony Sn= Tin
 - Once these particles are relocated; X-ray data is obtained to verify/determine their actual elemental composition. If a single particle is determined to contain Pb, Ba and Sb or Pb, Sn, Ba and Sb, it is considered a particle unique to gunshot residue.
 - The primary elements (Pb, Sb or Ba) can occur at major, minor or trace levels, though for particles involving more than one of

the primary elements at least one of them is typically present at a major level.

- Once a unique GSR particle is found, a photograph and X-ray spectrum is obtained for the analytical notes. In the event many unique GSR particles are found, a representative sample will be selected for photographs and X-ray spectra generation.
- NOTE: In a very few cases suspected particles of gunshot residue may have elemental composition of an unusual nature. A comparison of these particles to the particles present in the barrel of the weapon in question may then be desirable.
- A barrel swab prepared by the FIREARMS UNIT can be examined. The cloth swatch which has been run down the barrel can be sampled using a 1/2" aluminum round stub with a carbon conductive pad. The stub is then coated and examined manually in the SEM as in procedures A-C. The particles from the barrel swab can then be compared to the suspected gunshot residue particles from the case.

REPORTING OF RESULTS

- The results of the gunshot residue examination are given in a report format. The report should contain the general case information such as: victim, suspect, case #, crime, investigator, date of report, etc. This is followed by:
 - Items Examined
 - The specific sample information is given here. This includes the name of the individual tested, the barcode #, the item # (if applicable), and the GSR kit #.
 - Observations/Examination
 - This section includes a description of the analysis conducted and the observed results of that analysis. The analysis can be described with the following statement: "The adhesive lifts from the kit were examined for gunshot residue by scanning electron microscopy with energy dispersive x-ray. The number of particles if any, which are unique to gunshot residue are reported as present on the right and/or left hands. If no particles are detected, this is also stated.
 - Conclusion
 - The conclusions of the report provide information as to how gunshot residue can be deposited:

- Gunshot residue may be deposited on the hands from handling, firing or being in close proximity to a discharged firearm or by contacting a surface contaminated with gunshot residue. The lack of gunshot residue, alone, does not indicate lack of involvement with a firearm. No conclusions can be drawn from negative results.
- The significance of the findings is also given. Depending on the results for the sample, the following statements apply:
- Unique particles: The results from the sample indicate that the subject had exposure to gunshot residue by one of the means described above.
- No particles: No conclusions can be drawn from the results of this sample.

QUALITY ASSURANCE

- Control Testing
 - Gunshot residue collection kits/sample preparation
 - A gunshot residue collection kit will be chosen at random from each set of assembled kits made using the previously described method.
 - This control kit is then analyzed as any other kit by the procedures previously described.
 - No particles consistent or unique to gunshot residue should be present in the sample from the control kit.
- SEM – Instrument Evaluation
 - Backscatter detection setting (sensitivity)
 - The aim of the auto search method, when scanning for potential gunshot residue particles, is to be able to detect and report particles which are 1 micron or less.
 - To accomplish this aim, a known prepared gunshot residue sample (synthetic particle specimen—SPS) is placed in the SEM and auto searched as previously described. This sample contains 1-5 micron and smaller 3- component GSR particles in known locations on the stub. Once the location of the particles is established, the auto search program is setup. This SPS sample is commercially available from Plano GmbH. The finding of 75% or greater of the known number of particles in the selected area is acceptable. If 75% is not reached, adjustments or service to the instrument will have to be done.

- Adjustments can be made to the specimen current and/or backscatter settings to increase the sensitivity.
- The sample can again be auto searched and the above procedure followed.
- Results of the backscatter feature detection settings and results are logged in the appropriate document files.
- Routine calibration of the X-ray system is not necessary. Calibration will be performed, if needed, according to the Oxford procedure.
- The SPS is run with each analysis and the results are included in the case notes.
- Lens Wobble - Aperture Centering Procedure
 - The proper focusing position/aperture alignment for the final lens can be achieved with the following procedure.
 - After obtaining an in-focus image:
 - Place MAGNIFICATION on high. Pick a small particle to monitor in the center of the field.
 - Click on "lens wobble" under tools (Windows "SEM" control screen).
 - Determine if particle moves from side to side or diagonally.
 - If particle moves as in #3, adjust with OL aperture centering knobs located on the end and on the side of the aperture changer* until the particle appears to pulse in a stationary position.
- SEM - Instrument Maintenance
 - Filament exchange
 - The filament life expectancy in the range of 50 - 100+ hours depending on the operating conditions in the microscope. If no beam current is indicated during normal operation and no beam profile imaging is observed with normal settings of the controls the filament has failed and requires replacement.
 - filament replacement procedure is as follows:
 - Press "VENT" on electron beam table front to vent system.
 - Hinge electron gun open after releasing clasp.
 - CAUTION! All gun parts will be extremely hot if microscope has been in use.
 - Use jig/tool to carefully remove Wehnelt or grid cap.
 - Replace filament and check that slot - clamp filament using retaining screws.

- Clean tungsten deposits from Wehnelt aperture carefully – both inner and outer surfaces and bore. A wooden cocktail stick is useful for this – the complete unit should be carefully cleaned and polished with a fine metal polish, e.g. pol metal polish or diamond paste. Wash with organic solvent such as isopropyl.
 - Dry with inert gas duster and with gloved hands or clean tissue replace Wehnelt on gun assembly.
 - Check anode for cleanliness – clean and polish as for Wehnelt if necessary.
 - Replace anode, check O-ring for damage, dirt particles, etc. and clamp gun into position.
 - Pump down as for specimen change procedure.
- Manufacturer's Service/Periodic Maintenance
 - A service contract is maintained for both the scanning electron microscope itself and for the X-ray detector system.
 - The information is reflected in the instrument logbook, which reflects maintenance and usage.
- Proficiency Testing
 - In general, periodic proficiency testing of the SEM/EDAX procedures will be conducted.
 - Suspected gunshot residue samples previously prepared and coated will be auto searched and the results compared to the expected results.
 - These samples can originate from an independent testing firm (e.g. CTS) or from another laboratory which performs this type of analysis.
- Training on Use of GSR Kits
 - Use of gunshot residue collection Kits
 - individuals collecting gunshot residue can be trained as to the proper procedure to be followed by:
 - classroom training conducted by qualified laboratory personnel.
 - viewing of training videotape called "GUNSHOT RESIDUE".
 - In general, the training instructs one how to follow the basic steps which are outlined on the back of the GUNSHOT RESIDUE KITS:
 - Instructions
 - Immediately upon opening the envelope, wipe your hands with the enclosed towelette and put on the plastic gloves.

- Remove the enclosed folded paper and lay it out to provide a clean working surface for taking the samples.
- Remove the disc marked "R" from the clear plastic cylinder. DO NOT separate the disc from the plastic cap.
- Repeatedly, press the adhesive side of the disc onto the back of the subject's RIGHT HAND SLOWLY, concentrating on the thumb, index finger and the web area in between, extending to the tip of the finger. It is critical that this step be done slowly. If the disc is not pressed slowly against the surface no particles will be recovered. Using the same disc, continue sampling the palm side of the thumb, index finger and web area.
- Carefully return the disc to the plastic cylinder.
- Repeat steps 2, 3 and 4 using the "L" disc on the subject's LEFT HAND.
- Place both vials containing used discs into the envelope and seal with the evidence seal. Place initials, date and time on the seal.
- To aid in identification, obtain a finger impression from the subject in the space provided. (optional)
- For sampling of surfaces of inanimate objects (e.g. clothing, caps), see APPENDIX.

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GUNSHOT RESIDUE COLLECTION FROM INANIMATE OBJECTS

- Surfaces of inanimate objects (e.g. cars, clothing) can be sampled for the presence of gunshot residue. However, no time period can be assigned regarding when the deposits were made (i.e. recently or not). If the recent history of an object cannot be firmly established, sampling it would not provide meaningful information.
- Clothing
 - A laboratory sampling of a piece of clothing should take place in a separate room from the TRACE EVIDENCE room. A GUNSHOT RESIDUE KIT is used for sampling. The adhesive disks are pressed against the surfaces of interest (e.g. the sleeve cuffs of a shirt). Note or diagram should reflect the areas sampled. The disks can then be analyzed or any other kit.
- Vehicles
 - Sampling of the interior surfaces of a vehicle can be conducted using a gunshot residue kit. The adhesive disks are pressed against the surfaces of interest, e.g. door window frames, headliners, and seats. Appropriate documentation of these areas should be made. The discs are then analyzed as per usual.

GUNSHOT RESIDUE INFORMATION SHEET FOR INVESTIGATORS AND ATTORNEYS

- Please be aware of the following information regarding gunshot residue whenever the investigation or prosecution of a case involves this type of evidence:
 - Lead, barium and antimony are found in most primer mixtures and are commonly found in gunshot primer residue:
 - Any or all of these elements can be deposited on the hands of an individual who has fired a gun, handled a gun and/or its ammunition or been in close proximity to a discharging gun:
 - The test for GSR CANNOT tell which of the above methods caused the deposit of primer residue, therefore it will provide no meaningful information in cases where a subject is found to be in possession of a firearm; and
 - The lack of GSR on a subject's hands does NOT mean that this subject did not fire or handle a gun.
 - In many cases, the results will positively identify gunshot residue – not just chemical levels consistent with it.
 - Primer mixtures
 - .22 caliber rimfire from Federal and centerfire ammunition can produce unique gunshot residue particles.
 - Surfaces other than hands
 - Surfaces other than hands CAN be identified as possessing

GSR. In the case of inanimate objects, however, no time period can be assigned regarding when the deposits were made (i.e. recently or not). If the recent history of an object cannot be firmly established, sampling it would not provide meaningful information.

- Activity after shooting
 - In most cases, six or more hours of activity by a LIVING person will result in the loss of most or all of the GSR from the person's hands (even without intentional washing). In most cases, samples taken after this time would not be meaningful. Because we will be using a particle method for analysis, there is some flexibility with these time limits. Cases exceeding these limits will be considered on an individual basis.
 - If a subject has washed his hands, no meaningful information can be provided.
- Shooting victims and reconstruction of events
 - If a person has been shot at close range it is quite possible for him to get GSR on his hands (and body). Similarly, two individuals struggling with a gun (even if it does not discharge) may get GSR on their hands. GSR testing in such situations will rarely be able to sort out a sequence of events. The mere contact with a gun or another surface contaminated with GSR can transfer GSR onto a person's hands. Such cases will be evaluated on an individual basis to determine if testing will provide useful information.
 - If there is doubt about the usefulness of a sample in any case, it is always better to take the sample and decide later regarding its analysis. Further information on general policy or individual cases can be obtained by contracting the Trace Evidence Section of the Laboratory. Please notify the Laboratory thirty (30) days before a court date to obtain results.
- Old Style GSR Kits will no longer be analyzed by this laboratory.

3.1 REPORTING

Refer to the laboratory general policy for the standardized laboratory report format, case notes, technical review, and administrative review requirements. Trace Evidence Unit reporting policies are included in at the end of each method policy document.

CASE NOTES

Case note requirements follow the requirements stated in the laboratory quality assurance manual for technical records.

ABBREVIATIONS

- SEM/EDS or SEM/EDX: Scanning Electron Microscope with Energy Dispersive X-ray Spectroscopy
- GC/MS: Gas Chromatography with Mass Spectrometry
- GC-FID: Gas Chromatography with Flame Ionization Detector
- FT-IR: Fourier Transform Infrared Spectroscopy
- IR: Infrared
- PLM: Polarized Light Microscope
- Stereo: Stereoscope
- GSR: Gunshot Residue
- C-strip: Activated charcoal strip
- ACS: Activated charcoal strip
- n: Refractive index
- $n_{||}$: Parallel refractive index
- n_{\perp} : Perpendicular refractive index
- Birefrig or B: Birefringence
- Sign: Sign of elongation
- Delust: Delusterants
- BC: barcode
- X-section or XS: Cross-section
- LPD: light petroleum distillate
- MPD: medium petroleum distillate
- HPD: high petroleum distillate
- Poly: polyester
- Std: Standard
- TLC: Thin Layer Chromatography
- UV: Ultra violet
- R_f : Retention Factor
- Trans: Transmitted light
- Ref: Reflected light
- Mod: moderate
- Sl: slightly
- Dk: dark
- Lt: Light
- Inc: Incident
- PT: Property Tag
- Macro: macroscopic

- Micro: microscopic
- MP: melting point
- BP: boiling point
- Nyl: nylon
- Neg or -: negative
- Pos or +: Positive
- App: Apparent
- Recv'd: Received
- Cont'd: Continued
- Pass.: Passenger
- Diam or dia: Diameter
- Chem'l: chemical
- Brn: brown
- w/: with
- blk: black
- approx.: approximate
- app: apparent

STATISTICS

- Each analyst will generate a statistical report for each case worked. This includes proficiency tests. The statistical report will contain the following information:
 - Criminalist :
 - Unit : Trace Evidence/Case Type
 - Case (Key Case Number):
 - Number of Cases Involved:
 - Number of Items Examined:
 - Number of Reports:
 - Comments:
- Each case will count as one case. Each item examined will count as one item. Each report generate will count as one report. The statistical report will be included with the report for the supervisor to review. The Supervisor will keep the statistical report and use them for monthly unit statistical reports.

TECHNICAL REVIEW AND ADMINISTRATIVE REVIEW REQUIREMENTS

Technical review and Administrative review requirements follow the requirements stated in the laboratory quality assurance manual for technical records.

4.1 EQUIPMENT INFORMATION

The following equipment is specific for Trace Evidence:

- Scanning Electron Microscope-SEM with Energy dispersive X-ray analysis system
- Fourier-Transform infrared Microscope- FTIR microscope with data system
- Polarizing microscopes
- Stereomicroscopes with illuminators
- Transmitted Light Comparison Microscopes
- FID Gas Chromatograph with data system
- Gas Chromatograph/Mass Spectrometer
- Reference collection of hairs, fibers, accelerants, glass, etc.
- Open pan balance

EQUIPMENT GENERAL USE

The following equipment has the following general uses:

- Scanning Electron Microscope-SEM with Energy dispersive X- ray analysis system
 - gunshot residue and inorganic identification of particles and residues
- Fourier-Transform Infrared Microscope with data system
 - fiber analysis, paint analysis, explosives, tapes, polymers, unknowns, and identification
- Polarizing Microscopes
 - hair and fiber analysis, paint analysis, explosives, general trace evidence unknowns
- Stereomicroscopes with Illuminators
 - preliminary evidence evaluation
- Transmitted Light Comparison Microscopes
 - Side-by-side, microscopic comparison of trace evidence materials
- FID Gas Chromatograph with data system
 - ignitable liquid identification
- Gas Chromatograph/Mass Spectrometer
 - ignitable liquid identification and general organic analysis
- Open pan balance
 - weight of reagents and samples

5.1 QUALITY ASSURANCE MEASURES

GENERAL

- Any errors or issues will be reported to the Technical Lead. The Technical Lead will track and maintain records of all issues and errors within the Trace Evidence Unit.

PROFICIENCY TESTING

- Proficiency testing is completed on a cycle according to the lab Quality Manual. Proficiency Tests are completed as independent casework, following unit policies and procedures. Test results will be evaluated against published results, with consideration to unit policies and procedures, by the Latent Print Supervisor, or OCA, using the Proficiency Test Record form. The Technical Lead will assist to evaluate the results if there is a difference between the published results and individual examiner's result. If an examiner is unable to complete the test due to poor quality of the issued test samples, the examiner will confer with the unit supervisor to determine the course of action.

SECONDARY CHEMICAL CONTAINERS

- Secondary containers hold chemicals that have been transferred from a primary container. Secondary containers must be labeled according to the laboratory safety requirements to contain: chemical name, signal word, and pictograms. These labeling requirements are exempt if the material is used within the work shift of the employee who makes the transfer.

REAGENT TESTING

- A reagent is a chemical substance added to a solution of another substance to produce a chemical reaction, so as to detect, measure, or produce other substances. Reagents prepared by the Trace Evidence unit will be labeled to contain: reagent name, signal word, pictograms, and preparation date. These labeling requirements are exempt if the reagent is used within the work shift of the employee who prepares the reagent.
- Reagents will be tested upon preparation to verify contents. Appropriate standards and controls will be run to assure proper working condition. The date of preparation and the results of the standards and controls will be recorded in the case notes.

FIRE DEBRIS QA/QC

Materials and Supplies

- Reagent grade or higher (ACS specifications) chemicals will be used in all tests.
- All materials used must be checked for possible contamination or interferences on a regular basis. These materials include:
 - Evidence containers used for analysis of fire debris evidence in the Laboratory by scene investigators (metal paint cans and Kapak bags).
 - Materials used in the laboratory for the actual extraction of accelerants, e.g. charcoal strips, solvents, syringes, and sample vials.
- These materials will be tested for the presence of hydrocarbons or other compounds that may interfere with the detection of volatiles.
 - Solvents will be analyzed frequently by direct injection onto the GC column being used.
 - Evidence storage containers will be checked on a batch basis by using passive-

adsorption/elution with charcoal strips. Metal paint cans are purchased by the investigators and are submitted to the laboratory when new lots are received. A report of pass/fail is provided to the investigators as to whether or not the new cans are suitable for evidence collection.

- In the event that hydrocarbons or other interfering substances are detected the contaminated item(s) will be identified and removed from use in analysis and replaced with non-contaminated material. In the event that the metal evidence cans contain interfering substances, the investigators will be notified to purchase a new lot of cans.

Accelerant Reference Library

- A library of reference samples and chromatograms is maintained in the unit. Each reference is uniquely identified, and include many different commercial products. The samples can be analyzed as neat liquids, liquids diluted in an appropriate solvent, and/or adsorption/elution depending on the analytical conditions expected to be used for the analysis of actual case samples. Obtain reference chromatograms under standard GC conditions and display at the lowest attenuation so that all peaks remain on scale. The library should include the following:
 - Class 1 - Neat samples of products found in this classification (lighter fuels, rubber cement solvents, petroleum ether) including various brands or sources.
 - Class 2 - Gasolines (automotive) of several brands and grades. Run neat, 50 - 70% evaporated, and 95 - 98% evaporated.
 - Class 3 - Neat samples of the products typical of this class (paint thinners, charcoal starters, torch fuels, mineral spirits), including various brands and sources.
 - Class 4 - Any brand or grade of kerosene, run neat, Jet A (Aviation, JP-5), insect sprays.
 - Class 5 - Fuel oil No.2 (diesel fuel).
 - Class 6 - Neat samples of alcohols, ketones, aromatics, isoparaffinic solvents, camping fuels, lacquer thinners, carburetor cleaners, duplicating fluids, gum turpentine, etc
- The accelerant references are stored in a freezer. The temperature of storage of the accelerants is non-critical, and only done to help with evaporation; therefore, the temperature of the freezer is not recorded.

EXPLOSIVES

- Spot tests reagents for explosives will be prepared as needed and the results of controls recorded in the note pages for the analysis. The reagents will be discarded after use. Small micro-amounts of reagents can be prepared on the slide so that the spot tests become microchemical instead of bulk spot tests.

INSTRUMENTATION

General

- All data from the instruments is stored on the individual instruments under the

case or incident number. The data is also printed out and saved as a hard copy in each case file.

Equipment Performance Evaluation

- A log book will be maintained for each instrument in which maintenance is performed. All instruments will complete the appropriate performance check before returning to service. For the appropriate performance check refer the instrument sections below.

GC/MS and GC-FID

- Routine maintenance of the analytical equipment must be performed in accordance with the manufacturer's recommendations. For GC/MS and GC-FID this will include properly servicing the columns, gases, detectors, and injectors.
- The proper settings needed will depend on the specific equipment and column in use. However, the performance of the column will be checked against a reference chromatogram of resolution test mixture. The column and program together should allow baseline resolution of all components of such a mixture. If all components cannot be resolved, adjustments to the chromatography run parameters needs to be made to achieve proper resolution. A printout copy of the reference standard run will be filed in the GC-FID and GC/MS log book.
- A commercially available and traceable resolution test mixture that includes n-alkanes will be run every time there is significant maintenance on the instrument that could alter our retention times and after the yearly preventative maintenance service.
- Gas Chromatograph/Mass Spectrometer performance is checked by manufacturer established autotune methods.

Stereomicroscopes and microscopes

- Microscopes and stereomicroscopes are covered by the equipment maintenance policy in the laboratory quality assurance manual.

Balances

- Balances are checked quarterly for performance. They are also serviced annually.

FTIR

- To evaluate the performance of the bench, Nicolet has created a program within the Omnic software called ValQ. This program performs the following checks of the bench:
 - Background
 - 100% line
 - Signal to noise measurement
 - Two internal polystyrene standards to compare specific peak locations over time. These peak locations are only useful for the search function when searching commercially available libraries and have nothing to do with actual peak locations of chemical bonds.
 - A report is generated each time the ValQ is performed and a pass/fail score is given to the instrument in each of these areas. If a "fail" score is given then

something is wrong with the bench and more diagnostics need to be performed or a service technician will have to evaluate the instrument.

FTIR Bench

- Run the ValQ whenever the bench is used or annually, whichever is more frequent. Place a copy of the ValQ report in the FTIR notebook with the analyst's initials and date.

Continuum FTIR Microscope Alignment

- To evaluate the performance of the microscope, the microscope must be aligned properly prior to each use. When aligned properly the signal throughput for the microscope in the %T mode should be between 10 and 15 at a gain of 1 and in %R mode the signal should be between 8 and 10 at a gain of 1. The signal, along with the shapes and locations of the interferogram and the single beam spectra determine if the instrument is aligned and operating properly.
 1. Be sure the microscope is turned on and the detector dewar has been filled with liquid nitrogen.
 2. Select reflection viewing mode by pressing the mode selection switch on the front panel.
 3. Adjust the reflection illumination to medium intensity. Adjust the transmission illumination and Reflex aperture illumination to their lowest intensities. The field irises for transmission and reflection should be fully open and the aperture irises for transmission and reflection should be set for low contrast.
 4. Set the objective and condenser compensation rings to zero.
 5. Place the slide with the pinhole onto the stage and position the pinhole directly under the objective.
 6. Use the X and Y position knobs to center the pinhole on the reticle cross hairs and then refocus to a sharp image.
 7. Adjust the Reflex aperture illumination so that the apertured area is brighter than the rest of the field of view.
 8. Adjust the size, shape, and orientation of the aperture to match the reticle reference square. If you cannot match the reference square the viewer is out of alignment. A service call should be made to correct this situation.
 9. Position the large open hold of the slide directly under the objective.
 10. Select transmission viewing mode by pressing the mode selection switch on the front panel.
 11. Adjust the transmission illumination to medium intensity.
 12. Adjust the condenser focus knob and the condenser centering knobs so that the post-sample aperture image is sharply focused and matches the reticle reference square.
 13. Check the signal throughput using the Omnic software. Chose Experiment Setup from the Collect menu. Click on the Bench Tab. Check the following settings:
 - a. Gain =1

- b. Sample Compartment = Right μ Scope. %T
 - c. Velocity = 1.8988
 - d. Detector = MCTA
 - e. Beam Splitter = Kbr
 - f. Source = IR
 - g. Accessory = None
 - h. Window Material = none
14. Check the radio button to read Peak to Peak. The interferogram present in the window should look normal and centered in the window. If the microscope is aligned properly the peak-to-peak value should be between 10 and 15. If the signal is lower than 10, the microscope is not properly aligned. Repeat the alignment procedure. If the signal remains below 10 then you should call for technical support.
 15. Check the box marked Single Beam. The interferogram changes to a normal background infrared spectra. This should look normal with peaks for CO₂ and water and should be centered in the window.
 16. If an experiment will be done in the reflectance mode then check the signal throughput with the microscope in that mode using the following procedure:
 - a. Select reflection viewing mode by pressing the mode selection switch on the front panel.
 - b. Position the gold mirror of the sample side directly under the infrared objective.
 - c. Use the focus knob to obtain a sharp image of the mirror surface.
 - d. Adjust the size, shape, and orientation of the aperture image so that it matches the reticle reference square.
 - e. Chose Experiment Setup from the Collect menu.
 - f. Set the sample compartment on Right μ Scope. %R
 - g. Be sure the gain and velocity are set at 1 and 1.8988 respectively.
 - h. The peak-to-peak signal value should be between 8 and 10. If the signal is less than 8 you may not get a good reflectance spectra.
 - i. Check the interferogram and single beam spectra to make sure they are normal and centered.

SEM/EDS

Backscatter detection setting (sensitivity)

- The aim of the auto search method, when scanning for potential gunshot residue particles, is to be able to detect and report particles which are 1 micron or less.
- To accomplish this aim, a known prepared gunshot residue sample (synthetic particle specimen—SPS) is placed in the SEM and auto searched as previously described. This sample contains 0.5 – 2 micron and smaller 3- component GSR particles in known locations on the stub. Once the location of the particles is established, the auto search program is setup. This SPS sample is commercially available from Plano GmbH. The finding of 75% or greater of the known number of particles in the selected area is acceptable. If 75% is not reached, adjustments or service to the instrument will have to be done.

- When analyzing samples for elemental ID (non-GSR) a Quant-optimization is performed using the copper foil on the SPS stub. The Xray detector must identify the 3 copper peaks. If the peaks are not correctly identified, adjustments to the instrument or service will have to be done. This information will be documented in the note packet for each case

ARCHIVED

6.1 TRAINING

The unit supervisor coordinates the administration of the training program. The Technical Lead or other casework authorized individuals act as trainers. The trainer is responsible for the completion of the training, including lectures, practicals, and written and competency tests. Training outlines document the training process. Reading lists may be amended with more current references.

A formerly trained or experienced trainee may complete training in a more abbreviated form than what appears on the training outline, but will complete a competency test prior to performing independent casework in a scope of competency.

New trainees, who have not performed casework at the San Diego Police Department Crime Laboratory, will complete the following:

1. General knowledge introduction of forensic science
 - a. Powerpoint of forensic science services offered at the SDPD Crime Lab.
Tour of the SDPD Crime Laboratory
2. Application of ethical practices in forensic science
 - a. Review and acknowledgement of City of San Diego employee code of conduct.

6.2 TRAINING: EVIDENCE SCREENING TRAINING

INTRODUCTION

- This training will cover the basic concepts of screening evidence for the presence of trace evidence.

OBJECTIVES

The trainee should develop knowledge and skills in the following areas:

- Types of Evidence examined
- Methods of Collection and Screening
- Examination and characteristics of materials
 - Hairs
 - Fibers
 - Wood
 - Glass
 - Building materials
 - Paint
 - Natural material
 - Debris
 - Soil
 - Misc. items
- Identification of glass
 - Polarized light microscope
 - Edges
 - Fractures

PRACTICALS

RECOMMENDED CLASS

- Polarized light microscopy or trace evidence course
 - If not available the training will occur in house

SUGGESTED READING

- Saferstein, R. 1988. Forensic Science Handbook Volume II, Chapter 4, Englewood Cliffs, NJ: Prentice Hall
- Saferstein, R. 2010. Forensic Science Handbook Volume III, Chapter 1, Englewood Cliffs, NJ: Prentice Hall

FINAL WRITTEN EXAM

- Trainee must receive 80% or better with evaluation

COMPETENCY

- Trainee must receive 100% with evaluation

6.3 TRAINING: FIRE DEBRIS TRAINING

INTRODUCTION

- This training will cover the basic concepts of fire chemistry and ignitable liquid detection, and collection.

OBJECTIVES

The trainee should develop knowledge and skills in the following areas:

- Parameters required to start and maintain combustion
- Recognition and categorization of different types of ignitable liquids used to accelerate a fire
- Utilization of different methods for isolating ignitable liquids using laboratory methods including understanding the capabilities and limitations of each method
- Identification and/or comparison of ignitable liquids with a thorough understanding of the instrumental methods used in analysis.
- Flammability testing
- Examination and analysis of case materials with consideration of other types of evidence that may be associated with the samples
- Interpretation of the forensic significance of the analytical results obtained.

TRAINING

The following topics will be covered in fire debris training:

- Fire chemistry
- Fire investigation
- Evidence recognition, collection, packaging, and field investigation
- Petroleum products
- Naturally occurring products
- Ignitable liquid characterization
- Safety considerations
- Ignitable liquid recovery and isolation techniques
- Incendiary devices
- Chromatography
 - GC-FID
 - GC/MS
- Microbial degradation of petroleum products
- Ignition and flammability testing

PRACTICAL EXERCISES

SUGGESTED READING

- CCI Arson Accelerant Detection notebook
- DeHaan, John, 1991. Kirk's Fire Investigation, 3rd Ed., Prentice- Hall, Inc.
- Applicable ASTM methods
- CCI Fire and Explosive Investigation notebook
- Icove, D., and DeHaan, J., 2004. Forensic Fire Scene Reconstruction, Upper Saddle River, NJ: Pearson Prentice Hall.

- O'Connor, J., 1987. Practical Fire and Arson Investigation, New York, Elsevier Science.

FINAL WRITTEN EXAM

- Trainee must receive 80% or better with evaluation

COMPETENCY

- Trainee must receive 100% with evaluation

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6.4 TRAINING: DETECTION AND EXAMINATION OF IMPRESSION EVIDENCE

INTRODUCTION

- This training will cover the basic concepts of footwear and tire manufacturing, impression collection techniques, enhancement of impressions techniques, and methods for comparison of impressions.

OBJECTIVES

The trainee should develop knowledge and skills in the following areas:

- Basic concepts of forensic footwear and tire impression evidence
- Basic knowledge of tire and footwear manufacturing
- Impression characteristics and comparable features
- Impression collection techniques
- Impression enhancement techniques
- Comparison and examination procedures

TRAINING

The following topics will be covered in impression evidence training:

- Basic impression terminology
- Different types of impressions
- Value of impression evidence
- Footwear and Tire manufacturing methods
- Collection techniques for impression evidence
- Enhancement techniques for impression evidence
- Class characteristics in footwear and tires
- Wear characteristics in footwear and tires
- Identification characteristics in footwear and tires
- Examination procedures for impression evidence
- Collection of exemplars from known footwear and tires

PRACTICAL EXERCISES

SUGGESTED READING

- Bodziak, William, 2000. Footwear Impression Evidence, 2nd Ed., CRC Press.
- McDonald, Peter, 1993. Tire Imprint Evidence, CRC Press.

FINAL WRITTEN EXAM

- Trainee must receive 80% or better with evaluation

COMPETENCY

- Trainee must receive 100% with evaluation

6.5 TRAINING: HAIR EXAMINATION

INTRODUCTION

- This training will cover the basic concepts of hair characteristics, microscopy, recovery of evidence, and hair growth.

OBJECTIVES

The trainee will gain knowledge and skills in the following areas:

- Microscopy
- Hair morphology
- Human hair characteristics
- Animal hair characteristics
- Evaluation of hair standards
- Recovery of hair evidence
- Methods for comparison
- Methods for documentation
- Evaluation of the hair root for nuclear DNA analysis
- Evaluation of damage, color treatment, and other conditions to hair
- Laboratory report writing in this area

TRAINING

The following topics will be covered:

- Lectures on hair growth and structures, hair structure, hair comparisons, special topics in comparisons, DNA techniques of hair analysis
- Macroscopic and microscopic characteristics of hairs
- Scale casting
- Somatic origin
- Human vs. animal hair characteristics
- Examination of hair standards
- Comparison of question hair to hair standards
- Hair root documentation and nuclear DNA suitability

PRACTICAL EXERCISES

SUGGESTED READING

- Saferstein, R. 1982. Forensic Science Handbook Volume I, Chapter 5, Englewood Cliffs, NJ: Prentice Hall
- Guadette, B.D. and Keeping, E.S. "An attempt at determining probabilities in Human Scalp hair comparison". Journal of Forensic Science, 1974, Jul; 19(3): 599-606.

FINAL WRITTEN EXAM

- Trainee must receive 80% or better with evaluation

COMPETENCY

- Trainee must receive 100% with evaluation

6.6 TRAINING: FIBER

INTRODUCTION

- This training will cover the basic concepts of fiber manufacturing, fiber characteristics, microscopy, recovery of evidence, Fourier Transform Infrared Spectroscopy (FT-IR), and thin layer chromatography (TLC).

OBJECTIVES

The trainee will gain knowledge and skills in following areas:

- Fiber and textile manufacturing processes
- Manmade fibers v natural fibers
- Microscopy
 - Stereomicroscope
 - Transmitted light microscopy
 - Polarized light microscopy
- Cross sections
- Solubility
- FT-IR
- TLC

TRAINING

The following topics will be covered:

- Textile manufacturing
- Fiber manufacturing
- Fiber retention on samples
- Fiber type commonality
- Natural fibers
- Microscopy
- Polarized light microscopy
- Refractive Indices
- Birefringence and use of Michel-Levy Color Chart
- Cross-sections
 - Microscopic
 - Manual cutting methods
- FT-IR sample prep
- FT-IR sample run
- Solubility
- TLC for dye analysis
- Comparison methods

PRACTICAL EXERCISES

SUGGESTED READING

- Gaudette, B. The forensic aspects of textile fiber examination. In: Forensic Science Handbook (Vol. 2). Ed., R. Saferstein. Prentice-Hall, Englewood Cliffs, New Jersey, 1988.
- Robertson, J. The forensic examination of fibers: Protocols and approaches—An overview. In: Forensic Examination of Fibers. Ed., J. Robertson. Ellis Horwood,

- Chichester, United Kingdom, 1992.
- CCI Fiber Training Manual
- CCI Basic Practical Microscopy Manual
- SWGMAT Fiber training and analysis procedures

FINAL WRITTEN EXAM

- Trainee must receive 80% or better with evaluation

COMPETENCY

- Trainee must receive 100% with evaluation

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6.7 TRAINING: PRIMER GUNSHOT RESIDUE (GSR)

INTRODUCTION

- This training will cover the basic concepts primer gunshot residue and the Scanning Electron Microscope with Energy Dispersive X-ray Spectroscopy (SEM/EDS).

OBJECTIVES

The trainee will gain knowledge and skills in the following areas:

- Collection of primer gunshot residue from hands and clothing
- Limitations of collections and analysis
- Reaction when firearm is discharged
- Ammunition components and variations
- Composition and morphology of primer gunshot residue
- Scanning electron microscopy
- Electron dispersive x-ray spectroscopy

TRAINING

The following topics will be covered:

- History of GSR analysis
- Limitations of GSR analysis
- Ammunition and firearm differences and effect on GSR produced
- Gunpowder characteristics, intact v discharged
- Formation of GSR
- Composition of GSR
- Deposition of GSR
- Environmental and occupational materials similar to GSR
- Collection method of GSR
- Definition of a GSR environment
- Duration of GSR on individual hands, clothing, or objects
- SEM/EDS
 - Theory
 - Basic function/method
 - Specific use for GSR
 - Maintenance and troubleshooting

PRACTICAL EXERCISES

SUGGESTED READING

- JEOL and Oxford User Manuals
- Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F. "Final Report On Particle Analysis For Gunshot Residue Detection". Segundo, CA: Aerospace Corporation, 1977.
- Krishnan, S. S. "Detection of Gunshot Residue: Present Status." Krishnan, S. S. Forensic Science Handbook, Volume 1. Ed. Richard Saferstein. Englewood Cliffs, NJ: Prentice Hall, Inc., 1982. 572-591.

- White, R. S. and Owens, A. D. "Automation of Gunshot Residue Detection and Analysis by Scanning Electron Microscopy Energy Dispersive X-Ray Analysis (SEM/EDX)." *Journal of Forensic Sciences* (1987): 1595-1603.
- Wright, D. M. and Trimpe, M. A. "Summary of the FBI Laboratory's Gunshot Residue Symposium May 31-June 3, 2005." *Forensic Science Communications* (2006).
- Romolo, F. S. and Margot, P. "Identification of Gunshot Residue: A Critical Review." *Forensic Science International* (2001): 195-211.
- Wrobel, H., Millar, J. J., and Kijek, M. "Identification of Ammunition From Gunshot Residues and Other Cartridge Related Materials--A Preliminary Model Using .22 Caliber Rimfire Ammunition." *Journal of Forensic Sciences* (1998): 324-328.
- Wallace, JS. And McQuillan, J. "Discharge Residues from Cartridge-operated industrial Tools." *Journal of the Forensic Science Society* 1984; 24: 504.
- Kilty, J. W. "Activity after Shooting and Its Effect on the Retention of Primer Residue." *Journal of Forensic Sciences* (1975): 219-230.
- Berk, R. E. "Automated SEM/EDS Analysis of Airbag Residue as a Source of Percussion Primer Residue Particles I and Automated SEM/EDS Analysis of Airbag Residue as a Source of Percussion Primer Residue Particles II." *Journal of Forensic Sciences* (2009): 60-68, 69-76.
- Mosher, P. V., McVicar, M. J., Randall, E. D., and Sild, E. H. "Gunshot Residue-Similar Particles Produced by Fireworks." *Canadian Society of Forensic Sciences Journal* (1998): 157-168.
- Torre, C., Mattutino, G., Vasino, V., and Robino, C. "Brake Linings: A Source of Non-GSR Particles Containing Lead, Barium and Antimony." *Journal of Forensic Sciences* (2002): 494-504.

FINAL WRITTEN EXAM

- Trainee must receive 80% or better with evaluation

COMPETENCY

- Trainee must receive 100% with evaluation

6.8 TRAINING: PHYSICAL MATCH

INTRODUCTION

- This training will cover the basic concepts of physical matches with commonly encountered evidence.

OBJECTIVES

The trainee will gain knowledge and skills in the following areas:

- General properties from a variety of separated materials
- Discrimination of physical compositional features of separated materials including relevant manufacturing characteristics
- Influence of distortion and forces that cause distortion on different materials
- Types of separation forces
- Photography

TRAINING

The following topics will be covered:

- Comparing and contrasting visible properties of separated materials
- Manufacturing characteristics of materials commonly observed
- Physical characteristics of materials commonly observed
- Distortion of materials
- Forces that cause distortions
- Effect of the type of force of separation on material and match analysis
- Processing of evidence for physical match, such as undoing a wad of tape
- Photography, alternate light sources, and documentation

PRACTICAL EXERCISES

SUGGESTED READING

- ASTM Standard E2288, 2003, "Standard Guide for Physical Match of Paper Cuts, Tears, and Perforations in Forensic Document Examinations." ASTM International, West Conshohocken, PA, 2003.
- Saferstein, R., ed. *The Forensic Science Handbook, Volume I*, 2nd Edition. New Jersey: Pearson Education, Inc., 2002. Chapter 4: Forensic Glass Comparisons.
- Bradley, M.J., et al. "A Validation Study for Duct Tape End Matches." *Journal of Forensic Sciences* 51.3 (2006): 504-508.
- Dixon, K. "Positive Identification of Torn Burned Matches with Emphasis on Cross Cut and Torn Fiber Comparisons." *American Academy of Questioned Document Forensic Scientists* (August 1982).
- Laux, D. "Identification of a Rope by Means of a Physical Match Between the Cut Ends." *Journal of Forensic Sciences* 29.4 (1984): 1246-1248.
- VonBremen, U.G. and B. Lorne. "Physical Comparison of Plastic Garbage Bags and Sandwich Bags." *Journal of Forensic Sciences* 28.3 (1983).

FINAL WRITTEN EXAM

- Trainee must receive 80% or better with evaluation

COMPETENCY

- Trainee must receive 100% with evaluation

ARCHIVED

6.9 TRAINING: PAINT

INTRODUCTION

- This training will cover the basic concepts of paint manufacturing, paint characteristics and layers, and paint examinations.

OBJECTIVES

The trainee will gain knowledge and skills in the following areas:

- History of paint
- Paint manufacturing processes
- Search and collection techniques
- Microscopical examinations and characterization
- Binder Classification
- Pigment Identification
- Extender Examination
- Additive Examinations
- After-market treatments, weathering, aging, and contaminants
- Significance and interpretation of paint analysis and comparison

TRAINING

The following topics will be covered:

- History of paint
- General and Forensic terminology
- Uses and composition of paint
- Manufacturing processes
- Collection techniques
- Microscopical examination and characterization
- Sample preparation techniques
- Solvent examinations
- Microchemical examinations
- Infrared spectroscopy for binder identification, pigments, and extenders
- SEM/EDS for pigment and extender examination
- Additive types, functions, and methods of analysis
- Classification of paint
- Comparison and discriminations of paint

PRACTICALS

SUGGESTED READING

- Thornton, J.I., "Forensic Paint Examination, Forensic Science Handbook", 1982, pp 529 – 571.
- "Paint Examination and Comparison Reference Notebook", CCI, 1999. "Paint Examination and Comparison Class Workbook", CCI, 1999.
- McCrone, Walter C. and Delly, John G., "The Particle Atlas, vol. 2", Ann Arbor Science Publishers, Ann Arbor, Michigan: 1973.
- Crown, David A., "Forensic Examination of Paints and Pigments", Charles C. Thomas Publishing, Springfield, IL: 1968.
- Tillman, Warren L., "Automotive Paint Systems Identification", Proceedings of the

International Symposium on the Forensic Aspects of Trace Evidence, FBI, June 1991, pp 123-152.

FINAL WRITTEN EXAM

- Trainee must receive 80% or better with evaluation

COMPETENCY

- Trainee must receive 100% with evaluation

ARCHIVED

INTRODUCTION

- This training will cover the basic concepts of legal issues, testimony, court systems, and court presentations. This training module is provided to new Criminalists in the Trace evidence unit. A criminalist who has prior Trace Evidence testimony experience will not be required to complete this training module.

OBJECTIVES

The trainee will gain knowledge and skills in the following areas:

- Understand the Federal Rules of Evidence (FRE)
- Understand the Frye Standard, *Frye v. US* 1923.
- Understand the impact of *Daubert v. Merrell Dow Pharmaceuticals* 1993, *General Electric Co. v. Joiner* 1997, and *Khumo Tire v. Carmichael* 1999 on expert testimony.
- Learn the different court systems in which laboratory employees can testify to (superior, federal, civil).
- Understand the basis of criticisms of Trace Evidence examinations.
- Understand how discovery motions, court orders, and outside experts are handled by the SDPD Crime Laboratory.
- Courtroom etiquette
- Courtroom appearance and attire
- Understand voir dire
- Learn how to present qualifications, present the basis and method of examination, introduce evidence, present conclusions, and articulate the basis for conclusions.
- Be able to articulate laboratory accreditation standards and quality assurance policies and procedures.
- Be able to articulate Trace unit and Quality Manual policies and procedures.

LECTURES

- PREPARATION FOR COURT TESTIMONY
 - Jury's perception
 - Research current issues (Daubert, NAS report, PCAST, error rates)
 - Oral preparation prior to court (pre-trial conference)
- PREPARING COURT EXHIBITS
 - Purpose
 - Creating a PowerPoint
- PREPARE QUESTIONS AND ANSWERS FOR EXPERT TESTIMONY
 - Voir dire
 - Basic scientific principles
 - Defense questions

- **DISCUSS AND DEMONSTRATE EXPERT WITNESS TESTIMONY**
 - Court room etiquette
 - Communication with prosecutors and defense attorneys
 - Audio/video recording of testimony
 - Discuss and review testimony

SUGGESTED READING

- Vanderkolk, CH. 1 Objectivity-Subjectivity.
- National Academy of Sciences. Strengthening Forensic Science in the United States: A Path Forward. National Academies Press 2009.

PREPARATION FOR MOOT COURT

MOOT COURT

- Based on a Competency Test
- Court testimony evaluation form used to offer critique of testimony skills and effectiveness

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