Trace Evidence Unit

Quality Manual

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1 Scope

This manual follows the requirements specified by the Association of Crime Laboratory Directors/Lab Accreditation Board (ASCLD/LAB) International Program which utilizes the ISO/IEC 17025-2005 standards and the 2011 ASCLD/LAB International Supplemental Requirements.

1.1 The Trace Evidence Unit Quality Manual is written specifically for the analysts working in the Trace Evidence Unit and performing analysis in the following areas:

- Glass Analysis
- Gunshot Residue Analysis
- Ignitable Liquid Analysis
- Paint Analysis
- Hair and Fiber Collection
- Hair Examinations
- Fiber Analysis
- Fracture Match
- Tapes and Adhesives
- Lamp Filament Examinations
- Indented Writing
- General Chemical Analysis

1.2 Every case is unique and must be evaluated by the individual examiner. Not all possible analyses which may be encountered in casework can be appropriately covered in a procedures manual nor can all the possible variations be described. This quality manual is a guideline to help the analyst choose the best analytical scheme for the evidence submitted.

2 References

The documents generated by SWGMAT, the Scientific Working Group for Materials Analysis, and SWGGSR, the Scientific Working Group for Gunshot Residue, are used as a basis for creating the operational requirements in the testing methods section (5.4). SWGMAT consists of subgroups in the areas of Fibers, Glass, Paint, Hair, and Tape. Each of these subgroups is composed of analysts from within the area who help create and publish the peer-reviewed scientific documents. The documents are maintained on the website http://swgmat.org and http://www.swggsr.org/.

Additional references are contained within this manual.

Other requirements for the writing of this manual were provided by the laboratory quality manager.

3 Terms and Definitions

Reference the Labwide Quality Manual (section 3) for terms and definitions which apply to all sections.

3.1 Commonly Used Abbreviations

A list of abbreviations specifically used by each analyst is maintained on the trace drive (S:) under the Abbreviations folder.

Below are some common abbreviations used in the Trace Evidence Unit:

FTIR	Fourier Transform Infrared Spectrometer
µFTIR	microscope attachment with Fourier Transfer Infrared Spectrometer
MSP	Microspectrophotometer
GC/MS	Gas Chromatograph/Mass Spectrometer
GRIM	Glass Refractive Index Measurement System
SEM	Scanning Electron Microscope
EDS	Energy Dispersive X-ray Spectrometry
PLM	Polarized Light Microscopy
ESDA	Electrostatic Deposition Apparatus
ALS	Alternate Light Source
GSR	gunshot residue
pGSR	primer gunshot residue
IL	ignitable liquid
LPD	light petroleum distillate
MPD	medium petroleum distillate
HPD	heavy petroleum distillate
Std.	standard
env.	envelope
BC	bar code
w/	with
rep.	representative
v.	victim
s.	suspect
Cauc.	Caucasian
Neg.	Negroid
Mong.	Mongoloid
amt.	amount
pos.	positive
neg.	negative
sim.	similar
dissim.	dissimilar
GPF	gunpowder flake
DPA	diphenylamine

4 Management Requirements

4.1 Organization

Personnel Qualifications, Authorities, and Responsibilities

Section Chief (Chief Criminalist)

Qualification: A bachelor's degree in chemistry or a closely related field plus two years of experience in a forensic laboratory.

Authority and Responsibilities

- Coordinates unit activities by reviewing, prioritizing, and assigning new cases.
- Reviews case to become familiar with details of the crime and examines items of evidence to determine the appropriate testing methods.
- Performs analysis, prepares reports, and testifies in court.
- Consults with law enforcement officials, attorneys, and other public officials on crime scene investigations and methods of collecting, transporting and preserving evidence to ensure its integrity.
- Completes administrative duties by preparing activity reports, inventory reports, employee history binders, and other duties as assigned.
- Ensures compliance with ASCLD/LAB International requirements.
- Researches scientific literature and exchanges information with peers in other states or countries in order to stay abreast of the latest scientific advances.
- Maintains instruments and equipment used for the examination of evidence.
- Responsible for the technical operations and the provision of the resources needed to ensure the quality of laboratory operations.
- Reviews manuals annually.
- Reviews and approves all documents and forms.

Criminalists

Qualification: A bachelor's degree in chemistry or a closely related field.

Authority and Responsibilities

- Reviews case to become familiar with details of the crime and examines items of evidence to determine the appropriate testing methods.
- Performs analysis, prepares reports, and testifies in court.
- Consults with law enforcement officials, attorneys, and other public officials on crime scene investigations and methods of collecting, transporting and preserving evidence to ensure its integrity.
- Researches scientific literature and exchanges information with peers in other states or countries in order to stay abreast of the latest scientific advances.
- Maintains instruments and equipment used for the examination of evidence.

Unit Quality Manager

Qualification: The Section Chief will appoint one of the Criminalists to fulfill the role of Unit Quality Manager. This person should be qualified to conduct the majority of analyses performed within the unit.

Authority and Responsibilities

- Ensures quality assurance practices are being followed.
- Checks logbooks to make sure documentation procedures are being followed.
- Maintains the key log for the unit.
- Edits and revises manuals.
- Oversees validation of new instruments, methods, or procedures.
- Coordinates laboratory training of employees.

Unit Safety Officer

Qualification: The Section Chief will appoint one of the Criminalists to fulfill the role of Unit Safety Officer.

Authority and Responsibilities

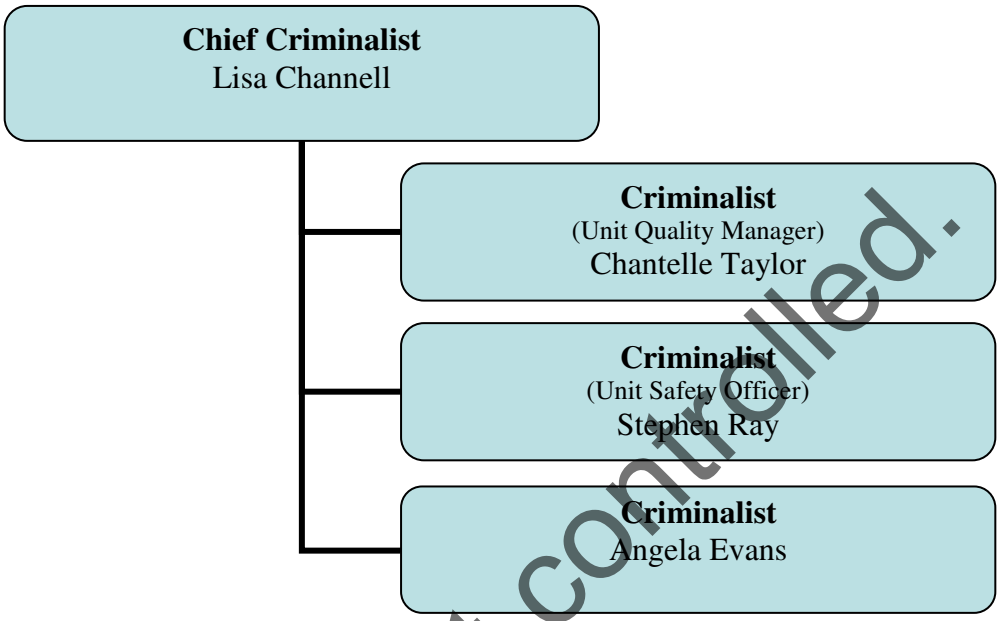
- Performs monthly checks of showers, eyewashes, and fire extinguishers located within the unit.
- Maintains the MSDS for the unit
- Maintains list of chemicals in the unit
- Fills out monthly reports to be sent to the Laboratory Health and Safety Officer
- Observes physical area for safety issues
- Keeps emergency contact information up-to-date

Laboratory Responsibilities

Managers have the responsibility and authority to receive and take action on employee concerns within their discipline.

The section chief has the responsibility for the technical operations and the provision of the resources needed to ensure the quality of laboratory operations. The section chief or designee will have the appropriate technical training and technical experience in each sub-discipline of trace evidence.

Organizational Chart



Each Criminalist is accountable to only one supervisor in the performance of their duties within the unit.

If the section chief will be absent for three days or longer, the section chief will designate an individual to assume their responsibilities during their absence. All affected personnel will be notified.

Employees will be notified of their responsibilities and expectations and will be given feedback on their actual job performance on a yearly basis during their performance evaluation.

Communication

Information concerning the laboratory will be conveyed to the unit by routine meetings, telephone, e-mails, or personal conversations.

4.2 Management System

Trace Evidence Quality Manual

The Trace Evidence Quality Manual (TR-DOC-01) is a compilation of policies and procedures for use in the Trace Evidence Unit. This manual is available on Qualtrax. Trace Evidence Analysts (Criminalists) are responsible for the familiarization and utilization of these policies and procedures. The Trace Evidence Quality Manual and Training Manual are reviewed annually by the Section Chief. Updates are made as needed to reflect changing organizational, technical, and procedural information. Reviews will be conducted on Qualtrax.

Circumstances may arise which require immediate deviations from the policies and procedures in this manual. In such situations, the request for exceptions to the policy will be submitted in writing to the Section Chief. The request must include an adequate description of the circumstances requiring the action, the proposed alternative policy or procedure, and the intended duration of the exception. The Section Chief will maintain documentation of the approved policy exception.

Mission Statement

The mission of the Trace Evidence Unit is to utilize scientific methodologies and instrumentation to examine physical evidence for the presence of fibers, hairs, paint, glass, tape, fire debris, lamp filaments, primer gunshot residue from suspects, and physical comparisons. Other miscellaneous analyses are performed when appropriate. Questioned samples are compared to known samples to determine if a common origin exists.

Supporting Manuals

The Trace Evidence Training Manual (TR-DOC-02) may be found on Qualtrax.

4.3 Document Control

Controlled documents will be prepared by personnel with adequate expertise in the subject.

Each new or revised internally generated controlled document is required to be reviewed and approved through Qualtrax. The official controlled documents and archived versions of all controlled documents will be maintained on Qualtrax.

The Trace Evidence Quality Manual must be reviewed and approved by the QA Manager, Section Chief, Scientific Operations Director, and Executive Director.

Trace Evidence Controlled Documents must be reviewed and approved by the Section Chief and QA Manager.

After the review(s) and approval(s) are completed, the document will be issued through the QA Manager or designee and posted on Qualtrax. All appropriate personnel will be notified and have access to the official electronic documents. Individuals may print hardcopies of internal documents as needed for personal use; however, these copies are unofficial.

Revised documents are subject to the same review, approval, documentation and issuance requirements of the original document as stated above.

External documents will be stored on Trace S: drive\Literature. Documents referenced in this manual will be found on Trace S:\Literature\Literature References in Trace QM. These documents will be the version or revision required for reference.

Documents will be available at all locations where work is performed. An uncontrolled copy is adequate for transportation to different areas or scenes.

Employees will destroy outdated documents upon receiving updated documents. It is the employee's responsibility to verify that they are using the current revision of any document.

The Section Chief is responsible for:

- Ensuring that reviews are completed annually on all documents in their section.
- Reviewing and approving all discipline specific controlled documents.
- Ensuring that the documents are scientifically suitable for issue.
- Ensuring that the documents contain the required quality assurance elements (i.e. QC, measurement of uncertainty, traceability)

4.4 Review of Requests, Tenders, and Contracts

Once accepted by the laboratory, any request for service submitted by a customer agency, either verbal or in writing, serves as a contract for service employing testing methods as described in Trace Evidence Quality Manual. The *ASCL Evidence Submission Form* (ASCL-FORM-12) shall normally be utilized to record the request, tender and contract with the customer.

By completing and submitting the submission sheet, the customer relinquishes all decisions regarding analytical processing and choice of methods to the ASCL.

Case related discussions are documented on an *Agency Contact Form* (ASCL-FORM-06), e-mail, or other document and recorded in the case file.

Before analysis begins, a review of the submission is conducted by the Section Chief and/or analyst to determine if there is anything more specific about the request and to determine if the laboratory has the capability and resources to perform the services requested (i.e. adequate standards, controls and approved test methods).

Requests from the medical examiner's office (Evidence Report in Justice Trax) should be added to the case file. ASCL-FORM-12 is not needed for these requests.

If the submission sheet needs to be amended, all affected personnel shall be notified.

4.5 Subcontracting of Tests and Calibrations

If the Arkansas State Crime Laboratory finds it necessary to transfer evidence to an outside laboratory (e.g. FBI, NMS), an *Inter-Laboratory Evidence Transfer Form* (see ASCL-FORM-07) must be completed and entered into the case file.

Any external laboratory that is to perform casework for the Arkansas State Crime Laboratory must be an accredited laboratory. This accreditation must be from an accrediting body that is recognized by the Arkansas State Crime Laboratory. These laboratories must provide the Arkansas State Crime Laboratory with a Certification of Accreditation.

If there will be a cost incurred to the customer, the customer must be notified and approve of the arrangement. This must be documented and placed in the case file.

4.6 Purchasing Services and Supplies

When the material or service must meet certain specifications in order to correctly perform the testing, these items and their specifications (i.e. manufacturer, type, grade or other technical data relevant to the supply or service) must be defined in the purchasing document.

Supplies, reagents and consumable materials that affect the quality of tests are not used until they have been inspected or otherwise verified as being in compliance with specifications established in the protocols section of the Trace Evidence Quality Manual. This verification will be documented in Qualtrax in the External Supply Request workflow.
As chemicals and reagents are requested, the analysts are responsible for initialing and dating containers with "Open Date". Supplies, reagents and consumable materials shall be stored in accordance with the manufacturer's recommendations.

An External Supply Request workflow shall be completed in Qualtrax for all items to be purchased.

Critical supplies

- GC Column
- MS filament
- Gunshot Residue Collection Kits
    - Provided to agencies at a minimum cost
    - Manufactured by Tri-Tech

Critical services

- Repair of analytical instrumentation
    - JEOL-SEM
    - Oxford Instruments-EDS
    - Foster+Freeman-GRIM3 and ffTA-1
    - Agilent Technologies-GC/MS
    - Perkin Elmer-FTIR

- Preventative Maintenance of analytical instrumentation
    - JEOL-SEM
    - Oxford Instruments-EDS
    - Provider approved by Quality Manager-balances

- Analysis of evidence
    - FBI Laboratory (soil, mitochondrial DNA cases)

Critical consumables

- Carbon disulfide
- ASTM resolution test mix
- Activated charcoal strips

4.7 Service to the Customer

The ASCL maintains open channels of communication with customers and is cooperative in providing a timely response to their concerns and questions regarding requests for services and the status of ongoing work. Any requests by the customer to be present during testing will be communicated to the Executive Director or Scientific Operations Director.

4.8 Complaints

Any staff member receiving a complaint should notify their supervisor. The complaint shall be documented and given to the supervisor. The supervisor shall forward the complaint to the Scientific Operations Director who will investigate the situation and notify top management when necessary. When the concern takes on the nature of a complaint about the laboratory's activities or deficiencies in the quality system, the supervisor will investigate the situation and forward all the information to the QA Manager.

Complaints or concerns may be submitted anonymously via electronic survey.

4.9 Control of Nonconforming Testing

All employees and supervisory personnel must be vigilant for any indication of nonconforming tests and work.

Refer to ASCL-DOC-01, Section 4.9 for the levels of non-conforming work and corrective action.

For Level 1 and 2 Non-conformities, the Section Chief and QA Manager should be notified immediately for consultation and to evaluate the significance of the nonconforming testing or work.

4.10 Improvement

The laboratory shall strive to continually improve the effectiveness of the Quality Management System. Opportunities for improvement are identified through various sources, including:

- Corrective and Preventive Action Requests
- Internal and external audits
- Employee suggestions

4.11 Corrective Action

Refer to ASCL-DOC-01, Section 4.11

4.12 Preventative Action

Refer to ASCL-DOC-01, Section 4.12

4.13 Control of Records

Historical case file records for Trace Evidence are kept with the labwide records, in the Trace Evidence library, or electronically in JusticeTrax.

Trace Evidence quality records are kept in the section and are accessible to Trace Evidence employees.

Observations, data and calculations shall be recorded at the time they are made and shall be identifiable to the specific task. If sampling has been performed by another analyst, it should be clearly indicated who performed the specific tasks. The laboratory case number, initials of the analyst, and date must be on every page of examination documentation.

The starting date will be recorded in the notes, dates should be recorded when the work is performed either in the notes or on the spectra. The ending date for the work will be the "draft complete" date recorded in JusticeTrax.

Any corrections to handwritten items will be made by an initialed, single strikeout (so that what is stricken can still be read) by the person making the change. Correction fluid or correction tape may not be used. If corrections are made to electronic documents, the original image will be kept in the case file and the corrected image will be added. The title of the document should indicate it has been amended or corrected and in what location, if possible.

When it is not feasible to incorporate the examination records in the LIMS case file, these records may be stored external to the LIMS case file. Large data files (such as those generated by the SEM/EDS during gunshot residue analysis) or extra spectra (FTIR, MSP, SEM/EDS, etc.) will be stored by case number on the instrument on which it is collected. Periodically, these files will be backed up and may be stored externally.

Verifications will be performed on positive hair associations which are probative in nature. The confirming analyst must be a competent, qualified analyst in the discipline. The confirming analyst will document their conclusions on the hair notes pages of the primary analyst. If a different conclusion is drawn by the confirming analyst, the case will be reviewed by another analyst, additional hairs will be mounted, or the hairs will be sent for another type of testing.

General abbreviations are located in section 3 of this manual. Those used by each analyst are stored on the S: drive.

5 Technical Requirements

5.1 Reagents/Chemicals

Purchased Reagents/Chemicals
Containers must be labeled with the following:

- Lot number
- Date opened
- Expiration date (if applicable)
- Initials upon opening
- Date received and initials

Prepared Reagents/Chemicals
Containers must be labeled with the following:

- Identity
- Date of preparation
- Date of expiration
- Initials of analyst

Prepared Reagents
Logbook must include the following:

- Identity
- Date of preparation
- Date of expiration
- Instructions on preparation of reagent
- Lot numbers of solvents and/or chemicals used in preparation of reagent
- A method to verify the reagent's reliability (if applicable)*
- Initials of the person preparing reagent
- Initials of the person verifying reagent (if applicable)

*The reliability testing shall occur before use or, if appropriate, concurrent with the test.
Note: Non-routine reagents prepared for one time use may be recorded with the above items in the laboratory case notes and any excess reagent discarded after use.

Prepared Chemicals
Logbook must include the following:

- Identity
- Date of preparation
- Date of expiration
- Instructions on preparation of chemical
- Lot numbers of solvents and/or compounds used in preparation of chemical
- Initials of the person preparing chemical

Chemicals and Reagents

- Solvents used for standards, extractions, or to dissolve samples or standards should be a high quality, low residue solvent.
- Water used in reagent preparation or as an extraction solvent should be either deionized (DI) or reverse osmosis (RO).
- All chemicals and commercial reagents are labeled when received and when opened with the date and initials of the individual receiving or opening them.
    - Working bottles of chemicals or reagents should be labeled at a minimum with the name of the chemical or reagent, lot number, expiration date, and initials of the analyst.
- All chemicals and commercial reagents should be disposed of and replaced when they fail to perform adequately under controlled conditions.
- All bottles of reagents prepared in the laboratory must be labeled with the contents, the date prepared, the expiration date, and initials of the analyst who prepared the reagent.
    - Directions for the preparation of commonly used reagents are found in the Reagent Logbook located in the Trace Evidence Unit Library.
    - The reagent preparation and quality check will be recorded in the reagent logbook.
    - The reagent should be checked prior to use in casework. The quality check should be recorded in the reagent logbook.
    - If a reagent fails the quality check, immediately dispose of the remainder of the reagent.
- Reagents prepared for non-routine examinations are tested with known samples and blanks. The results are recorded in the case file notes.
- Test strips are checked with appropriate standards and results are recorded in the case notes.
    - Test strips may continue to be used past their stated expiration date as long as they pass the quality control check.
- Flammable chemicals should be stored in a flammables cabinet.
- Each time a new chemical is received in the Trace Evidence Unit, a MSDS sheet should be obtained and provided to the section safety manager.

5.2 Personnel

Section Chiefs shall ensure the competence of all who operate specific equipment, perform tests, evaluate results and sign test reports. This is measured by completion of training according to the training manual. Past work experience and training may be substituted for the training program to the extent that it has been demonstrated to be relevant and sufficient, with the approval of the Section Chief and Scientific Operations Director.

Records will be kept as to when training is performed and completed. Each new employee will keep a training binder.

Personnel in training shall be supervised by a qualified analyst acting as the training officer in the specific discipline and are overseen by the section chief.

Training Program

Each category of testing within the Trace Evidence Unit has a section within the training manual. Basic training on laboratory procedures and a moot court are required with the completion of the first category of testing.

As an analyst trains in more areas within the unit, it will be up to the training officer and supervisor to determine if additional moot courts are needed.

Job Descriptions

Current job descriptions for personnel involved with testing shall be maintained in their Employee History Binder.

Competence Documentation

Section Chiefs shall authorize personnel to perform sampling, testing, issuing of reports, and operating particular types of equipment. This competency documentation shall be dated and signed by the Section Chief at the completion of each training unit and maintained in the Employee's History Binder. Each Employee's History Binder shall also contain a curriculum vitae or resume that includes educational and professional qualifications, training, skills, experience, and certifications.

Technical Personnel Qualifications

Analysts working in the Trace Evidence Unit shall possess a baccalaureate or an advanced degree in a natural science, preferably chemistry, or a closely related field.

Competency Testing

All trace evidence analysts that generate analytical results, regardless of academic qualifications or past work experience, shall satisfactorily complete a competency test (examination of sufficient unknown samples) in each category of testing in which they intend to perform casework. Satisfactorily completing a competency test means achieving the intended results. Failure to achieve the intended results would require review or retraining until testing achieves the intended results or the analyst is removed from performing that type of casework.

Literature

Trace Evidence analysts are encouraged to read and review appropriate new literature pertaining to the field of trace evidence. A library of textbooks, journals, and training materials is maintained within the section. In addition, a "Literature" folder on the S: drive contains numerous articles. Reading is documented on an excel spreadsheet for Literature Review maintained on the S: drive.

5.3 Accommodation and Environmental Conditions

Laboratory Separation

The Trace Evidence Unit is separated from other sections by being a key access only work area. The work rooms are shared with serologists. Cross-contamination may be prevented by working items recovered from suspect and victim or from different crime scenes in different rooms. Personal protection equipment and paper is changed between items and areas.

Access/Security

The Trace Evidence Unit is secured by lockable doors. The scrape down room is also secured by a lockable door. Each analyst in the unit as well as serologists working in the area has a door key and a set of lockable drawers and cabinets. Keys are issued only to the analyst and to the section supervisor. However, master keys that can open any door are issued to administrative personnel. Keys are also kept in the master key box; the Quality Assurance Manager has a key to the master key box. A section key box is located in the library. The section supervisor maintains the key to the section key box.

A key log is kept on the S: drive. A signature sheet key log is kept by the section chief.

Health and Safety Program

The laboratory has a *Health and Safety Manual* (ASCL-DOC-08) that must be followed by all employees and guests.

5.4 Test Methods and Method Validation

The Trace Evidence Unit analyzes a wide variety of different materials. A number of different methods may be needed in order to complete the requests made by the agency.

5.4.1 Glass Analysis

Scope

Fracture match examination should always be the first consideration when comparing glass samples. A physical match provides conclusive association between samples.

Glass analysis usually involves the comparison of a questioned glass sample with a known sample. The samples are analyzed by determining physical properties, refractive index, and elemental composition. The size of the glass particles may not allow for the determination of all physical properties.

Glass identification may also be performed by either identifying a substance as glass or by identifying the type of glass present in a sample. Physical properties and/or refractive index may be used to reach a conclusion.

The direction of an impact in glass or the sequence of impacts in glass may be determined in non-tempered glass samples. The glass must be intact in order to determine the sequence of impacts in a pane of glass.

Other substances may be present on glass samples. Care should be taken to preserve and or analyze other substances present.

Sample Recovery

From clothing and objects

- Visually examine the item for glass.
- Remove any visible glass fragments with forceps or probes.
- Hang clothing items to be examined on a rod over a clean white sheet of butcher paper. Small items may be held over the paper.
- Use a spatula to tap the item and then scrape it. Probes may be used for smaller objects with indentions.
- Collect the debris in an appropriate container.
- Examine the debris under the stereomicroscope.
- Remove any glass fragments with forceps and place in folded paper or a suitable container.

Known samples

- Check known sample submitted to determine if there is any difference within the sample.
- If a large sheet of glass is submitted, collect a representative glass sample. If it appears that differences may exist within the glass, samples may be collected from several areas.

Quality Assurance/Quality Control

- Examination area should be cleaned and paper changed between known and questioned items.
- Change gloves and lab coat between examining questioned and known items.
- Clean tools between use on questioned and known samples or use two sets of tools.
- Glass particles recovered from debris may need to be cleaned prior to analysis.
- A representative sample should be chosen from the known source to include the variations that may be seen within a glass sample.
- Record data in the logbooks.
- A safety shield or glasses which provide protection from UV light should be used when examining samples under UV lighting.

Testing Techniques

Physical Properties

- Check for a physical fit if adequate known and questioned samples are submitted. Fracture match determinations should be performed in accordance with Fracture Match protocols.
- Glass fragments may be distinguished from plastic by their relative hardness. If the careful application of pressure from a needle causes deformation of a particle, it is not glass. See optical properties below for further analysis of glass determination.
- Record visual observations regarding the known and questioned glass samples such as condition, color, fluorescence, surface features, curvature and thickness.
    - Prior to cleaning, visually inspect the glass using a stereomicroscope. Glass fragments to be tested should appear freshly broken. The relative sharpness of the edges, the appearance of conchoidal fracture, and transparency serve to recognize a freshly broken surface.
    - Comparing color may distinguish between two or more sources of glass. Sample size may affect apparent color. Side-by-side comparisons should be made with fragments of approximately equal and sufficient size and compared on edge over a white surface using natural light for optimal color observation. Significant color differences between glass fragments

can be used as the basis for exclusion. It is usually not possible to compare the color of microscopic glass fragments.
    - Fluorescence is determined by observing a glass fragment with short-wave (254 nm) and long-wave (350 nm) ultraviolet light. The surface of float glass that was in contact with molten tin during manufacturing should fluoresce under short-wave ultraviolet light. Different colors of fluorescence may be observed from glass fragments and should be recorded.
    - Surface features may be formed intentionally or accidentally as a result of manufacturing or during use. Surface features may include coatings, thin films, and mirrored backings. Etching, frosting, and texturing are intentional surface features. Accidental surface features may include mold marks, rough pits, and polish lines. Container class can have a distinct "orange peel" texture. Surface scratches, abrasions, pitting, and extraneous materials adhering to the glass should be examined and characterized prior to cleaning.
    - Determination of a curved surface can help distinguish flat glass from other types of glass including container, decorative, and ophthalmic. This may be done with the aid of a stereomicroscope if fragments are large enough.
    - Thickness measurements may be made on fragments possessing both original parallel flat surfaces. A micrometer or caliper with a precision of +/- 0.02mm or better should be used. Numerous fragments should be measured, if possible, to determine the variation in measured thickness. When two glasses show significant differences in thickness, as determined by the variation in the known glass, they can be eliminated as originating from the same source.
- If physical characteristics are dissimilar, no additional analysis needs to be performed.
- If examination of the physical properties have not excluded fragments from originating from a known source, further examination is required.

Cleaning and Sample Preparation

- Fragments may be cleaned manually using an appropriate solvent. The fragment may also be cleaned using detergent in an ultrasonic cleaner, then rinsed and dried.
- Large pieces of glass should be broken to create small fragments for mounting. If crushing a fragment is required, it should be done in a manner that will allow for the maximum recovery. Care should be taken to maintain the original surfaces on questioned fragments. If possible, use non-ultraviolet fluorescent surfaces for testing.
- The examiner should retain a portion of every sample analyzed and preserve it. If the entire sample is required for analysis, attempts should be made to maintain the sample in its tested state.
- Select an appropriate immersion liquid and mount the crushed glass on a slide. Some particles may require further crushing within the mounting medium. Cover with a cover slip.

Sampling vs. sample selection

- If it is reasonable to assume homogeneity of the sample (such as large pieces of glass fragments recovered from a specific area of broken glass), the report will state conclusions about the whole based upon the testing of a representative sample.
- If it is not reasonable to assume homogeneity of the sample (such as pieces from a pocket or small fragments from clothing debris), the report will state conclusions based upon the portion tested.

Optical Properties

- In order to determine if a particle is glass, it may be necessary to examine the particle under crossed-polarized light using a polarizing light microscope. Particles that exhibit complete extinction during 360 degrees of rotation are isotropic and indicative of glass. Anisotropic particles are eliminated as being glass.
- Automated Glass Refractive Index measurement (See GRIM3, Section 5.5)
    - Place the slide on the hot stage on the phase contrast microscope. Allow the slide to come to the temperature of the hot stage.
    - Focus on the image and align the microscope, including the substage condenser.
    - Adjust the phase rings until the rings are superimposed.
    - Change the temperature of the hot stage until the glass disappears. Note the temperature and begin the analysis using a temperature a few degrees higher than the disappearance temperature.
    - Upon starting the analysis, the instrument lowers the temperature through the match point for the glass. The contrast between the fragment and liquid is monitored and the match point, defined as the temperature at minimum contrast, is noted. This process is repeated as the temperature is then raised through the match point. These values are recorded and are averaged to give the match point temperature for the sample. The refractive index of the sample is automatically calculated from the calibration data.
    - Refractive indices for the questioned and known should be determined using the sodium D filter. In addition, C and F filters may be used.
    - A glass of known refractive index should be analyzed prior to each use to ensure that the instrument is operating within acceptable parameters.
    - Measure at least 5 fragments of the reference standard, at least 10 of the known (control) glass, and 5 to 10 of the questioned (recovered) glass, if possible.
    - Questioned glass particles with refractive index values that are within or overlap the observed RI range of the known glass sample are considered to be consistent in refractive index.
    - Questioned glass particles with refractive index values that are outside the observed RI range of the known glass sample are dissimilar. No additional analysis needs to be performed.

Elemental Analysis

- See SEM-EDS (Section 5.5).
- Place small fragment of glass on the carbon adhesive tape of a Scanning Electron Microscope (SEM) stub. Try to use flat sample surfaces when possible.
- In order to reduce charging, use the Low Vacuum mode of the SEM.
- Acquire spectra using scanning electron microscope-energy dispersive X-ray spectrometry (SEM-EDS).
- Spectra should be acquired from replicate samples, if possible, in order to measure the variability within the sample.
- Collect spectra and determine if there is a similar elemental composition between the questioned and known samples.
- If differences in the elemental composition of the questioned and known glass are observed, the samples may be considered dissimilar in elemental composition.

Glass Fractures

- Low-velocity impact fractures
  - Low-velocity projectiles produce cracks in glass which radiate outward from the point of impact (radial cracks). Concentric cracks can form around the point of impact.
  - The sequence of multiple impacts can be deduced when the cracks caused by a subsequent impact terminate at previously formed cracks.
  - Observation of the ridges on the radial cracks can often determine the direction of force. The ridges on the radial cracks nearest the point of impact should be observed. A stereomicroscope may be used to observe the ridges.
  - Ridges on radial cracks are at right angles to the rear (side opposite) of the impact (4R Rule). This rule is unreliable for laminated glass, tempered glass, and small windows tightly held in a frame.

- High-velocity fractures
  - High-speed projectiles striking glass produce a cone or crater. If the projectile passes through the glass, the opening on the exit side will be larger than the opening on the entry side.
  - The size of the hole and the diameter of the crater cannot be used to reliably predict the size of the projectile.
  - Projectiles passing through the glass at an angle produce an elongated hole.
  - Radial cracks may also develop from high-velocity impact. The 4R Rule applies. A stereomicroscope may be used to observe the ridges.
  - The sequence of multiple impacts can be deduced when the cracks caused by a subsequent impact terminate at previously formed cracks.

- Thermal fractures
  - In non-tempered glass, a typical heat crack is curved, has a smooth edge, and has no indication of the point of origin of the crack.
  - Localized heating of thick pieces of glass can cause cracks with a feathered appearance. The side to which the heat was applied cannot be determined from fracture edges.

Glass Fibers

- Compare the physical properties of known and questioned glass fiber samples. The color of resin, UV fluorescence, presence of slugs, and other outstanding characteristics should be recorded.
- Mount a sample of the fibers in a suitable mounting liquid for microscopic examination. Determine the diameter range of the fibers using a calibrated reticule.

Interpretation of Results

- For a positive association in a glass comparison:
  - If all physical properties are consistent, the thickness of the questioned glass should fall within the range of the known glass samples.
  - The refractive index of the questioned glass sample should be within the range of the known glass sample.
  - The elemental content of the questioned sample should be consistent with the elemental content of the known sample.
- If any of the physical, optical, or elemental properties are not consistent, the questioned and known items cannot be associated.

Reporting Suggestions

- The techniques (e.g. ultraviolet light, caliper, glass refractive index measurement system, stereomicroscopy, scanning electron microscope/energy dispersive X-ray spectroscopy, etc.) used for the analysis will be reported.
  - Item ___ was examined using stereomicroscopy, ultraviolet light, and glass refractive index measurement system.
  - Items ___ and ___ were examined visually.

- For glass samples or fragments with a positive association:
  - The glass fragments recovered from Item ____ were consistent in physical properties, refractive index, and elemental composition with the known glass sample.
  - (Number examined) of (total particles) glass particles recovered from Item ____ were consistent in physical properties, refractive index, and elemental composition to the known glass sample.

    - The glass fragments from Item ___ could have originated from the broken glass represented by known Item ___ or another source of broken glass with the same properties.

- For glass with no association:
    - The glass fragments from Item ____ were dissimilar to the known glass sample in (physical properties, refractive index, and/or elemental composition).

- If no glass particles are recovered:
    - Item ____ was examined for the presence of glass fragments. No glass was recovered.
    - No glass fragments were recovered from Item ____ for comparison to Item ___.

- If known samples are needed:
    - Glass fragments were recovered from Item ____. If further testing is desired, known samples of broken glass are needed for comparison.

Literature References

- ASTM E1967, Standard Test Method for the Automated Determination of Refractive Index of Glass Samples Using the Oil Immersion Method and a Phase Contrast Microscope.
- Scientific Working Group for Materials Analysis Documents (SWGMAT):
    - "Initial Examinations of Glass"
    - "Collection, Handling, and Identification of Glass"
    - "Glass Refractive Index Determination"
    - "Elemental Analysis of Glass"
    - "Glass Fractures"

5.4.2 Gunshot Residue

Scope

This procedure is for the analysis of primer gunshot residue by scanning electron microscopy coupled with energy dispersive X-ray spectrometry. The residue may be collected by the officer in pre-packaged gunshot residue collection kits or by the analyst in the laboratory. The analysis of primer gunshot residue can determine if particles characteristic to gunshot residue are present. However, the analysis of primer gunshot residue cannot determine who fired a weapon.

Gunshot residue collection kits and clothing from suspects may be tested. Other objects which have been sampled using a collection kit or are sent in for analysis may also be tested.

Shoes and inner garments will not routinely be tested.

Gunshot residue collection kits and clothing from gunshot wound victims will not be tested for primer gunshot residue.

Kits collected more than six hours after the time of the shooting will not be tested.

Kits or clothing collected from individuals with a firearm in their possession, who admit being in the vicinity of a firearm at the time of discharge, who admit or were allowed to wash their hands, or who admit to firing a weapon will not be tested.

Kits containing only swabs will not be tested for primer gunshot residue.

Quality Assurance

- The trace evidence unit analysis area is located on a different floor than the main firearms work area.
- Items of clothing should be opened one at a time and labeled.
- All items should be examined on a clean sheet of white butcher paper.
- All bench areas should be cleaned between each case.
- Items to be examined for blood or tape lift collection should have the gunshot residue collected before any other analysis.
- Each stub should be labeled with the item number and area sampled or the pre-stamped number from the manufacturer should be recorded on the worksheet. The stub holder should also include the case number, item number, and sampled areas.
- Each stub should be marked so that it can be returned to the same position in the holder if removed.
- Samples from different suspects are not placed in the chamber together. Only one gunshot residue case is placed in the chamber for analysis.
- Gloves and lab coats should be changed between items from different suspects.

Sample Collection and Preparation

Suspect clothing or other items

- Place the item to be examined on a clean white sheet of paper, if possible.
- Label stub.
- Press carbon-coated adhesive stub on area to be tested. Store stubs in holders labeled with case number, item number, and area sampled.
- Record information in notes.

Gunshot residue kits

- Open gunshot residue kits one at a time.
- Inspect gunshot residue kits for the type used. Record this information in notes.
- Label each plastic stub holder. Label the stub or record the unique ID number.
- Record name of person sampled from information sheet. Scan the information sheet into the case file.

Testing Techniques

See Scanning Electron Microscopy/Energy Dispersive X-ray Spectrometry (Section 5.5)

Standards and Controls

- An area on the control stub will be analyzed prior to every run to ensure proper setup.
- A blank stub is run with each batch of stubs.
- The internal "quant calibration" is established by examination of the Cobalt standard.
- The "threshold" is established by examination of the Cobalt and Rhodium standard.
- Performance is checked monthly using the Plano standard.

Automated Analysis

- Place stubs in the sample holder of the SEM.
- Close sample compartment and obtain a vacuum.
  - "HV" or high vacuum mode may be used for most samples with carbon coated adhesive stubs.
  - "LV" or low vacuum mode should be used for samples that do not have carbon adhesive or which contain a large number of particles (such as fibers) that may produce excessive charging.
- Optimize the SEM conditions for Energy Dispersive X-ray Spectrometry (EDS) collection.
- Enter case information in the EDS system.

- Use the cobalt and rhodium standard stub to set the threshold levels of the backscatter detector.
- Include control stub. This is usually sampled from a firearms analyst immediately after test firing a firearm. Test threshold level on known area of stub.
- Start automated gunshot analysis.

Confirmation of particles

- Potential GSR particles detected by automated analysis are relocated and confirmed manually by acquiring an x-ray spectrum from the particle.
- If no GSR-related particles are detected on a sample, the analyst should ensure that the focus and general operating conditions have not varied and are correct. If changes to the operating conditions have occurred, the automated analysis should be performed again.
- Include summary of instrument results in case record.
- Include spectra and images of confirmed GSR particles in case record.
- Raw data files are very large and are stored on the hard drive of the EDS computer.

Samples from Ammunition

- In some cases, it may be relevant to establish whether the particles detected were consistent with the ammunition that was used. This may be especially helpful in cases where the ammunition does not contain one of the three main elements commonly found in gunshot residue.
- Collect particles from cartridge case.
    - Using a wooden applicator stick, transfer particles from the fired case to a collection stub.
    - Cover the opening of the cartridge case with the collection stub and firmly tap the base of the cartridge case.
    - Ensure that the particles are firmly attached to the carbon adhesive.
- Run these samples for comparison.
- Almost all GSR classified as characteristic in elemental composition is derived from ammunition primers based on the "Sinoxid" formulation which contains lead styphnate (possible with other lead components), antimony sulfide, and barium nitrate. The particles from this type of primer must contain lead, antimony, and barium. Other additional components may be present from the primer composition, other ammunition components, or from the firearm.
- Older ammunition or specialty ammunition may not contain one or more of the three typical elements.

Interpretation of Data

Individual particles may be classified as characteristic of, consistent with, or commonly associated with GSR based on their elemental composition and morphology. The morphology of GSR related particles should indicate formation at highly elevated temperatures.

Characteristic

- Particles classified as characteristic of GSR have elemental compositions rarely found in particles from any other source.
- Characteristic particles from Sinoxid-type primers must contain lead, antimony, and barium.
- Characteristic particles from calcium silicide based primers with tin foil (older S & B) must contain lead, barium, calcium, silicon, and tin.

Consistent

- Particles classified as consistent with GSR have compositions that are also found in particles from a number of relatively common, non-firearm sources.
- Particles consistent with gunshot residue contain a combination of two of the three elements (lead, antimony, and barium).
- Some ammunition (e.g. non-Sinoxid primers or "lead-free" ammunition) will not generate characteristic lead-antimony-barium particles. Consequently, the following elemental profiles are considered to be consistent with GSR:
    - Barium, calcium, silicon
    - Antimony, barium
    - Lead, antimony (levels higher than trace amounts)
    - Barium, aluminum
    - Lead, barium
    - Lead, barium, calcium, silicon
    - Titanium, zinc
    - Strontium
- Consistent particles are not usually reported unless there is an indication that the ammunition used may not generate characteristic particles.

Commonly Associated

- Particles classified as commonly associated with GSR have compositions that are commonly found in environmental particles from numerous sources. However, when present in addition to particles that are characteristic of and/or consistent with GSR, these particles can be of significance.
    - Lead (with only trace levels of antimony)
    - Lead
    - Antimony
    - Barium (in the absence of sulfur)
    - The above particles may also contain one or more of the following: Silicon, calcium, aluminum, copper, and trace amounts of iron, sulfur, phosphorus, zinc, nickel, potassium, chlorine, and tin.
- Particles commonly associated with GSR are not generally reported but may be used as supporting particles.

Negative

- Insufficient gunshot residue particles were identified – No characteristic particles were identified.
- The absence of particles may be from many factors:
    - The person did not discharge a firearm.
    - Hands or clothing were washed.
    - Hands were wiped.
    - Gloves were worn.
    - Sweating profusely.
    - Environmental factors including wind or rain.
    - Excessive blood or debris on the hands
    - Normal physical activity within 4 to 6 hours passing between firing and sampling.
    - The weapon did not produce primer residue when discharged.
    - Physical barrier preventing discharge.

Reporting Suggestions

- The instrumentation and criteria should be included in the report:
    - The stubs were examined by scanning electron microscopy with energy dispersive x-ray spectrometry and analyzed for morphology and elemental composition of gunshot residue particles.

- Characteristic particles
    - Particles characteristic of gunshot residue were present on the sample from _____.
    - One particle characteristic of gunshot residue was present on the sample from _____.

- A disclaimer should be included on items containing characteristic particles:
    - The presence of these particles may be the result of discharging a firearm, handling a firearm, being in the proximity of a firearm at the time of discharge, or coming into contact with an object bearing gunshot residue.
    - The presence of these particles is consistent with the item having been in the vicinity of a firearm when it was discharged or having come into contact with another item bearing gunshot residue.

- Consistent particles with indication of ammunition that does not produce characteristic particles:
    - Particles consistent with gunshot residue were present on the samples from ____. These particles are found in gunshot residue but may also originate from other sources.

- If gunshot residue results are negative:
    - Insufficient gunshot residue particles were identified. The results may be considered negative. The absence of gunshot residue does not eliminate that person from having discharged a firearm.

- If comparison to known ammunition is conducted:
  - The elemental composition of the particles from item ___ corresponded to the elemental composition of the particles from the cartridge case. This proportion, however, could be found in other ammunition.

- Not analyzed:
  - Admits to discharging a firearm:
    - The gunshot residue kit from ____ was returned unexamined. It is the policy of the Trace Evidence Unit that gunshot residue kits collected from an individual who admits to discharging a firearm will not be analyzed. The individual was known to have been in a gunshot residue environment.
  - Admits to being near a discharging firearm:
    - The gunshot residue kit from ___ was returned unexamined. It is the policy of the Trace Evidence Unit that gunshot residue kits collected from an individual admitting to be near a firearm at the time of discharge will not be analyzed. The individual was known to have been in a gunshot residue environment.
  - Firearm in possession:
    - The gunshot residue kit from ___ was returned unexamined. It is the policy of the Trace Evidence Unit that gunshot residue kits collected from an individual with a weapon in their immediate possession will not be analyzed. The individual was known to have been in a gunshot residue environment.
  - Kit collected after 6 hours:
    - The gunshot residue kit from ____ was returned unexamined. It is the policy of the Trace Evidence Unit that gunshot residue kits collected longer than six hours after a shooting will not be analyzed.
  - Victim:
    - The gunshot residue kit from ___ was returned unexamined. It is the policy of the Trace Evidence Unit that gunshot residue kits collected from the victim of a firearm discharge will not be analyzed. The victim was known to have been in a gunshot residue environment.
  - Swabs:
    - The gunshot residue kit from ___ was not analyzed. The laboratory does not have the capability of analyzing kits containing only swabs.

Literature References

- ASTM E1588, Standard Guide for Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray Spectrometry.
- Scientific Working Group for Gunshot Residue (SWGGSR), “Guide for Primer Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray Spectrometry.

5.4.3 Ignitable Liquid Analysis

Scope

Evidence may be submitted for the detection of ignitable liquids. The evidence is examined, residues are extracted, and samples are analyzed by gas chromatography – mass spectrometry.

The evidence must be submitted in an airtight container to protect the integrity of the evidence and the validity of the result.

Analytical Approach

- Briefly open each container, make a visual inspection and record a description of the contents on the Ignitable Liquid Analysis Worksheet.
- Do not intentionally smell the contents but make a notation of any noticeable odor.
- If a non-aqueous liquid is present, it may be tested "neat". See direct injection procedure.
- If an aqueous liquid is present or for most debris samples, the sample will be collected by Passive Adsorption-Elution. See procedure below.
- Solvent extraction may be the best testing method for some types of evidence (e.g. a dried liquid on glass or swabs).
- If a latent print examination is requested on a container, remove contents of the container to a clean airtight jar for further analysis. If no liquid is present, a solvent extraction of the interior of the container may be conducted.
- The samples are examined by GC-MS. See procedure
- The GC-MS Total Ion Chromatogram (TIC) and extracted ion chromatography (EIC) for case samples are compared to known reference samples.
- Any sample extract vials generated during the analysis are returned in the container.

Safety Considerations

- Intentional inhalation of fumes may be harmful.
- Carbon disulfide should be used in the hood.
- Samples which have been in the oven may be very hot. Caution should be taken when removing these items.

Minimum Standards and controls

- Each new bottle of carbon disulfide must be tested prior to use in casework. Approximately 2 mL of carbon disulfide is placed in a vial and a sample is injected. It must be free of any significant peaks which would interfere with the

analysis. The spectra for the date the bottle was opened are kept in the QC folder on the GC/MS with the lot number recorded.

- Each new package of carbon strips should be tested prior to use in casework. A strip will be placed in a vial with carbon disulfide and tested. It must be free of any significant peaks which would interfere with the analysis. The spectra for the date the package was opened are kept in the QC folder on the GC/MS with the lot number recorded.
- A sensitivity standard (ASTM E1387) will be run with each sequence to ensure minimum detection limits and reproducibility of retention times. The spectra are kept in a logbook beside the GC/MS.
- A sample of alcohols, gasoline, kerosene, and diesel fuel (AGKD) will be run at the beginning of each sequence to ensure the reproducibility of chromatographic patterns. If the sequence runs longer than the work day or overnight, then the AGKD sample should be included at the end of the sequence. The spectra are kept in a logbook beside the GC/MS.
- There must be at least one blank between each case sample injected. The blank is solvent only and must be the same solvent used on the other samples in the case. A blank for each item is included in the case file.
- Reagents used will be at least reagent grade.
  Clean tweezers used to remove carbon strips from paper clip in carbon disulfide between each sample.

Sampling Techniques

Passive Adsorption-Elution

This technique is used for most samples. It is a headspace concentration method also known as the charcoal strip method in which volatile ignitable liquids are removed from the static headspace above the sample and trapped on a charcoal strip then eluted with a solvent and analyzed.

- A charcoal strip attached to a bent paper clip is placed in the container by a string.
- For heated passive adsorption, the container is resealed and placed into an oven with temperature between 60º C and 70º C for a minimum of four hours. The container may be left overnight.
- For ambient passive adsorption, the container is resealed and left at room temperature for a minimum of sixteen hours.
- If heated, the container is removed from the oven and allowed to cool.
- The charcoal strip is removed and placed in an injection vial.
- The charcoal strip is eluted with a small amount of carbon disulfide.
- The eluent is analyzed by gas chromatography-mass spectrometry (GC-MS).

Direct Sampling

This technique is used for non-aqueous liquid samples. Non-aqueous samples may also be examined with passive adsorption-elution. The whole liquid is tested.

- Neat liquid samples are diluted with carbon disulfide and analyzed via gas chromatography-mass spectrometry.
- A solvent blank must be included.

Solvent wash or solvent extraction

This is a sensitive technique which can be useful for the extraction of petroleum products from non-porous surfaces, debris, or very small samples.

- Place as much of the sample as is practical into a clean beaker.
  - If possible select a representative portion of the sample.
  - If only a representative portion is extracted, include which portion in notes.
- Pour a small amount of pentane or carbon disulfide over the sample.
- Decant the solvent into a separate clean beaker.
- Evaporate the solvent to a small volume to concentrate any ignitable liquid residues that may be present.
- The solvent blank shall be treated in the same manner as the sample.
- Analyze by gas chromatography-mass spectrometry.

Headspace

The procedure is useful when volatile oxygenated products are suspected. It is the least sensitive technique but the sample remains in approximately the same condition in which it was submitted therefore repeat analyses are possible. The volatile components present in the air space above debris are collected.

- Punch a hole in the top of the lid and cover hole with tape or place a rubber sleeve stopper in the hole.
- The container is left at room temperature or placed in an oven with temperature between 60º C and 70º C until equilibrium is reached.
- Upon removal, a gas-tight syringe is inserted through the tape or stopper into the hole. The syringe is pumped three times and then removed from the lid. The hole is resealed.
- The optimum sample size will vary with chromatograph column and conditions but should be 0.5 to 2.0 ml.
- Inject vapor sample directly into GC-MS.

Testing Technique

See Gas Chromatography-Mass Spectrometry (Section 5.5)

Samples are analyzed by GC-MS which provides a total ion chromatogram (TIC) and extracted ion chromatograms (EIC) for groups of specific ions.

Analytical Procedures

- An autotune must be performed prior to testing case samples.
- Each day the GC-MS is used for case samples, the AGKD sample and the Sensitivity sample must be injected prior to case samples. This will serve to check the resolution and sensitivity of the instrument.

- Visual comparison is made to previous sample to verify consistency.
- Mass spectra of specific peaks may be verified.

- Inject case samples
  - The autosampler may be used. Blanks must be injected between each case sample.

Interpretation of Results

All peaks used to determine the presence or absence of an ignitable liquid should be identified by their mass spectrum. This may be done by pattern-matching the extracted ion chromatograph for selected ions with that of a known primary standard of an ignitable liquid or by identifying specific compounds by their mass spectrum and comparing these to that of a known primary standard.

Characterization of ignitable liquids by class should be done in accordance with the ASTM Ignitable Liquid Classification Scheme. (Section 10.2 of ASTM E1618)

Compound Class	**Ions of Interest for Petroleum Distillates**
Alkane	43, 57, 85, 99
Aromatic	91, 105, 119
Cycloparaffin	55, 69, 83
Naphthalene	128, 142, 156

Compound Class		**Ions of Interest for Gasoline**
Alkane		43, 57, 71, 85
Aromatic	Alkylbenzene	91
	C_2 Benzenes	106
	C_3 Benzenes	120
	C_4 Benzenes	134
Naphthalene	Naphthalene	128
	Methylnaphthalene	142

- A chromatogram with no significant peaks may be reported as negative given that all QA/QC checks were acceptable.
- A chromatogram with significant peaks which may be attributed to the sample matrix but that do not match that of an ignitable liquid may be reported as negative given that all QA/QC checks were acceptable.
- A chromatogram with significant peaks not attributed to the sample matrix that do not match that of an ignitable liquid may be reported as inconclusive given that all QA/QC checks were acceptable.
- A chromatogram with significant peaks that match that of an ignitable liquid that are not attributed to the sample matrix may be reported as positive for that class of ignitable liquid given that all QA/QC checks were acceptable.

Report Wording Suggestions

- The report should include a brief description of the sampling technique and analytical technique used.
    - (example) The samples were extracted by passive adsorption-elution and examined by gas chromatography and mass spectrometry.

- Negative
    - No ignitable liquid residues were detected in item _____.
    - Ignitable liquids may evaporate or can be totally consumed during a fire. A negative result does not preclude the presence of an ignitable liquid during a fire.

- Light Petroleum Distillate
    - Item _____ contained residues consistent with the light petroleum distillate class of ignitable liquid.
    - A residue of a light petroleum distillate was detected in item _____.
    - Examples of a light petroleum distillate include some camp fuels, some cigarette lighter fuels, and petroleum ether.

- Gasoline
    - Item _____ contained residues consistent with gasoline.
    - A residue of gasoline was detected in item _____.

- Medium Petroleum Distillate
    - Item _____ contained residues consistent with the medium petroleum distillate class of ignitable liquid.
    - A residue of a medium petroleum distillate was detected in item _____.
    - Examples of a medium petroleum distillate include some charcoal starters, some paint thinners, and some dry-cleaning solvents.

- Heavy Petroleum Distillate
    - Item _____ contained residues consistent with the heavy petroleum distillate class of ignitable liquids.
    - A residue of a heavy petroleum distillate was detected in item _____.
    - Examples of a heavy petroleum distillate include kerosene, some charcoal starters, some jet fuels, and Diesel fuel.

- Isoparaffinic Products
    - A residue of an isoparaffinic product was detected in item _____.
    - Note: may be designated as light, medium, or heavy range.
    - Examples of isoparaffinic products include aviation gasoline, some charcoal starters, some paint thinners, some copier toners, and some specialty solvents.

- Aromatic Products
    - A residue of an aromatic product was detected in item _____.
    - Note: may be designated as light, medium, or heavy range.
    - Examples of aromatic products include some paint and varnish removers, some automotive parts cleaners, xylene and toluene-based products, some specialty cleaning solvents, some insecticide vehicles, and fuel additives.

- Naphthenic-Paraffinic Products
    - A residue of a naphthenic-paraffinic product was detected in item _____.
    - Note: may be designated as light, medium, or heavy range.
    - Examples of naphthenic-paraffinic products include cyclohexane based solvents, some charcoal starters, some insecticide vehicles, some lamp oils, and some industrial solvents.

- Oxygenated Solvents
    - A residue of an oxygenated solvent was detected in item _____.
    - Note: may be designated as light or medium range.
    - Examples of oxygenated solvents include alcohols, ketones, some lacquer thinners, some fuel additives, surface preparations solvents, some industrial solvents, metal cleaners, and gloss removers.

- Miscellaneous
    - Item _____ contained residues consistent with a miscellaneous class of ignitable liquids.
    - Item _____ contained a miscellaneous class of ignitable liquids.
    - Note: may be designated as light, medium, or heavy range.
    - Examples of a miscellaneous class of ignitable liquids include single component products, some blended products, some enamel reducers, turpentine products, and some specialty products.

- Terpenes
    - Terpenes were identified in item _____. Terpenes consistent with those detected are essential components of turpentine and are naturally occurring in some types of wood.

References

- ASTM E1618: Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry
- ASTM E1386: Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction
- ASTM E1387: Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography
- ASTM E1388: Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples
- ASTM E1412: Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Activated Charcoal.

5.4.4 Paint Analysis

Scope

Fracture match examination should always be the first consideration when comparing paint samples. A physical match provides conclusive association between samples.

Paint analysis usually involves the comparison of a questioned paint sample with a known sample. The samples are analyzed by a variety of macroscopic, microscopic, chemical, and instrumental methods.

Automotive, architectural, industrial, marine, tools, and other paints and coatings may be examined.

An important aspect of forensic paint analysis is the identification of the possible makes, models, and years of manufacture of motor vehicles from paint collected at the scene of a crime or accident. Some samples may be searched against the Paint Data Query (PDQ) Database.

Other substances may be present in or on the paint samples. Care should be taken to preserve and or analyze other substances present.

Sample Recovery

From clothing and objects

- Visually examine the item for paint.
- Remove any visible paint fragments with forceps or probes. Cut out any paint smears present if large chips are not found.
- Hang clothing items to be examined on a rod over a clean white sheet of butcher paper. Small items may be held over the paper.
- Use a spatula to tap the item and then scrape it. Probes may be used for smaller objects with indentions.
- Collect the debris in an appropriate container.
- Examine the debris under the stereomicroscope.
- Remove any paint fragments with forceps and place in folded paper or a suitable container.

Known samples

- Known samples should include all layers of paint and substrate, if possible.
- Known samples should be collected from near the area of damage.
- Check known sample submitted to determine if there is any difference within the sample.
- If a liquid paint sample is submitted, the liquid may be applied to a clean glass slide and dried for analysis.

Quality Assurance/Quality Control

- Examination area should be cleaned and paper changed between known and questioned items.
- Change gloves and clean tools between known and questioned items or use two sets of tools.
- Solvent tests are destructive and should only be performed if enough sample is present.

Testing Techniques

Physical Examination

- Examine the samples for a potential fracture match.
- Examine the sample with a stereomicroscope.
  - Note the layer sequence, color, layer thickness (relative), texture, and any unusual features.
  - A cross-section or angled cut may help to determine the layer sequence.
- Thin cross-sections may be cut along an edge of the paint sample. The thin sections may be mounted in a suitable medium for examination with a comparison microscope using transmitted and/or reflected light.
- If differences are observed between the questioned and known samples, the samples are dissimilar. No further testing is needed.

PLM

- This method is optional and presumes training and experience in advanced microscopy.
- A comparison of the pigment particles, fillers, and extender particles present in paint may be conducted by polarized light microscopy.
- Suitable samples for examination by PLM include but are not limited to thin sections, smears, and dispersed particles in a mounting medium.
- PLM examinations may be especially useful in the examination of architectural paints.

Instrumental analysis

FTIR (See Section 5.5)

Fourier transform infrared spectroscopy may be used to obtain information about binder, pigments, and additives used in coating materials. Transmittance is the preferred method but reflectance may be used in some cases. The microscope accessory permits the analysis of a small sample area.

- The sample may be prepared by cutting a thin cross-section or a small piece of a single layer and flattening with a roller or compression cell.
- The sample is placed on a KBr window or left in the compression cell and placed on the FTIR microscope stage.
- Spectra are obtained for each layer.
- Replicate analyses may be performed to ensure reproducibility or to show variability within a sample.
- Compare the spectra for the questioned and known layers.
- If any significant differences are observed in the spectra, the samples are dissimilar. No further testing is needed.

Microspectrophotometry (MSP)

A comparison of the pigments used in paint can be accomplished by transmission microspectrophotometry.

- Microspectrophotometry will be performed on brightly colored samples only. White, gray, black, and brown paint samples are not usually suitable for MSP analysis.
- Samples should be prepared to a similar thickness. This may be performed by flattening the sample on a slide.

Scanning Electron Microscopy/Energy Dispersive X-ray Spectrometry (SEM/EDS)

SEM/EDS can be used to characterize the elemental composition of the paint samples.

- The paint samples should be flat and mounted on a carbon coated adhesive stub.
- A spectrum is collected from each layer of the questioned and known sample.
- Comparison of the composition of layers is generally performed by a non-quantitative method using a larger beam raster area.
- The analysis of individual pigment or metallic particles in paint layers by spot analysis may be useful.

Solvent/Microchemical Tests

Solubility or microchemical tests are optional. They may be used for further characterization of a paint sample.

- The known and questioned paint particles should be of the same approximate size, treated in the same manner, and compared side-by-side.
- Place the paint particles in welled slide or spot plate in the field of view of a stereomicroscope.
- Apply the reagent or solvent directly to the particles. Any resulting reaction, or lack thereof, is noted.

- Acetone
    - Soluble: Lacquer
    - Insoluble: Enamel
- Xylene (applied to lacquer)
    - Soluble: Dispersion Lacquer
    - Insoluble: Solution Lacquer
- Diphenylamine reagent (applied to solution lacquer)
    - Positive (vivid blue at acetone solvent fringes): Nitrocellulose
    - Negative: Acrylic Lacquer
- Chloroform (for confirmation of diphenylamine reagent results):
    - Insoluble: Nitrocellulose
    - Soluble: Acrylic Lacquer

Paint Data Query (PDQ)

If the request is made for determination of possible make/model of a vehicle, the sample may be entered into the PDQ.

- To be suitable for a PDQ search, the original paint must be present with at least one layer of the primer present.
- Each layer of the sample is first analyzed by FTIR. The appropriate information from each layer is entered into the PDQ and a search is conducted.
- If successful, the search results will be reported as to the possible make, model, and range of years in which the paint may have been used.
- It should be noted that in order to use the Paint Data Query (PDQ), sixty known paint samples must be submitted each year to the Royal Canadian Mounted Police (RCMP) PDQ Maintenance Team via the FBI.

Interpretation of Results

- For a positive association of a paint comparison:
    - The questioned paint exhibits the same physical properties as the known sample. Some variations may exist due to the transfer and subsequent damage of the questioned paint.
    - The optical properties of the questioned sample should be consistent with the known sample.
    - The chemical properties (from FTIR or microchemical testing) of the questioned sample should be consistent with the known sample.
    - The elemental properties of the questioned sample should be consistent with the known sample.
- If any of the physical, optical, or chemical properties are not consistent, the questioned and known samples cannot be associated.

Report Writing Suggestions

- Techniques used in the analysis will be listed on the report.
  - (example) Item __ was examined using stereomicroscopy, Fourier Transform Infrared Spectroscopy (FTIR), Microspectrophotometry (MSP), and Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (SEM-EDS).
- Single or Multi-layer samples:
  - The (color) paint in item _____ was consistent in color, texture, and chemical composition to the known paint sample from item _____.
  - The (color) paint in item _____ consisted of (number) layers of paint which were consistent in color, texture, layer sequence, and chemical composition to the paint in the known paint sample from item _____.
- Conclusion Statement:
  - The paint from (questioned) items could have come from the (known) item or any other source with similar characteristics.
- Smears:
  - (Color) smears were located on item _____. These paint smears were similar in color to the paint from item _____. However, due to the lack of sample, no further examinations were conducted.
- Dissimilar:
  - The (color) paint in item _____ was dissimilar to the (color) paint from item _____ in __ (color, layer structure, chemical composition, etc.).
- None:
  - No paint was recovered from item _____.
  - No automotive paint was recovered from item _____.
- PDQ Search:
  - A search of the PDQ database indicated that the (color) paint recovered from item ( ) was consistent with paint used on the following vehicles:
    Year – year make model
    Year – year make model
    It should be noted that not all makes/models/years of vehicles produced by each manufacturer are present in the PDQ database.
  - The paint from item _____ was consistent with a repaint (or only topcoats). Therefore, no make/model determination could be made.

Literature References

- ASTM E1610: Standard Guide for Forensic Paint Analysis and Comparison.
- Scientific Working Group for Materials Analysis Documents (SWGMAT):
  - "Forensic Paint Analysis and Comparison Guidelines
  - "Standard Guide for Microspectrophotometry and Color Measurement in Forensic Paint Analysis"
  - "Standard Guide for Using Infrared Spectroscopy in Forensic Paint Examinations"
  - "Standard Guide for Using Scanning Electron Microscopy/X-ray Spectrometry in Forensic Paint Examinations"

5.4.5 Hair and Fiber Collection

Scope

Hairs and fibers are collected from evidence for further evaluation and analysis.

Tape lifts used for the collection of hairs and fibers may also be examined by the DNA section. If the tape lifts will be sent to DNA, the hairs should be removed from the tape lifts first. Fiber comparisons should also be conducted before the tape lifts are sent to DNA.

Hairs and fibers may not be of evidentiary value if the victim and suspect are co-habiting. However, it may be necessary to examine some items. Each case should be evaluated based on the circumstances.

Preparation

- Determine which items are from the victim, suspect, scene, etc. Ensure that items from different people, places, etc. are worked in different areas or on different days.
- Review the officer's request for the items to be examined.
    - If gunshot residue is requested on an item, it must be collected BEFORE tape lifts are collected from the item.
        - If the item is from the suspect, contact a Trace Evidence examiner.
        - If the item is from the victim, contact a Firearms examiner.
    - If the item is to be examined by latent prints, contact an examiner in the Latent Prints Section if they need to examine the item first.
- Items which are covered in mold, decomposed bodily fluids, or are otherwise unsuitable for tape lift analysis may be returned untested.

Testing Techniques

Collection from clothing or other items

- Visually examine item and note description of item and fabric content, if listed.
- Take care to preserve evidence that other sections may need to examine (i.e. blood stains, latent prints, etc.). It may be necessary to collect fibers and/or hairs with forceps and place in an envelope or on tape rather than taping the item directly.
- A section of clear adhesive tape is pressed on the item and pulled away. Fibers and/or hairs adhere to the tape which is then placed on a clear transparency sheet. Continue collecting with sections of tape until the entire item has been covered.
- Label the tape lifts on the transparency sheet. It should be noted if tape lifts are collected from a specific area (i.e. inside the underwear).

- Known samples of all the fiber types and colors are cut from the item and placed on the transparency sheet with clear tape or in an envelope. White cotton, denim, light-colored fabrics and smooth fabrics (such as nylon windbreakers) are not suitable target fibers.
- Place transparency sheets and/or envelopes in a manila envelope and itemize.

Collection from Sexual Assault Kits

- Examine the contents of the sexual assault kit to locate the "Pubic Hair Combings" envelope and "Underwear" bag. Also note any extra items that may have been included for hair and fiber examination.
- If samples were not collected according to the information supplied on the package, no further analysis is needed for that item. Record in notes.
- Open the "Pubic Hair Combings" envelope and remove all hairs from the comb, cotton, and/or napkin. Place the hairs in a folded piece of paper or tissue and package in a labeled coin envelope. Return the "Pubic Hair Combings" envelope to the kit.
- Examine the underwear according to the procedures listed above and return to the kit.
- If extra items are included for hair or fiber analysis, retain the entire envelope and contents.
- Place envelopes and/or tape lift transparency sheets in a manila envelope and itemize.

Removing hairs from tape lifts

- A stereomicroscope may be useful for removing hairs.
- It may be necessary to loosen the hair from the adhesive along the length of the hair.
- Gently pick up the end of the hair with tweezers or gloved fingers and remove hair from tape.
- Place the hair in a folded piece of paper or tissue and place in a labeled coin envelope.
- Itemize the package of hairs removed from the tape lift.

Quality Assurance/Quality Control

- Examine items over clean white paper.
- Victim and suspect items should be collected in different rooms.
- Change lab coats, gloves, and other supplies between examination of items from victim and suspect.
- Maintain adhesive tape in a manner to avoid contamination.
- Examine only one item at a time.
- Cut known samples of fiber types and colors from clean areas which do not appear to contain biological stains.

- Follow biohazard procedures and practice universal safety precautions when examining evidence.
- Retaining entire envelopes said to contain hairs will reduce the risk of loss during the transfer of the items to different containers.
- Label all evidence removed from items with a unique item number. For example, "E-2TL" for tape lifts from E-2, "E-2H" for hairs from E-2, "E-2KF" for known fibers from E-2.

Assessment of Evidence Collected

- If known samples are present and/or additional work needs to be completed, forward to the appropriate section or retain the evidence and notify the section chief or analyst who will continue the examination.
- If known samples were not submitted, write a report requesting known samples, if necessary.
- If tape lifts will be sent to DNA for analysis, remove the hairs from those items.

Report Writing Suggestions

- Tape lifts were collected from the items listed. They are being retained.
- Hairs and/or fibers were collected from the items listed. They are being retained.
- If further analysis is desired, the following items are needed: (may request one or more of the following)
    - Head hair sample (40 – 60 pulled hairs) from the victim and any suspects.
    - Pubic hair sample (30 – 40 pulled hairs) from the victim and any suspects.
    - Fiber samples (clothing, carpet, etc.) from any suspects.
    - Oral swabs.

Literature References

- "Trace Evidence Recovery Guidelines", Technical Working Group on Materials Analysis, Evidence Handling Committee.

5.4.6 Hair Examination

Scope

Items may be submitted to be identified as hair. This may be of human, animal, or synthetic fiber (wig) origin.

Human hairs may be examined to assess their suitability for nuclear DNA testing. Hairs not suitable for nuclear DNA testing might be considered for mitochondrial DNA testing. These hairs will be sent to an outside agency for further examination.

If known hairs are submitted, a hair comparison between a questioned hair and known hair samples may be conducted.

Preparation

- If tape lifts have previously been collected by another analyst, the items should be recovered from secure storage and transferred into the possession of the examiner.
- Determine which items are suitable for examination.
- Record the general description of the hair and approximate length.
- Either count the number of hairs present or give a general approximation on the amount of hair present.
- Hairs may be mounted with Permount or another mounting medium for microscopic comparison. If a medium other than Permount is used, it should be noted.
- Hairs should be mounted in such a way to avoid frequent crossovers and bubbles. The ends of the hairs should not stick out from under the coverslip.
- Slides should be labeled with the case number, item number, analyst's initials and date the slide was mounted.

Standards and Controls

- Permount, Meltmount, Cargille liquid, or other mounting media with a stated expiration date will not automatically be discarded after the stated date. As long as the mounting media continues to flow properly, as determined by the examiner, it may continue to be used.
- A collection of known human and animal hairs is kept for reference.

Testing Techniques

Hair Identification

The purpose is to differentiate human hairs from animal hairs or to identify fibers which resemble hairs.

- The item may be examined with a stereomicroscope. Morphological features may easily be distinguished to determine the identification as human, animal, or synthetic fiber in origin.
- The item may be temporarily or permanently mounted on a glass slide for microscopic evaluation.
  - Using a compound microscope, examine the hair.
  - Based upon microscopic observations, determine if the hair is human, animal, or a synthetic fiber. Record the observations used in the determination (e.g. color, banding, scales, medulla, root, etc.)

Evaluation of Root for nuclear DNA

Human hairs are evaluated for the suitability of further testing by the DNA Section. Only hairs which are in the active growth stage (anagen or catagen phase) or hairs with tissue attached may be suitable for nuclear DNA analysis.

- Recovered hairs may be screened using the stereomicroscope to determine if the item is a hair or hair fragment. If tissue is visible, additional microscopic analysis may not be needed.
- Mount the hair for microscopic examination using a compound light microscope.
- If the hair is in the telogen phase with no tissue attached, it is not suitable for nuclear DNA testing.
- If tissue is present on the root or shaft of the hair, the hair may be sent to the DNA Section for further analysis.
- Package the hair for transfer.
  - If the hair was mounted in Permount, remove the hair and clean with xylene.
  - If the hair was in a temporary mounting media, clean the hair.
  - Hairs may be taped to a clean glass slide with the root ends noted or folded in a tissue or piece of paper. Place in an envelope.
  - Itemize the hairs with a unique tracking number.
- Notify the DNA section and transfer the evidence to FD Secure Storage or the analyst working the case.

Hair Comparisons

If a known hair sample has been submitted, a comparison may be conducted between questioned and known hair samples.

- Questioned hairs may be screened with a stereomicroscope before determining which hairs to mount.
- Known head hair samples should consist of fifty to sixty hairs pulled from different areas of the head. These areas include the front, back, top, left side and right side. All hairs from the head of an individual may be placed in one envelope. This standard should represent all types of hair on the head.

- Known pubic hair samples should consist of thirty to forty pulled hairs from different areas of the pubic region.
- A sample of the known hairs representing different colors and lengths of hairs should be mounted for comparison.
- A comparison compound light microscope is used for hair comparisons.
- Enlarged photocopies of the hair slides may be made to help the note taking process. Photocopies of the hair slides, enlarged to 200%, are for documentation purposes not analytical purposes.
- Notes should be made on the microscopic characteristics of the hairs, structures which are visible or absent, and any other outstanding features. Similarities or dissimilarities between the questioned and known samples should be noted.

Other requests

Additional information may be obtained from examining the hairs. Each case should be evaluated to determine if probative information may be gained.

- Forcibly removed
    - Hairs which have been forcibly removed exhibit a ribbon-like appearance of the root.
    - This information may be reported if it is useful in the case.
- Putrid roots
    - Hairs which remain on the body as decomposition takes place form dark bands near the root.
    - This information may be useful in identifying a hair or for determining when a questioned hair was deposited (i.e. post mortem).
- Singed hairs
    - Hairs which have been exposed to extreme heat or flames may exhibit bubbles and a darkened appearance.
    - This information may be reported if it is useful in the case.
- Mitochondrial DNA (mtDNA)
    - Hairs without tissue present may still be suitable for mitochondrial DNA testing.
    - The hair should be at least one centimeter (approximately ½") in length. However, shorter hairs may be attempted.
    - A known hair sample from possible contributors must be submitted.
    - The Arkansas State Crime Laboratory does not conduct mitochondrial DNA analysis. Hairs for mtDNA testing will be submitted to an FBI Regional Laboratory or a private testing company.
        - Prior approval from the FBI must be obtained for submission to the FBI Regional Laboratory. No cost is incurred for this service.
        - If a private company is used for mtDNA analysis, authorization from the Arkansas State Crime Laboratory and the submitting agency must be obtained. The cost of this analysis will not be covered by the ASCL.

Interpretation of Results

- For microscopic hair comparisons:
  - The microscopic features observed in the questioned hair must be observed within the known sample of hairs in order for the hairs to be similar.
  - However, other considerations may be taken into account for physical differences in the hair (e.g. time elapsed from incident to time of collection of known samples, cutting, chemical treatment, etc.). These physical differences may be noted but may not affect the microscopic examination.
  - All hairs which are microscopically similar and have probative value must be examined by a second qualified hair examiner.
- If any of the macroscopic or microscopic features of the questioned hair are different from the hairs within the known hair sample, the hairs cannot be associated.

Report Suggestions

- The techniques used (i.e. stereomicroscopy, light microscopy, comparison light microscopy, polarized light microscopy, etc.) should be reported.

- If identification only:
  - The hairs recovered from Item ____ were of animal origin.
  - The hairs recovered from Item ____ were animal hairs from the family _____.
  - The hairs recovered from Item ____ were synthetic fibers resembling hairs. If further testing is desired, known fibers (wigs or extensions) are needed for comparison.
  - (Head or pubic) hairs were recovered from Item ___.
  - (Head or pubic) hairs indicative of (Caucasian, Negroid, Mongoloid) origin were recovered from Item ____.
    It should be noted that designation of a racial group is based upon an evaluation of the microscopic characteristics present in the hair and may not coincide with the physical appearance of an individual.
  - Body hairs were recovered from Item ____. Body hairs do not usually contain sufficient unique characteristics for comparison purposes.
  - The hairs (or hair fragments) recovered from Item ___ were not suitable for further examination.

- If hairs are microscopically similar:
  - Hairs recovered from Item ___ were microscopically similar to the known hair sample from (person).
  - It is noted that microscopic hair comparisons are not a basis for personal identification.

- If hairs are dissimilar:
    - Hairs recovered from Item ___ were microscopically dissimilar to the known hair sample from (person).

- If known hairs are needed (one or all may be requested):
    - If further analysis is desired, the following items are needed from the victim and any suspects:
        1. Pulled head hair sample (50 – 60 pulled hairs).
        2. Pulled pubic hair sample (30 – 40 pulled hairs).
        3. Oral swabs.

Literature References

- "Forensic Human Hair Examination Guidelines", Scientific Working Group on Materials Analysis.
- Oien, Cary, "Forensic Hair Comparison: Background Information for Interpretation", *Forensic Science Communications*, Vol. 11, No. 2, April 2009.

5.4.7 Fiber Analysis

Scope

Fibers may be identified as being animal, vegetable, or synthetic fibers. Fibers may further be tested to identify the type of fiber.

Questioned fibers can be compared to known fibers to determine if a common origin could exist. The fibers are examined microscopically and instrumentally.

A request may be made to compare not just a fiber but a piece of fabric, rope, or cord to a known sample.

Impressions made by cloth or fabric may also be compared to known items.

See previous section "Collection of Hairs and Fibers"

Preparation

- Known fiber standards are examined using a stereomicroscope. Tape lift sheets from questioned items are examined for any fibers similar in macroscopic appearance to the known fibers.
- Questioned fibers may be circled on the transparent sheet or immediately removed.
- Mount the fibers in a suitable temporary mounting medium. Mount a representative sample of the known fibers using the same mounting medium.
- After analysis is completed, fibers should be mounted for permanent storage in Permount or Meltmount.

Testing Techniques

Fiber Identification

The purpose of fiber identification is to identify fibers as being of animal, vegetable, or synthetic origin. Further testing may be performed to determine the specific fiber type.

- Macroscopic qualities of the sample should be noted. These may include amount of sample, texture, color, etc. If the information required for the identification is sufficient after macroscopic examination, it is not necessary to continue.
- Microscopic examination of the sample may be required. The mounted sample should be examined with the polarizing light microscope (PLM).
- For identification of the type of synthetic fiber, optical properties observed by PLM should be recorded.
    - Microscopic color
    - Pleochroism
    - Diameter range

    - Delusterants
    - Cross-section (optical)
    - Relative refractive indices
    - Birefringence (relative)
    - Sign of elongation
- After the PLM examination, the fiber may be identified. If the sample can be sufficiently identified after microscopic examination, it is not necessary to continue.
- If further confirmation is desired, the fiber may be analyzed using FTIR and/or micro-chemical tests.

Fiber Comparisons

The purpose of a fiber comparison is to compare questioned fibers to a known sample of fibers.

- The initial step in fiber comparisons is to identify the fibers following the procedure above.
- In addition, the questioned and known fibers are compared side-by-side by PLM.
- If any of the observed properties between the questioned and known fibers are different, it is not necessary to continue the analysis.
- If the samples are similar, the following testing may be performed.

Microspectrophotometry (MSP)

If the fibers have sufficient color, an instrumental comparison of the color of the questioned and known fibers will be performed using a microspectrophotometer.

- Fibers may be tested using the mounted samples.
- Absorbance or transmission spectra of the fibers are obtained.
- The range of colors and color intensity should be considered when determining which fibers to test.
- Comparison should be performed by overlaying the known and questioned spectra.
    - Any major discrepancies between the questioned and known spectra are reason for elimination.
    - If the general shape and the wavelength accuracy (+/- 5 nm) between the questioned and known samples are similar then the fibers could have originated from the same source.
- The mean of the known and questioned spectra may be included in the case file.
- The first derivatives of spectra may be compared to show any differences in the slope.
- All of the spectra will be stored on the hard drive of the instrument under the case number.

FTIR

This technique may be used to further characterize synthetic fibers.

- Fibers or a portion of a fiber may be flattened by rolling the fibers and placing them on a KBr window or by using a compression cell.
- The spectra of questioned and known fibers are compared.
  - If the general shape and wavenumber accuracy (+/- 8 nm) between the questioned and known samples are similar, then the fibers could have originated from the same source.
  - Any major discrepancies between the questioned and known spectra are reason for elimination.
- Samples which need interpretation (e.g. subgeneric class determination) will be compared to spectral libraries present in the software.
- Spectra used for comparison or to determine fiber class will be included in the case file. All of the spectra will be stored on the hard drive of the instrument under the case number.

Cross-sectioning

- If enough sample is present, the examiner may cut an actual cross-section of the fibers.
- A cross-section may be cut with a scalpel or by placing the fiber between two sheets of a polyolefin material.
  - The polyolefin is placed between two glass slides and heated on a hot plate. Pressure is applied with the eraser of a pencil until the polyolefin melts together to take on a translucent appearance.
  - Cool the slides and then slice thin cross-sections of the embedded fiber.
  - Mount the cross-sections in a suitable mounting medium for microscopic examination.
  - A sketch or photograph of the cross-section may be helpful.
- The modification ratio of non-circular fibers may be determined from the cross-section.
  - Ratio in size between the outer diameter of the fiber and the diameter of the core.

Solubility/Microchemical Tests

Solubility and microchemical tests are destructive in nature. They should be performed for further discrimination of some fiber types after completing other testing. They should only be performed when enough sample is present that the entire sample is not consumed.

- Nylon 6, 66
- Acetate, triacetate
- Other solubility tests

Fabrics and Cordage

A request may be made to compare not just a fiber but a piece of fabric, rope, or cord to a known sample.

- Examine the general appearance of the questioned and known samples.
  - Note and collect any trace evidence on the evidence (stains, hairs, fibers, etc.).
  - Examine the questioned and known pieces for the possibility of a fracture match.
- Examine the sample to determine the physical construction properties. These may include but are not limited to the following:
  - Construction (woven, knit, non-woven)
  - Yarns per inch in warp and weft direction
  - Yarn twist (S or Z)
  - Color of fabric, yarns, fibers, or backing
  - Number and twist of plies
  - Number of filaments in each ply
  - Carpet pile (loops, cut, etc.)
  - Coatings
  - Sewing threads, buttons, decorations, etc.
- Additional microscopic and instrumental testing may be performed on the fibers.

Fabric Impression

Impressions of clothing or other fabric may be left on items of evidence. These may be compared to a known source.

- Preliminary examination of the impression
  - May be of actual impression or a reproduction (photo, lift, cast)
  - Examine actual impression for transfer of fibers. If fibers are found, carefully remove and analyze according to procedures above.
  - Examine the shape of the impression.
    - Glove impression
    - Finger marks, thumb, palm
    - Fabric, leather, etc.
  - Clothing impressions
    - Identify as to whether woven, knitted, etc.
    - Unusual marks, seams, buttons, etc.
- Select a portion of the known material and produce an impression for comparison to the questioned impression.
  - Use different amounts of force or pressure, or ink, clay, etc.
- Perform actual comparison
  - Count the number of "ribs" per inch in each direction.
  - Count the number of yarns or loops per inch in each direction.
  - The general pattern of the impressions must be identical to be a match.

Sampling vs. sample selection

- If it is reasonable to assume homogeneity of the sample (such as a thread or piece of cloth), the report will state conclusions about the whole based upon the testing of a representative sample.
- If it is not reasonable to assume homogeneity of the sample (such as recovered fibers), the report will state conclusions based upon the portion tested.

Interpretation of Results

- For a positive association in a fiber comparison:
  - The questioned fiber should be consistent with the known fibers in microscopic properties observed by polarized light microscopy.
  - The questioned fiber should be consistent with the known fiber in optical properties.
  - The questioned fiber should be consistent with the known fiber in chemical properties (as observed by FTIR or microchemical testing).
- If any of the physical, microscopic, optical, or chemical properties are not consistent, the questioned fiber cannot be associated with the known fiber sample.

Report Suggestions

- The techniques used in testing should be reported.

- If the sample was homogeneous (e.g. cloth or thread) and a positive association:
  - Item ___ and ____ were consistent in color and overall construction and were composed of fibers which were consistent in physical, chemical, and optical properties. It was concluded that the Item ____ piece of fabric could have originated from the Item ____ or another source composed of fibers with the same physical, chemical, and optical properties.

- If the sample was not homogeneous and a portion of the sample was analyzed:
  - Based upon the fibers analyzed, (color and type) fibers recovered from Item ____ were consistent in physical, chemical, and optical properties with the fibers used in the construction of Item ____. It was concluded that these (color type) fibers from Item ___ could have originated from the (known) Item ____ or another source of fibers composed of the same physical, chemical, and optical properties.
  - Based upon the fibers analyzed, the Item _____ fibers could not be associated with the Item _____ fibers due to differences in _____ (state color, microscopic properties, optical properties, chemical properties and/or fluorescence as the reason for the differences).

- If known samples are needed for comparison:
    - (Color and type) fibers were recovered from Item ___. If further testing is desired, known fiber samples (clothing, carpet, etc.) are needed for comparison.

Literature References

- "Forensic Fiber Examination Guidelines", Scientific Working Group on Material Analysis, Fiber Subgroup.
- ASTM E2228 Standard Guide for Microscopic Examination of Textile Fibers
- ASTM E2225 Standard Guide for Forensic Examination of Fabrics and Cordage
- ASTM E2224 Standard Guide for Forensic Analysis of Fibers by Infrared Spectroscopy

5.4.8 Fracture Match

Scope

A comparison of fractured edges of materials may show that they physically fit together. This examination provides conclusive evidence that the materials were at one time joined.

Fracture matches may be conducted on tape, glass, paint, plastic, or other items which have been broken, torn, or cut creating a unique edge.

Quality Assurance/Quality Control

- Review the list of other sections to examine items. Ensure that the integrity of other evidence is not compromised in the analysis (i.e. fingerprints, blood, etc.).

Testing Techniques

- The possibility of a fracture match should be the first consideration when examining evidence.
- Visually examine the evidence. Photograph or document the questioned and known items.
- If an item contains multiple pieces, it should be put back in to the largest piece possible. Items originating from the same area may be combined.
- Place the questioned and known samples in close proximity to ensure that they have a unique edge in common.
- Document the match.

Interpretation of Results

- Determine if the fractured pieces match together in such a way that the match is unique.
- If the edges or ends of a piece of evidence have been damaged or altered, it may not be possible to conduct a physical match. Further testing should be performed according to the type of evidence.

Reporting Suggestions

- The technique used in the analysis will be listed on the report.
  - Items _____ and _____ were examined visually.
  - Items _____ and _____ were examined visually and using stereomicroscopy.

Positive association:

- The fractured (end, piece, cord, etc.) from the questioned item matched the fracture on the known item (i.e. roll of duct tape, broken vase, cord, etc.). These items were at one time joined.

Additional testing performed if positive association not made but items are similar:

- Items _____ and _____ could not be physically matched together. Items _____ and _____ were consistent in physical and chemical properties and could have had a common origin.
    - Documentation would follow the sub-discipline of the type of analysis performed.

Negative association:

- Item _____ and _____ could not be associated due to differences in (color, width, thickness, construction, etc.).

Literature References

- Bradley, M. J., Keagy, R. L., Lowe, P. C., Rickenbach, M. P., Wright, D. M., and LeBeau, M. A. A validation study for duct tape end matches, *Journal of Forensic Sciences* (2006) 51:504–508.

5.4.9 Tapes and Adhesives

Scope

Tapes may be submitted for comparison to a known sample.

A macroscopic examination of the tape is conducted. If appropriate, a fracture match comparison may be conducted.

The physical and chemical properties of the tape may be used for characterization.

The size and condition of the evidence may affect the ability to compare all properties. It may be necessary to detach tape that is folded or waded.

This procedure may be applied to other similar types of evidence.

Macroscopic and Microscopic Examination

The tape is examined for the possibility of a physical match as well as potential trace evidence (hairs and fibers) or for further analysis by other sections of the laboratory (latent prints, transfer DNA).

If a potential fracture match exists, see the Fracture Match section of this manual.

Physical Properties

The following information should be recorded:

- Condition of tape ends (i.e. unique tear, cut, serrations, etc.)
- Width of tape
- Backing
    - Color
    - Texture/Marks
- Adhesive color
- Thickness
    - Overall (backing and adhesive)
    - Backing only (if adhesive is removed)
- Fiber reinforcement (Scrim)
    - Weave pattern
    - Number of yarns per inch in machine direction (warp)
    - Number of yarns per inch in cross direction (weft)

If differences are observed in physical properties between a questioned and known sample, no further testing is necessary.

Additional Testing

- Tape may be manipulated or dissected using a scalpel blade to thoroughly characterize the physical properties.
- A solvent may be used to separate the adhesive from the backing or fiber reinforcement. (Hexane for duct tape, methanol for black electrical tape.)

- Following the removal of adhesive, the duct tape backing should be cross-sectioned to determine layer structure.
- Transparent tape backings should be examined by cross polarized light (using PLM). Record observations.
- Fiber analysis should be conducted in accordance with the procedures in this manual.
- Ultraviolet or an alternate light source may be used to examine the tape. Record observations.
- The offset of the fibers in the machine direction from the edges of the tape should be noted.
- If no differences are observed in physical properties, instrumental examinations should be performed.

Instrumental Analysis

FTIR

- Spectra may be obtained from the layers composing the backing and the adhesive.
  - The UATR or a compression cell with the microscope may be used when examining polymers and adhesives.
- The spectra for the known and questioned samples are compared.
- If any significant differences are observed in the spectra, no further analysis is necessary.

SEM/EDS

- If known and questioned tape backings and/or adhesives are generally in good condition, they may be tested for elemental composition.
- Samples are mounted with carbon adhesive to an aluminum stub.
- Samples are placed in the chamber and analyzed by SEM/EDS for the elemental composition.
  - Comparison of the elements present is by a qualitative basis.

Interpretation and Report Suggestions

- Techniques used in the analysis will be identified in the body of the report.
- If the known and questioned tapes can be eliminated based upon any of the testing:
  - Item __ could not be associated with item __ due to differences in ____ (e.g. color, physical properties, chemical properties, etc.)
- If the known and questioned tapes cannot be eliminated based upon any of the testing:
  - The tape (item __) could not be physically matched to the end of the roll of tape (item __). The questioned tape was consistent with the known roll of tape in overall construction and in physical and chemical properties. The questioned tape could have originated from the known tape sample or another source of tape manufactured in the same manner.

Literature References

- Scientific Working Group for Materials Analysis Documents (SWGMAT):
    - "Guideline for the Forensic Examination of Pressure-Sensitive Tapes"
    - "Guideline for Assessing Physical Characteristics in Forensic Tape Examinations"
    - "Guideline for Using Light Microscopy in Forensic Examinations of Tape Components"
    - "Guideline for Using Fourier Transform Infrared Spectroscopy in Forensic Tape Examinations"
    - "Guideline for Using Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy in Forensic Tape Examinations"

5.4.10 Lamp Filament Examination

Scope

The primary purpose of a lamp filament analysis is to determine whether the lamp was incandescent ("on") or not incandescent ("off") at the moment of impact. Characteristic deformations or fractures of the filament may be produced from hot or cold filaments during an impact. Further evidence may arise if the glass bulb is broken during the accident.

Information provided by the investigating officer may be useful in the examination. Accident reports, photographs, or diagrams of the vehicle may be helpful. The position and location of all the bulbs is also useful information for the examiner.

Any lamp having a tungsten filament may be analyzed. LED lights are not suitable for lamp filament examination.

Safety Considerations

- Broken bulbs have very sharp edges. Caution should be taken with broken glass or sharp metal.
- Use caution when examining lamps and testing circuits.

Minimum Standards and Controls

- Known lamp standards may be used for comparisons. They may be on file or be purchased from any retail distributor.

Testing Techniques

Multiple testing techniques are available to determine deformation, discoloration, deposition and pitting of the filament. The analyst will determine which tests are appropriate for the particular case.

General Examination

- Notes should be made on the type of lamp, brand, base arrangement, bulb shape, voltage/wattage, and filament configuration and purpose in vehicle (if possible).
- Document the physical appearance of the lamp. Photographs may be taken.
- Note the following (stereomicroscopic examination may be useful)
  - Physical condition of the glass envelope (intact, sealed, breakage, discoloration)
  - Physical condition of the terminal posts (distortion, fractures, deposits)
  - Physical appearance of the filament (distortion, stretching, breakage, etc.)
    - Presence or absence of any discoloration of the filament or oxide deposits

        - Presence or absence of age sag, pitting of the filament
        - Presence of cold fracture or molten ball
        - Presence of melted glass (if envelope is broken)
    - Any other unusual observations

Polarizing Light Microscope

The purpose of the polarizing light microscope examination is to identify small particles that may be adhering to the filament but have not been fused. This test differentiates between particles of sand (quartz) and glass.

- Carefully remove questioned particles and mount on a microscope slide in a drop of Cargille refractive index liquid.
- Examine the particle under cross-polarized light.
    - Glass particles are isotropic and will remain dark with the rotation of the circular stage.
    - Quartz particles are anisotropic and will exhibit birefringence.

Scanning Electron Microscope

The advantage of the scanning electron microscope in the examination of lamp filaments is its great depth of focus over the range of magnifications used. In addition, the SEM is useful for examining filaments for signs of age and fatigue as well as a more careful study of its fractured ends.

- The filament piece is carefully mounted on a stub with carbon-coated adhesive tape.
- The sample is placed in the SEM chamber and examined at low to medium magnifications.
    - Aged filaments show a pitted surface and lack the delineations produced by the extrusion process.
    - Cold fractures will show a rough break.
    - Hot failures will appear bulbous and smooth.
    - Glass particles may fuse to the filament when the envelope is broken and the filament it hot.

Continuity Test

The purpose of the continuity test is to determine whether the lamp is still operational and may be conducted on lamps that still have the outer glass envelope, filament, and terminal posts intact. The continuity test should be conducted before opening any lamps.

- A multimeter may be used to verify each filament circuit by testing the continuity through each circuit. Set the multimeter to the sound wave icon.
    - In the case of small bulbs, one connection should be made at one of the contact points on the bottom of the bulb and the other connection made with the copper shell at the base of the bulb.
    - In the case of a single beam headlamp, the connections should be made at each of the two lugs on the back of the lamp.
    - In the case of a dual beam headlamp, there are three lugs in a triangular shape on the back of the lamp. Ensure that the connection is made between the lug for the proper beam and the ground lug.

- A beep indicates an intact circuit (and filament).
- No beep and a display of OL (Open Loop) indicates a broken circuit and/or filament.
- It may be useful to use known lamps for comparison to the readings from the evidence lamps.

Opening glass envelope

If the filament cannot be properly observed through the glass envelope, it may be necessary to open the lamp. Caution should be used when breaking glass. All continuity tests should be performed before opening the lamp.

- Wear protective eye covering.
- For small lamps, carefully scribe the glass, wrap in plastic wrap, and use a vice to separate the base from the bulb.
- For intact sealed-beam headlamps, the outer glass envelope will have to be broken to examine the filaments.
    - The lamp is placed on a table, lens side down, and a circle is drawn with a suitable marker around the lamp surface approximately equidistant from the top and bottom edge of the lamp.
    - A diamond-tipped marker is used to scribe over the circle made by the ink marker. The vacuum inside the lamp should then be broken by crushing the glass tit located on the back of the lamp inside the triangle formed by the terminal lugs with a pair of pliers.
    - A portable propane burner or charcoal igniter is used to heat the area outlined by the ink marker until all the color from the marker has disappeared. The lamp is quickly immersed in a pan of water, lens side first, utilizing a pair of pliers to hold one of the terminal lugs or a cold wet towel is thrown over the lamp. The lamp should fracture along the scribe marks.
    - The portion of the lamp containing the terminal posts and filament(s) should be set on a table and allowed to cool. The other section after cooling should be checked for any loose parts from the inside of the lamp and retained for inclusion upon completion of the analysis.

Interpretation of Results

- The filament was incandescent (on) at the time of impact (glass envelope broken)
    - Physical characteristics would include an oxidized filament (discolored), possibly with beaded ends, some degree of filament distortion, possibly molten glass on the filament, possibly white tungsten oxides present on the inner surface of the bulb and/or on the colder surfaces within the bulb (posts or other filament).
- The filament was incandescent (on) at the time of impact (glass envelope NOT broken)
    - Physical characteristics include some significant degree of distortion and/or stretching of one or both filaments.

- The filament was not incandescent (off) at the time of impact
    - The filament has a cold fracture. The absence of oxidation, melted glass, or filament distortion cannot definitively determine that the filament was "off" when the glass is broken. It may indicate that the bulb was not incandescent at the time the glass was broken.
    - If the filament had previously burned out and the glass envelope is then broken because of an impact, the lamp would show no sign of oxidation.
- Insufficient characteristics to determine the incandescence at the time of impact
    - The lamp appears normal.
    - There is insufficient distortion to make a determination.
    - There is not enough left of the filament or supports to show signs of incandescence.

Report Suggestions

- The techniques used for the analysis will be reported.
- The type of bulb (single or dual filament) will be reported.
- The filament was incandescent:
    - Examination of the (single/dual) filament lamp revealed characteristics consistent with the (upper/lower/both) filament being incandescent (on) at the time of an impact.

The filament was not incandescent:

- Examination of the (single/dual) filament lamp revealed characteristics consistent with the (upper/lower/both) filament not being incandescent (off) at the time of an impact.

- Insufficient characteristics:
    - Examination of the (single/dual) filament lamp revealed it to be normal. Therefore, it is not possible to determine the incandescence of the lamp at the time of an impact.
    - Examination of the (single/dual) filament lamp revealed insufficient characteristics to determine the incandescence of the lamp at the time of an impact.

Literature References

- Baker, J. Stennard "Lamp Examination for On and Off in Traffic Accidents." Topic 823 of the Traffic Investigation Manual", The Traffic Institute, Northwestern University Evanston Illinois 60204, 1986.

5.4.11 Indented Writing

Scope

This procedure is for the interpretation of indented writing through lighting techniques or enhancement by the use of an electrostatic deposition apparatus.

Preparation and Sampling Techniques

- The item should be carefully removed from the packaging
- The outer packaging of the evidence should be marked after the removal of the evidence so indented writing is not added by the analyst.
- Documents will not be folded or otherwise physically altered unless necessary to complete an examination. Permission must be obtained from the submitting agency prior to a necessary alteration, such as removing a page from a notebook.

Testing Techniques

The analyst will determine which tests are appropriate for the particular case.

Oblique Lighting

- Examine the evidence from all angles using side lighting.
- Use magnifiers or stereomicroscope, if needed.
- If impressions are found, photograph using side lighting.

Electrostatic Deposition Apparatus (ESDA)

Conduct ESDA examination of documents that are suspected to have indented writing, even if nothing is noted with oblique lighting.

- Before examination with the ESDA, the document will be put in the humidifier for 2 minutes. This assures that from day to day the papers tested are at the same humidity.
- The corona shall be passed over the document a minimum of 3 times.
- Create a permanent record by preserving the results with adhesive-backed clear acetate sheets. These results will be scanned into the case file. The ESDA lift will be considered evidence that will be itemized and retained or returned to the agency.

QA/QC

- A small strip of paper will be folded lengthwise and the case number, item number, date, and analyst initials will be written on it using a ballpoint pen. This piece of paper will be unfolded and placed blank side up on the vacuum bed to ensure that the ESDA is functioning properly. The test strip will be documented by scanning in the case file.

Documentation

- The document should be photographed, photocopied, or scanned prior to examination to record its original appearance.
- Pictures of indentions viewed with oblique lighting will be scanned into the case file.
- ESDA lifts will be scanned into the case file.

Interpretation of Results and Reporting suggestions

- The method used for examination should be reported.
    - The letter (Item _____) was examined by oblique lighting and use of an Electrostatic Deposition Apparatus (ESDA).

- Deciphered text which is relevant to the investigation will be reported. Words or parts of words which cannot be clearly deciphered will not be reported but may be left as blanks or questioned (?) within other deciphered material.
    - Examination of the letter (Item _____) revealed indented writing and was deciphered to read: “My name is John” and “555-1212”. Nothing more was decipherable.

- If nothing is visible:
    - Examination of the letter (Item _____) did not reveal any indented writing of evidentiary value.

Literature Reference

- ASTM E2291 Standard Guide for Indention Examinations

5.4.12 General Chemical Analysis

Scope

Many samples are sent in which may require chemical analysis. Some of these requests may fall under a category that has previously been performed. Other samples may require the analyst to rely on their training as a chemist to develop a testing procedure. No procedure manual could encompass methods for every general chemical type analyzed.

Good scientific principles and a logical analysis scheme are applied to those evidence types that have not been encountered before.

Analytical Approach

- Each sample requires special consideration based on the type and amount of sample present.
- A macroscopic examination and notes on the evidence will be taken.
- The examiner will determine the value of the samples and the method most suitable for analysis.
- It may not be possible to fully identify an unknown sample. However, general classification may be possible.
- When comparison samples are submitted, it may be possible to state that the unknown sample shared the same physical and/or chemical properties with the known sample.

Minimum Standards and Controls

- These are dependent on the type of evidence being submitted and the examinations that are performed.
- Generally, reference standards, controls, and blanks are run along with the evidence items and the results are placed in the case file.
- All documentation to support the conclusion should be documented in the case file.
- Instrument procedures should be followed as detailed in other sections of this manual.

Testing Techniques

The examination of the samples may include but is not limited to:

- Stereomicroscopic examination
- Polarized light microscopy
- Comparison polarized light microscopy
- Microspectrophotometry
- SEM/EDS
- GC/MS
- FTIR/UATR
- Microchemical or solubility testing

Some of the general chemical methods which have been developed include:

5.4.12.1 Acids and Bases

This method is used for identify common acids and bases that may be encountered in casework.

Safety Considerations

- Acids and bases may be very corrosive. Eye and skin protection must be used.
- Acids may be very reactive with certain compounds. Extreme care must be taken when mixing acids.
- Sulfuric acid reacts with bases in a violent manner.
- Ammonium nitrate is explosive when mixed with organic solvents and an ignition source. Care should be taken when mixing and disposing of ammonium salts.

Minimum Standards and Controls

- Treat the questioned, known, and/or control samples in the same manner.
- Controls will be run with each case when performing microchemical tests. The results should be recorded in the reagent logbook and/or the case notes.

Analytical procedure/Interpretation of Results

- If the sample is a solid or on a solid substrate, dissolve a portion of the sample in deionized water.
- Check the pH with pH test strips.
- If the pH is acidic:
    - Silver Nitrate: Add a drop of dilute Nitric Acid to acidify sample and remove possible interfering ions. Test sample with a few drops of silver nitrate reagent.
        - Hydrochloric acid→A white precipitate indicates the presence of hydrochloric acid.
        - Hydriodic acid→A yellow precipitate indicates the presence of hydriodic acid.
        - Hydrobromic acid→A pale yellow precipitate indicates the presence of hydrobromic acid.
        - Hypophosphorous acid→A black precipitate indicates the presence of hypophosphorous acid.
        - Sulfuric, phosphoric, nitric, and phosphorous acids will not produce a precipitate.
    - Addition of Concentrated Ammonium Hydroxide after silver nitrate test:
        - Hydrochloric acid→ The white precipitate (silver chloride) dissolves.
        - Hydriodic acid→The yellow precipitate (silver iodide) turns milky white and does not dissolve.

        - Hydrobromic acid→The pale yellow precipitate (silver bromide) dissolves.
        - Hypophosphorous acid→The black precipitate (metallic silver) remains unchanged.

    - Barium Nitrate: Test sample with a few drops of barium nitrate reagent.
        - Sulfuric acid→A white precipitate forms under acidic and basic conditions.
        - Phosphoric acid→Does not form a precipitate under acidic conditions. Forms a white precipitate under basic conditions.
        - Hydrochloric, hydriodic, hydrobromic, hypophosphorous, nitric, and phosphorous acids will not produce a precipitate.

    - Positive identification of the acids is determined by reacting the acids with ammonium hydroxide to form ammonium salts and identify by FTIR.
        - Add a few drops of the sample to approximately 0.5 mL of ammonium hydroxide then add approximately 40 mL of acetone.
        - Filter the precipitate and dry.
        - Identify the ammonium salt by Fourier transform infrared spectroscopy.

- If the pH is basic:
    - Ammonium hydroxide→Test the sample with Nessler's reagent. (Obtain Nessler's reagent from Drug Section.) The formation of an orange to brown precipitate indicates the presence of ammonium ions.
    - Sodium or potassium hydroxide→If the pH is greater than 11, differentiate by SEM-EDS.

Reporting Suggestions

- Techniques used in the analysis will be identified in the body of the report.
- If the pH is neither acidic or basic:
    - No acids or bases were identified in item ____.
- If the results of the pH test and/or spot tests were sufficient for the case:
    - The liquid in item ___ was indicative of (name of acid or base).
- If the acid or base was identified:
    - Item ___ was an acidic/basic solution that contained (name of acid or base).
    - Item ___ was an acidic/basic solution consistent with (name of acid or base).

- If appropriate, common sources may be included:
    - Hydrochloric acid, commonly found as muriatic acid…
    - Sulfuric acid, commonly found as battery acid…

Literature References

- Oulton, S. and Skinner, H., "Identification of Common Inorganic Acids Encountered at Clandestine Laboratories," *Journal of the Clandestine Laboratory Investigating Chemists Association,* Vol. 8, 1993, pp. 17-25.
- Feigl, F., and Anger, V., Spot Tests in Inorganic Analysis, 6th ed., Elsevier Publishing Company: Amersterdam, The Netherlands, 1972. (Book on shelf in Trace Library)
- Chamot, E. and Mason, C., *Handbook of Chemical Microscopy*, Vol. II, McCrone Research Institute, Chicago, IL, 1989. (Book on shelf in Trace Library)

5.4.12.2 Bank Dyes, Pepper Sprays, and Tear Gases

The most common exploding bank dye packs encountered in casework have contained the red dye 1-methylaminoanthraquinone (MAAQ) sometimes in the presence of the lachrymator o-chlorobenzylidenemalononitrile (CS). The red dye, MAAQ, may also be found in devices containing red smoke mixtures. The procedure describes a means of extracting and identifying these compounds.

Tear gas products (lachrymators) may be encountered in casework as defense sprays. They may contain 2-chloroacetophenone (CN), o-chlorobenzylidenemalononitrile (CS) and/or oleoresin capsicum (OC) which contains the active component capsaicin. The procedure described may also be used for extracting and identifying these compounds.

Safety considerations

- MAAQ is a dye which is not water soluble.
- Lachrymators are compounds which may irritate the eyes and mucous membranes and should be handled with caution.
- When working with spray products or highly concentrated samples, analysis should be performed in a fume hood.

Minimum Standards and Controls

- Questioned and known samples and control or reference samples should be examined in the same manner.
- When performing extractions, a blank of the extraction liquid and an unstained portion of the substrate should also be analyzed, when possible.

Analytical Procedure

- If a known sample of a spray product is submitted, spray onto a clean white cloth for examination and testing.
- Visually examine the evidence for any stains.
    - Red, red-orange, or pink stains from bank dyes.
    - Pepper sprays may come in a variety of colors.
- Examine the evidence using ultraviolet light or an alternative light source, if needed.
    - Several brands of pepper sprays contain an ultraviolet dye.
- Cut out a small portion containing the stain of interest and extract with a minimum amount of methylene chloride.
    - If the area of interest cannot be cut out, rub a cotton-tipped swab dampened with methylene chloride over the stained area.
    - If the stained area is dark and powdery, scrape the material into a sample vial and add the solvent.
- If no color is observed on the evidence, no further testing is necessary.
- If stained samples do not extract into the methylene chloride, no further testing is necessary.

- Use the same approximate size of unstained material, if available, for the substrate control or analyze the extraction solvent or unstained swabs.
- If necessary, concentrate the extract. Concentrate the blank in the same manner.
- Analyze the extract using GC/MS.
    - Attempt to identify both the dye and the lachrymator (when present) in cases involving bank dyes.
    - Attempt to identify the lachrymator (if present) and the capsaicin in pepper sprays.

Interpretation of Results and Report Suggestions

- Techniques used in the analysis should be identified in the report.
- Bank dye
    - If the stains are not soluble in methylene chloride:
        - The red stains on Item ____ could not be associated with the red dye from the known dye pack due to differences in physical properties.
    - If CS and/or MAAQ are identified:
        - The stains from Item ___ contained 1-methylaminoanthraquinone, a red dye and o-chlorobenzylidenemalononitrile, a lachrymator.
        - It should be noted that these materials are commonly found in exploding bank dye packs.
    - If CS and/or MAAQ are not identified:
        - No 1-methylaminoanthraquinone, a red dye commonly found in exploding bank dye packs, or o-chlorobenzylidenemalononitrile, a lachrymator, was identified on Item _____.
- Pepper Sprays or Tear Gas
    - If pepper spray and/or tear gas was identified:
        - Item ___ contained _____. This is consistent with the labeling on the capsicum-based pepper spray container.
        - Capsaicin (and dihydrocapsaicin) was identified on the stains from Item ___. This (These) is a common component of capsicum-based pepper sprays.
        - O-chlorobenzylidenemalononitrile (CS) and/or 2-chloroacetophenone (CN), lachrymators, were identified in the stains from ___.
    - If no pepper spray or tear gas was identified:
        - No (names of pepper spray or tear gases) were identified on Item _____.

Literature References

- Reilly, C., Crouch, D., and Yost, G., "Quantitative Analysis of Capsaicinoids in Fresh Peppers, Oleoresin Capsicum and Pepper Spray Products", *Journal of Forensic Sciences,* Vol. 46, No. 3, 2001, pp. 502-509.
- Reynolds, P., "Analysis of Bank Dye Evidence and the Challenges of Daubert Hearings", *Forensic Science Communications,* Vol. 10, No. 1, Jan. 2008, pp. 1-5.

5.4.12.3 Sugars

Common sugars may be encountered in case work. Sugar may be added to the fuel supply or oil of motor vehicles. Because the solubility of sugar in gasoline is so low (1.5 mg/L), a sampling of solid residues from the fuel tank or filters may also prove helpful.

Safety Considerations

- Fuels may have strong odors. Work should be performed in a vent hood.

Minimum Standards and Controls

- Treat the questioned sample and the control or standard samples in the same manner.
- When performing extractions, also analyze a blank of the extraction liquid and an undisturbed portion of the substrate, if possible.

Testing Technique

- Examine the sample for any obvious crystals present and to determine if the sample is homogeneous. If crystals are observed, remove them and perform solubility tests.
- If solid material is present, it may be necessary to wash away residue left on the solid material (i.e. gasoline or oil). Sugar is not soluble in hexane. Hexane, pentane, or another suitable solvent may be used.
- If there is no solid material present, perform a water extraction of the sample containing the suspected sugar.
    - Rinse with distilled water. If there are sugars present they should be present in the water layer.
    - If the sample is very thick or dirty, it may be necessary to thin the sample with hexane.
    - Additional hexane washes of the water layer may be necessary if the sample is still dirty.
- Perform the anthrone test for carbohydrates. Examine sugar as a positive control.
    - Anthrone Test
        - Place a few mL of the water extract in a test tube.
        - Point tube away and slowly trickle the anthrone reagent down the side of the tube.
        - Reagent passes through the water layer. If carbohydrates are present, a green ring will appear on top of the reagent below the interface with the water.
        - Alternatively, test could be performed on a micro scale.
- Evaporate some of the sample from the water extraction to dryness.
- Identify crystals by FTIR.

Interpretation of Results and Report Suggestions

- Techniques used in the analysis shall be identified in the body of the report.

- If the sample does not show a green ring with the anthrone test:
    - No sugars were identified in the item __ extract.
- If the solid material or the extracted residues are identified as sugar:
    - The item __ white crystalline solid was identified as _____(name of sugar).
    - The item __ extract contained _____ (name of sugar).
- If appropriate, common sources of the sugar may be named:
    - The item __ white crystalline solid was identified as sucrose, commonly referred to as table sugar.

Literature References

- Inman, K., Hardin, G., Sensabaugh, G. and Thornton, J., "Concerning the Solubility of Sugar in Gasoline", *Journal of Forensic Sciences*, Vol. 38, No. 4, July 1993, p. 757.
- Lentini, J., "A Discussion of 'Concerning the Solubility of Sugar in Gasoline'", *Journal of Forensic Sciences*, Vol. 39, No. 2, March 1994, pp. 303-304.

5.4.12.4 Gunpowder flakes

Particles may be submitted for identification as gunpowder flakes. The medical examiner may send samples from a victim for identification of discharged gunpowder flakes. The flakes are identified by their morphology and microchemical testing.

Safety Considerations

- The acid used in microchemical testing is harmful and corrosive. Use proper eyewear, gloves, and lab coat.
- Wash spot plates immediately after use.

Minimum Standards and Controls

- The diphenylamine solution should be tested on a known sample of gunpowder flake before being used in casework.
- Documentation of the testing is recorded in the reagent logbook.

Testing Technique

- Examine the sample using a stereomicroscope and document the number (or relative abundance) of particles, color, and shape (i.e. round, flattened ball, irregular, rods, etc.).
- Chose a representative flake (or more than one if different colors or shapes are observed) and place in the well of a spot plate.
- Perform the diphenylamine solution test for the presence of nitrates/nitrites. Nitrates present in gunpowder particles oxidize diphenylamine in a sulfuric acid solution to produce a blue color reaction.
    - Examine a known gunpowder flake as a positive control.
    - While observing under a stereomicroscope, place one drop of acetone on the particle. Gunpowder flakes will begin to swell.
    - Place a drop of diphenylamine solution in the well. Nitrates or nitrites will turn bright blue.
- Finer particles may be tested for the presence of lead by the Potassium Iodide Test.
    - Place a small amount of sample on a glass slide.
    - Add one drop of 10% nitric acid and cover with a coverslip.
    - Add one drop of potassium iodide on the slide near but not touching the coverslip.
    - Place slide on microscope stage.
    - Drag the drop of potassium iodide to the coverslip with a glass rod.
    - Observe the reaction. The formation of yellow hexagons is a positive identification of the presence of lead.

Interpretation of Results and Report Suggestions

- Techniques used in the analysis shall be identified in the body of the report.

- If the sample has the color and shape typical of gunpowder flakes and tests positive with the diphenylamine solution test:
    - (Number) discharged gunpowder flakes were identified in the sample from ____.
- If lead residues were identified, they may be added to the information.
    - Lead residues were identified on item ____.
- If the sample is negative with the diphenylamine solution test OR if the particles do not have the proper morphology:
    - No discharged gunpowder flakes were identified on item __.
    - No discharged gunpowder flakes or lead residues were identified on item ____.

Literature References

- Feigl, F., and Anger, V., Spot Tests in Inorganic Analysis, 6th ed., Elsevier Publishing Company: Amersterdam, The Netherlands, 1972, pp. 359-361. (Book on shelf in Trace Library)
- Chamot, E. and Mason, C., *Handbook of Chemical Microscopy*, Vol. II, McCrone Research Institute, Chicago, IL, 1989, pp. 203-206. (Book on shelf in Trace Library)

Selection of Methods

The current version of a method is documented in the performance verification for each area or instrument and is readily available to the analyst for reference unless it is not appropriate or possible to do so.

- Standard Method – published; performance verification
- Laboratory Developed Method – modification of a standard method; validation and performance verification
- Non-Standard Method – not covered by a standard method; validation and performance verification

For validation of methods, see the labwide quality manual.

Electronic Data

Computer software developed by the Trace Evidence Section must be documented and verified. Macros developed for use in Ignitable Liquids Analysis on the GC/MS is documented with the GC/MS Validation.

Data collected and stored on instrumental computers should be backed up yearly.

5.5 Equipment

5.5.1 Fourier Transform Infrared Spectrophotometer (FTIR)

Safety Considerations

- The microscope MCT detector must be cooled with liquid nitrogen. Insulated gloves and safety shield should be worn when filling the dewars.

Maintenance

- Records of all maintenance and performance checks are maintained in the FTIR logbook located next to the instrument.
- Bench:
    - Desiccant should be changed every six months or if the indicator on top of the instrument turns pink.
        - Rechargeable desiccant blocks should be placed overnight in an oven over 80 degrees C.
        - Allow to cool in bench top desiccator before placing in instrument.
- Microscope:
    - The MCT detector must be filled with liquid nitrogen prior to use.
    - The cassegrain mirrors and other optics of the microscope may be cleaned with a stream of dry, clean, nitrogen gas (canned air), when needed.
    - The MCT detector will lose vacuum and a drop in function will be noticed by observing the signal-to-noise. When the signal-to-noise drops below the accepted level, the detector must be sent for reconditioning.
        - Upon return of the detector, it must be re-installed and aligned.
- Universal Attenuated Total Reflectance (UATR):
    - The surface of the diamond, top plate, and bottom of the pressure arm should be cleaned with water, acetone, or another organic solvent after use.

Performance Checks

- Daily prior to use:
    - Bench: Instrument Verification
        - From the Spectrum menu, select Measurement/Instrument Checks/Instrument Verification. This test checks the abscissa, ordinate, noise, and ASTM (which includes a built-in polystyrene sample).
        - A report is generated which will indicate a “Pass” or “Fail” for each item. If any of the items are marked “Fail”, the system should be examined for the problem and the verification rerun.
        - The report is saved in C:\pel_data\Instrument Verification
    - Bench: Signal to noise ratio
        - Calculate the signal to noise ratio by taking 141.4 divided by the %P to P (found on the Noise test of the report).
        - Passing signal to noise ratio (S:N) is 10000:1

- Microscope: Energy
  - Monitor the energy and maximize by moving the condenser (correction adjustment bar)
  - Record the energy in the FTIR QC log. A drop in energy is the first indication that the detector is starting to lose vacuum pressure.
- Microscope: Signal-to-noise determination
  - Beam should be through the microscope with the path cleared for transmission or in air for reflectance or microATR.
  - Scan background using 100 x 100 µm aperture from 4000 to 500 cm^{-1} at a resolution of 1 cm^{-1} for 128 scans.
  - Without a sample in place, scan ratio using the same parameters.
  - Save the spectra in the S to N folder. View full scale from 2200 to 2100 cm^{-1}.
  - Calculate the signal-to-noise ratio by dividing 141.4 (constant) by the difference of the y-axes. Signal-to-noise must be greater than 1000:1 to proceed with case work.

- UATR: Ready Checks
  - From the Spectrum menu, select Measurement/Instrument Checks/Ready Checks/Run Selected. This test checks the abscissa and noise (including an internal polystyrene).
  - A report is generated which will indicate a "Pass" or "Fail" for each item. If any of the items are marked "Fail", the system should be examined for the problem and the ready checks rerun.
  - The report is saved in C:\pel_data\Ready Checks
- UATR: Signal to noise ratio
  - Calculate the signal to noise ratio by taking 141.4 divided by the %P to P (found on the Noise test of the report).
  - Passing signal to noise ratio (S:N) is 1000:1

- Monthly prior to use:
  - Bench:
    - There are no monthly tests for the bench.

  - Microscope: The polystyrene card should be run.
    - Check reference peaks 2849.1, 1601.4, and 1028.4 cm^{-1}. The peaks should fall within 2.0 cm^{-1} of these reference peaks.
    - Five peaks should be present in the 3110 to 3000 cm^{-1} range.
    - Save a copy in the polystyrene folder.

  - UATR:
    - There are no monthly tests for the UATR.

Quality Control

- The card holder for the bench unit can be removed to place the UATR component in place. A sensor in the unit detects what is attached.
- Ensure that the beam path is in the proper setup for analysis. Switching the beam back and forth from the bench or UATR to the microscope should be performed through the Spectrum software.
- It is recommended that the Spectrum software be placed offline before starting the Spectrum Image for the microscope control.
- After use of the microscope, the beam should be returned to the bench or UATR unit when finished or at the end of the day.
- The spectra for samples are compared to other known samples or to standard samples.
- Data manipulation (i.e. baseline correction, smoothing, etc.) is discouraged. If any changes are made to the spectra, a copy of the original, uncorrected spectrum will be included in the case file.
- The proper sample preparation techniques should be chosen depending on the type of sample to be analyzed.
    - Pellet
        - A KBr pellet may be made using infrared grade KBr.
        - The sample and KBr are mixed by grinding with a mortar and pestle.
        - A press is used to place the sample onto a windowed card.
        - A KBr only window may be made and a liquid sample dropped onto the window.
        - These pellets are for bench use.
        - A blank KBr pellet should be used as a background control.
    - IR Cards
        - Pre-manufactured PTFE (polytetrafluoroethylene) or PE (polyethylene) cards may also be used for bench analysis.
        - The major peaks of the PTFE or PE film should be checked to make sure they do not interfere with the sample.
        - These cards are best suited for bench use.
        - An unused card should be used as a background control.
    - KBr plate (pre-purchased), 1mm thick
        - The sample is prepped on a glass slide and transferred to the surface of a KBr plate which is in a holder.
        - These plates are for microscope transmission use.
        - A clean portion of the plate should be used for a background control.
    - Compression cell
        - Two KBr plates, up to 2 mm thick, are used.
        - The sample is placed on one plate and then another KBr plate is place on top. The sample holder is tightened to flatten the sample.
        - Care must be taken not to over tighten the sample which may cause the plates to break.
        - This compression cell is best used for microscope transmission use.

        - A clean portion of the plate should be used for a background control.
    - Diamond Window Microcompression cell
        - The bottom diamond plate is placed in the holder with a rubber o-ring.
        - The sample is placed on the plate and the other diamond plate is placed on top.
        - The cell is tightened to compress the sample.
        - The diamond substrate will produce some interference.
        - This compression cell is ideal for rubbery substances or small cross-sections for microscope transmission use.
        - A clean portion of the plate or an area containing KBr only should be used for a background control.
    - ATR Accessory (Germanium tip)
        - The ATR crystal should be placed on the surface to be tested.
        - The stage should not be moved once the crystal is in place.
        - This type of accessory is for flat surface analysis with microscope only.
        - Air should be used for the background control.
    - Reflectance
        - The microscope accessory is changed from Transmission to Reflectance.
        - The sample should be placed on a slide or mirrored surface.
        - This type of accessory is for reflective samples with microscope.
        - Air should be used for the background control.
    - UATR
        - This external accessory may be placed into the bench unit.
        - The crystal is zinc selenide topped by a thin diamond.
        - Little to no sample preparation is required for using the UATR. Only a small amount of powder is needed to be compressed by the pressure arm. A drop of liquid or smear of a thicker substance on the surface is sufficient and may not require the use of the pressure arm. Samples such as tapes and polymers may be placed directly on the crystal.
        - If the pressure arm is used, the pressure measured by the force gauge should not exceed 100.
        - A clean crystal without the pressure arm in place is used for the background control.

Emergency Shutdown Procedures

- Shut down all software programs.
- Turn computer off.
- Turn off IR bench (toggle switch on back).
- Turn off microscope (toggle switch on back).

References – All manuals for the instrument are on the computer desktop.

5.5.2 Gas Chromatograph/Mass Spectrometer (GC/MS)

Safety Considerations

- The injector port may be hot. Caution should be taken when changing the septum or liner.
- The oven should be cooled before changing the column.

Maintenance

Records of all maintenance and performance checks are recorded in the GC/MS logbook located next to the instrument.

- Routine:
  - The septum should be replaced before the next run after reaching 200 injections.
  - The injection liner should be replaced before the next run after reaching 400 injections.
  - The compressed gas cylinder providing helium to the system will be changed as needed.
- Non-routine:
  - The source should be cleaned.
  - Used filaments should be replaced.
  - Diffusion pump oil should be inspected and replaced if necessary.
  - Rough pump oil should be checked and filled or replaced if necessary.

Performance Checks

- Daily prior to use:
  - Standard Autotune (PFTBA internal standard used to optimize parameters)
    - Check autotune report:
      - If any m/z peaks below 69 m/z are above 10% relative abundance to the 69 m/z, it is an indication of a leak. The instrument should be removed from service until it is repaired or has passed the performance check.
      - Electron Multiplier (EM) Voltage maximum is 3000. Normal ranges vary from instrument to instrument. If the EM voltage is between 2500 and 3000, the instrument should be removed from service until it is repaired or has passed the performance check.
      - Iso Ratio for masses 69:219:512 should be approximately 1:4:10.
    - A copy of the autotune is printed and kept in the logbook beside the instrument.
      - The autotune should be initialed by the analyst to signify that the parameters above were checked.

Quality Control

- Check samples required by type of analysis should be recorded on the worksheet or in the case notes. (e.g. AGKD and ASTM Sensitivity).
    - A copy of the check sample should be printed and placed in the binder or for non-routine samples, in the case notes.
- Blanks shall be run between sample injections. The extraction blank may be used as a blank that is run between samples.
- The blank, standard, and samples must be run under the same chromatographic conditions and data acquisition parameters.
- For a single compound identification, a mass spectrum should be included in the case file.
- For identification of a compound or substance, no rigid mass spectral probability based match criteria are defined. Flexibility is given to the experienced analyst. The identification will be based on a number of factors which may include retention time, extracted ions, ion abundance, and literature references.
- When case samples are weak, the samples may be concentrated and sampled in a microvial insert. It may be useful to also inject a larger volume and examine the sample using selected ion monitoring (SIM).
    - It is necessary to know the major ions of interest for SIM monitoring.

Emergency Shutdown Procedures

- Note: Shutdown process will take approximately one hour.
- From the software, select Vacuum Control/Vent/OK
    - A time frame will be given on how long it will take to cool the filament.
    - A prompt will appear with notification that the instrument may be turned off.
- Turn off the buttons on the bottom front of the mass spectrometer and the bottom front of the gas chromatograph.
- Shut down the computer.

References– Located above the instrument

- "HP 5973 Mass Selective Detector. Hardware Manual", Hewlett Packard Company, P/N G1099-90027, November 1998.
- "HP 6890 Series Gas Chromatograph Operating Manual", Hewlett Packard Company, P/N G1530-90440, October 1996

5.5.3 Glass Refractive Index Measurement System (GRIM3)

Safety Considerations

- Care should be taken when preparing samples of broken glass for analysis.
- Do not directly touch the hot stage which can reach high temperatures. Slides removed from the hot stage may also be hot.
- Do not look directly into the microscope if the lamp intensity is turned up for direction through the photo/video port.
- Do not remove the glass heat shield from the top slit of the hot stage apparatus.

Maintenance

- Records of all maintenance and performance checks are maintained in the GRIM3 logbook located in the drawer below the instrument.
- Dust the microscope, monitors, and computer as needed.
- The microscope should be set for Köhler Illumination and phase contrast.
- Clean the hot stage sample holder as needed to remove excess silicone oil which may be deposited.

Performance Checks

- Daily prior to use:
  - A glass reference standard will be examined in the respective oil.
    - At least five particles are tested from one prepared slide.
    - The mean of the measured RI should be +/- 0.00010 of the reported value for the sample.
      - A4/488 1.54639
      - A4/590 1.53998
      - A4/656 1.53729
      - B6/488 1.52245
      - B6/590 1.51671
      - B6/656 1.51428
      - C1/590 1.48648
      - C1/656 1.48440
- Monthly prior to use:
  - The SRM 710 control sample is used to verify the B 590 curve.
    - At least ten particles are tested from one prepared slide.
    - The mean of the measured refractive index should be 1.52346 +/- 0.00010.
- Yearly:
  - The system will be checked annually by using the "re-calibrate*" selection in the software for each silicone oil (A, B, and C) and each filter (488nm, 590nm, and 656nm).
    *It is noted that this is not a recalibration but a performance check. However, the internal software choice which must be selected is designated "recalibrate".
    - Six readings from each of the glass standards will be tested.
      - The dRI for each glass standard should be between -10 and +10.

- The correlation coefficient for each oil and wavelength filter curve should be >+/-0.9000.

Quality Control

- Make sure the upper plunger is on the GRIM setting.
- The microscope settings (on the right of the base) should be at 400-700µ and the daylight filter at 3000.
- There should be no filter in the top (DF/BF) so the setting is for the empty slots of 2 or 3.
- The aperture should be opened to the PH setting and the PH1 aperture rotated into place.
- Each time a new sample is placed on the hot stage, the sampled should be focused and the phase rings aligned.

Emergency Shutdown Procedures

- Turn down the microscope lamp intensity.
- Turn off the microscope.
- Close down any software programs.
- Turn off the GRIM3 tower.
- Turn off computer.

References– Located in the drawer beside the instrument

- ASTM E1967 Standard Test Method for the Automated Determination of Refractive Index of Glass Samples Using the Oil Immersion Method and a Phase Contrast Microscope.
- "ffTA-RI (GRIM3), Glass Refractive Index Measurement System, User Manual", foster + freeman.
- "GRIM: Stage Manager, Glass Refractive Index Measurement System, Software Manual", foster + freeman.
- "GRIM: Glass Software, Glass Refractive Index Measurement System, Software Manual", foster + freeman.

5.5.4 Microscopes

Safety Considerations

- Do not look directly into microscope with lamp on highest setting.
- The lamps can get hot. Caution should be taken when replacing a bulb that has recently burned out.

Maintenance

- Records of all maintenance and performance checks are recorded in the logbook located next to each instrument.
- Microscopes should be cleaned to remove dust.
- Light bulbs may need to be changed if a change in illumination is detected or if the bulb burns out.
- Lenses (eyepiece, objective, condenser, etc.) should be cleaned as needed with lens paper.

Performance Checks (does not apply to stereomicroscopes)

- Daily prior to use:
  - Confirm that the microscope is set for proper Köhler illumination.
- Monthly prior to use:
  - Set the microscope up for Köhler illumination.
  - Record in logbook.

Quality Control (does not apply to stereomicroscopes)

- Yearly prior to use:
  - Recalculate the eyepiece micrometer for each objective using a stage micrometer.
  - Document the conversion from units to micrometers in the logbook.

Emergency Shutdown Procedures

- Turn lamp brightness down.
- Turn microscope off.

References– Located in the Trace Evidence library

- McCrone, Walter C., *The Particle Atlas*, 1967 (and other editions).

5.5.5 ffTA-1 Microspectrometer (ffTA-1 MSP)

Safety Considerations

- The high lamp intensity required for effective microspectrometer operation may cause eye injury if viewed through the eyepieces. Always set up Köhler illumination using a low lamp intensity before turning up the power to stabilize.
- Allow bulbs to cool before changing.
- Do not look directly into LED light.

Maintenance

- Records of all maintenance and performance checks are recorded in the ffTA-1 logbook located in the drawer below the instrument.
- Lamp may need to be changed periodically.
- Clean dust on optics with a stream of dry, clean, nitrogen gas (canned air) when needed. A cloth may be used to clean the stage and computer screen.

Performance Checks

- Daily prior to use:
    - 100% Transmission line
        - Turn on transmitted light source
        - Find clean spot on slide
        - Record white reference (100 scans)
        - Record dark reference (100 scans)
        - Record spectra (100 scans)
        - Save in QC folder.
        - Spectrum should be essentially flat at 100% transmission +/-5%.
    - Wavelength Accuracy and Precision using the didymium standard
        - Make sure plunger is on µSpec and the Phase Contrast ring has been removed.
        - Set up Köhler Illumination.
        - White balance camera on a clean area of the slide
        - Record white reference
        - Record dark reference
        - Place didymium filter cube over light source
        - Record spectra (1 count)
        - Select Calibration/Auto calibration/calibrate didymium filter
        - Copy to clipboard and save as word file.
        - Save spectra as C:\users\fftaUser\QC\didy
        - Selected peak wavelengths (472.2nm, 512.4nm, 681.3nm, and 876.14nm) should be within +/-1 nm of the expected value.

- Monthly prior to use:
    - Check position of measurement box.
        - Turn on reflectance light source
        - Put in BF filter
        - Focus on aluminum foil
        - Turn off reflectance light
        - Turn on LED light (in ffTA-1 program)
        - Focus light
        - Right click on video box
        - The LED spot should be in the center of the cross-hairs.
            - Click center image
            - Use arrows to center light
        - Turn off LED light and reflectance light source.

    - Photometric Accuracy and Precision using neutral density filters
        - A scan of each neutral density filter (0.1, 0.5, and 1.0 O.D.) will be examined.
            - Place the filter on the light source.
            - Take spectra (100 scans in absorbance) of each of the filters.
        - Save in QC folder
        - Noise should be at a minimum.

Quality Control

- Turn instrument and accessories on and allow to warm up for at least 30 minutes.
- Ensure that the round collection spot is positioned entirely on the sample.
- Record a new white reference each time the sample is changed.
- Make sure the upper plunger is on the µSpec setting.
- The microscope settings (on the right of the base) should be at 400-1000µ and the daylight filter at 6000.
- The darkfield filter (DF 1) should be positioned in the top.
- Ensure filter on lower lens has been removed.
- Move the aperture wheel to an open position (not PH1).
- Use the thick PLAN EPI objectives

Emergency Shutdown Procedures

- Turn down the microscope lamp intensity.
- Turn the microscope off.
- Shut down all software programs.
- Turn off the computer.
- Turn off the power to the µspec box.

References– Located in the drawer beside the instrument

- “Transmitted Light Examinations Using the ffTA Microspectrometer”, foster + freeman.

5.5.6 Electrostatic Deposition Apparatus (ESDA)

Safety Considerations

- The corona wire contains high voltage. Do not touch the wire. Do not operate without the yellow protecting bars.
- Always rest the corona unit with the emitting side downwards on a flat surface.

Maintenance

- Records of all maintenance and performance checks are recorded in the logbook located near the instrument.
- Cascade developer should be removed from the catch tray after use.
- The cascade developer will periodically require recharging with toner. This is indicated when development consistently produces weak images with very little background color. Add one-half teaspoon of toner for each cup of glass beads. Shake to coat the beads with the toner.
- Fill the humidifier with water to a depth of 20 mm before using.

Performance Check/Quality Control

- Daily prior to use:
    - A control strip of paper is folded lengthwise and the case number, item number, date, and analyst initials will be written on it using a ballpoint pen.
    - The paper is then unfolded and procedures for analyzing documents are followed.
    - The indentions on the test strip should be visible on the opposite side from the writing.
        - If indentions on test strip are not visible, case work cannot continue.
    - Document this information in the case file.

Emergency Shutdown Procedures

- Ensure that corona switch is in the "off" position.
- Turn the pump off.
- Note: This instrument stays off when not in use.

References (located in the room near instrument)

- ESDA Operating Instructions, Foster & Freeman, Ltd.

5.5.7 Scanning Electron Microscope/Energy Dispersive X-Ray Spectrometer (SEM/EDS)

Safety Considerations

- The SiLi EDS detector system must be cooled with liquid nitrogen. Insulated gloves and safety shield should be worn when filling the dewars.
- Care should be taken when removing the Wehnelt cap from the instrument. If the filament has been in operation, it may be hot.
- Do not open the SEM chamber door without first turning off the vacuum.

Maintenance

- Records of all maintenance and performance checks are recorded in the SEM/EDS logbook located next to the instrument
- A service contract is maintained for both the Scanning Electron Microscope and the Energy Dispersive X-Ray Spectrometer.
    - The Scanning Electron Microscope preventative maintenance occurs twice a year. Copies of the paperwork are kept in the SEM maintenance folder.
        - Pump oil is replaced
        - Apertures are cleaned or replaced
        - Magnification is verified
    - The Energy Dispersive X-Ray Spectrometer preventative maintenance occurs annually. Copies of the paperwork are kept in the EDS maintenance folder.
        - Brightness/Contrast correlation between EDS and SEM is checked.
        - Field overlap is checked.
- The liquid nitrogen dewar on the X-ray detector is filled to ensure that it does not go dry. A calendar posted in the room shows when it was filled.
- When a filament burns out, the Wehnelt cap is cleaned and the filament is replaced in the instrument.
    - Cleaning the Wehnelt cap:
        - Sonicate in an approximate 10% solution of Micro 90 and water; rinse well. (optional)
        - Polish with metal polish; rub clean.
        - Sonicate in ethanol.
        - Sonicate in acetone.
        - Allow to dry.
        - Place a new or re-tipped filament assembly in cap.
        - Using stereomicroscope, align filament.
        - Store wrapped in foil until ready for use.
    - Replacing the filament:
        - Place a clean Wehnelt cap into the instrument and turn on the vacuum.
        - Ensure that the lid has a proper vacuum seal.
        - Allow the instrument vacuum to pump down for at least 10 minutes.
        - Make sure the gun load current has been turned down.
        - Turn the filament (HT) ON.

        - Slowly raise the load current to the mark and then gradually increase to the orange bar. This should be done over a 10 minute to 2 hour period.
        - Allow the filament to warm up for a minimum of 30 minutes.
    - Align the filament:
        - Saturate the filament.
        - Obtain an image by aligning the filament using the SEM controls for spotsize, shift and tilt.
        - Set the Bias.
        - Adjust the wobble.
        - Adjust the astigmatism (STIG).
- The detector may be conditioned.
    - To condition the detector:
        - Close the INCA program and open the INCA Monitor program from the right hand toolbar.
        - Open Thermal Options
        - Condition detector; the process takes over two hours.

Performance Checks

- Daily prior to use:
    - The detector's resolution should be measured and recorded in the log book each time the instrument is used.
        - This is performed by measuring the fwhm (full width at half max) of the Cobalt peak collected on the calibration stub at 20 keV.
        - The EDS software calculates this value under the GSR "Quant Cal". The sample is measured at least twice and the results are saved to report.
        - The Quant Cal report is printed on screen and the value recorded in the logbook.
        - The value should not exceed 150 eV.
    - When running the automated gunshot residue program, the EDS threshold levels should be set using the high Z / low Z standard stub of cobalt and rhodium.
        - The brightness and contrast are adjusted to optimize the levels at 128 and 255.
    - A known sample is tested prior to use.
        - For GSR, metals, and other unknowns, a known reference area is collected (Spike). At least 30 particles must be present in the threshold detected features. The number of particles found is documented in the logbook.
        - For paint, the OU1 layer of a known paint sample (Paint1) is collected. All of the major elements must be identified. The logbook is checked as passing.
        - For glass, a known glass sample (SRM 710) is collected. All of the major elements must be identified. The logbook is checked as passing.

    - Before running the automated gunshot residue program, the recipe, parameters, and focus should be checked for each stub that has been placed in the holder.
- Monthly prior to use:
    - Before using the automated gunshot residue program, a known stub containing various sizes of characteristic gunshot residue particles will be tested. (PLANO)
        - Finding 39 (or more) characteristic particles is considered passing.
        - Document in logbook.

Quality Control

- Before testing, each stub is marked on the bottom with the item number or the pre-stamped number is recorded on the gunshot residue worksheet.
- Each stub is marked and placed in the stub holder to align the mark on the stub with the mark on the holder. If the stub is removed, it may be returned to the same position to locate particles.
- Only stubs from one individual at a time are placed in the chamber for gunshot residue analysis.
- A blank stub is tested with each automated gunshot residue run.
    - If characteristic or bi-component gunshot residue particles are found, then the results for the automated run must be discarded.
        - Replace the blank and rerun the samples.
        - If the blank still has characteristic or bi-component particles, immediately cease case work. The problem must be corrected before resuming case work.
    - If other indicative particles are found, the blank will be replaced with a new blank stub.
- During the automated gunshot residue run, the threshold (high Z / low Z) is checked every 30 minutes. If the check does not pass, the programmed run will stop.
- Prior to the manufacture of gunshot residue kits distributed by the Arkansas State Crime Laboratory, the manufacturer sends a sample kit for approval.
    - Inspect the kit to ensure all components are present.
    - Visually inspect the sample stubs. The stubs should be completely covered with the carbon adhesive.
    - Run one stub from the kit using the automated gunshot residue program to ensure that no primer gunshot residue particles are present.
    - Return the information sheet to the manufacturer.

Emergency Shutdown Procedures

For normal shutdown, vacuum pump should be left on (omit last step).

- Turn filament (HT) OFF.
- Close computer software programs.
- Shut down both computers.
- Turn key on front of SEM to OFF.

5.5.8 Other Equipment

Balances

- All balances are cleaned, serviced, and calibrated every five years by an outside vendor.
    - The service report will be kept in the Balance Log located next to the balance.
- The performance verification of the balance should be checked before use with a known weight set from the Drug Chemistry Section.
    - Measurements should fall within the tolerance for the weight set used.
    - The performance verification is verified and recorded on a balance log sheet.
    - If a result from the performance verification check is outside the acceptable range, ensure that the balance is clean and level and recheck.
    - If the result is still outside the acceptable range, the balance should be removed from service immediately.
- Weights measured in case work in the Trace Evidence Unit may be used for comparison purposes but will not be reported.
- Weights used in the preparation of reagents are non-critical.

Chemicals and Reagents

- Solvents used for standards, extractions, or to dissolve samples or standards should be a high quality, low residue solvent.
- Water used in reagent preparation or as an extraction solvent should be either deionized (DI) or reverse osmosis (RO).
- All chemicals and commercial reagents are labeled when received and when opened with the date and initials of the individual receiving or opening them.
    - Working bottles of chemicals or reagents should be labeled at a minimum with the name of the chemical or reagent, lot number, expiration date, and initials of the analyst.
- All chemicals and commercial reagents should be disposed of and replaced when they fail to perform adequately under controlled conditions.
- All bottles of reagents prepared in the laboratory must be labeled with the contents, the date prepared, the expiration date, and initials of the analyst who prepared the reagent.
    - Directions for the preparation of commonly used reagents are found in the Reagent Logbook located in the Trace Evidence Unit Library.
    - The reagent preparation and quality check will be recorded in the reagent logbook.
    - The reagent should be checked prior to use in casework. The quality check should be recorded in the reagent logbook.
    - If a reagent fails the quality check, immediately dispose of the remainder of the reagent.
- Reagents prepared for non-routine examinations are tested with known samples and blanks. The results are recorded in the case file notes.
- Test strips are checked with appropriate standards and results are recorded in the case notes.

    - Test strips may continue to be used past their stated expiration date as long as they pass the quality control check.
- Flammable chemicals should be stored in a flammables cabinet.
- Each time a new chemical is received in the Trace Evidence Unit, a MSDS sheet should be obtained and provided to the section safety manager.

Micrometers and Calipers

- Performance verification checks are performed annually.
    - The performance verification is checked with two certified standard gauge blocks from the Firearm/Tool Mark Section.
    - The measured values are recorded on the log sheet located in the cabinet with the equipment.
    - The measured values shall be within +/-0.02mm or 0.001".
        - If the values are outside the appropriate range for a given block, then measure again.
        - If the values are still outside the appropriate range, then the micrometer or digital caliper must be sent to an outside vendor for recalibration.
        - Alternatively, the device may be removed from service and a new device purchased.
    - If any micrometer or digital caliper is dropped or otherwise subjected to extreme temperature or shock, then the performance verification should be checked prior to use in casework.

Refrigerators, Thermometers, and Ovens

- The temperature should be documented on the log sheet beside the refrigerator and ovens before placing evidence into or removing evidence from a refrigerator or oven.
    - The refrigerator temperature should be between 0 and 10°C.
        - If the temperature is outside this range, document in case file notes (if applicable).
        - Move all evidence, standards, reference materials, and any other items stored in the refrigerator to another laboratory refrigerator until the problem is fixed.
        - Some standards, reagents, and/or reference materials last longer when refrigerated. The temperature should be recorded before removing any of these items.
    - The oven used for fire debris samples should have a temperature between 60 and 70°C.
        - If the temperature does not read as specified, then the thermostat will be adjusted to obtain the correct temperature.
        - If the correct temperature cannot be achieved, the oven will be removed from service until repaired.
    - Any maintenance or repair to a refrigerator or oven will be documented on the log sheet.

5.6 Measurement Traceability

Calibration requirements for each instrument are recorded in section 5.5. Performance verification for each instrument is recorded in section 5.5. In addition to the normal checks, the verification will be performed after any maintenance or repair to the instrument.

The performance check of the instrumentation/equipment, whenever possible, will be traceable to the International System of Units (SI). When using external calibration services, traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability. The calibration certificates issued by these laboratories shall contain the measurement results, including the measurement uncertainty and/or a statement of compliance with an identified metrological specification.

There are certain performance verifications that currently cannot be strictly made in SI units. In these cases, performance verifications shall provide confidence in measurements by establishing traceability to appropriate measurement standards such as:

- The use of certified reference materials provided by a competent supplier to give a reliable physical or chemical characterization of a material;
- The use of specified methods and/or consensus standards that are clearly described and agreed by all parties concerned.

All of the instruments in the Trace Evidence Unit are category 2. Trace Evidence does not record measurements in external reports.

Reference Standards and Reference Materials

Reference standards and reference materials are listed in sections 5.4 and 5.5. Reference Standards used in length may be found in the Caliper and Measurement (C/M) logbook located in the cabinet above the GRIM3/ffTA-1. Reference standards used in weights may be found in the Balance logbook located on the counter in room 348.

Each reference standard or reference material should be handled with care. Gloves may be worn when handling reference standards or materials. Samples should be stored and transported in the storage cases provided, if possible. Most reference materials are kept near the area of use.

Each reference collection of items in the Trace Evidence Unit will be documented on the S: drive in an Excel spreadsheet. The items are given a unique identifier and kept in storage within the unit. Other collections within the unit are used for training purposes but not for comparisons to casework.

Spectral libraries used as reference are maintained on the instrument. Each has a library listing of the contents and a unique identifier.

The schedule for performance verification of standards is recorded in section 5.4 and/or 5.5.

5.7 Sampling

The sampling plans are recorded in section 5.4 under the specific type of analysis, if appropriate.

Deviations from the sampling plan and procedures as requested by an agency or as deemed appropriate by the analyst should be approved by the section chief.

The sampling plan used in routine casework, as documented in the Trace Evidence Quality Manual, is not required to be recorded in the case notes. Any deviations or elaborations should be documented in the case notes.

5.8 Handling of Test Items

In order to determine the items most likely to assist in the investigation and prioritize those items for examination, an agency representative may be consulted. This conversation should be documented in the Case Images and/or the Request in Justice Trax.

Evidence Retention Policy
Tape lifts, hairs, paint chips, glass fragments, SEM stubs, etc. which have been collected from or removed from items of evidence should be given a unique identifier and itemized in the LIMS. The items may be placed in a separate envelope and retained in secure storage, transferred to another section for analysis, or returned with the original evidence.

Carbon strips collected for ignitable liquids analysis are placed in a vial in the original container and returned with the evidence.

If an entire originally submitted item is retained (i.e. a hair, tape lift, paint chip, etc.), the original packaging should also be kept with the item.

Evidence collected and retained will be stored in the Trace Evidence Secure Storage lockers or transferred to another analyst or secure storage location (i.e. PE Secure Storage or FD Secure Storage).

Evidence collected and retained in the Trace Evidence Secure Storage location is periodically returned to the agency. The items are usually retained for at least 5 years. If the case is active, they may be kept for a longer duration.

All evidence in the possession of an analyst but not in the process of examination or analysis shall be maintained in the secured work area of the analyst or within the Trace Evidence Unit which has limited access.

Unattended evidence, especially bulky items, may be left in the secured Trace Evidence Unit area. Care should be taken to protect the loss, damage, or change of evidence on specific areas.

When evidence can only be recorded or collected by photography, the photographic image must be treated as evidence.

Evidence collected by trace evidence analysts from a crime scene or vehicle should be protected from loss, cross transfer, contamination, or other change during transport to the ASCL. The evidence should be appropriately identified, packaged, and entered into the LIMS as soon as possible.

5.9 Assuring the Quality of Test Results

This manual contains quality control procedures to monitor and ensure the validity of test results. See sections 5.4 and 5.5. Quality control data is documented in logbooks or recorded in the case file. Some instruments have more detailed quality control information stored on the hard drive.

Work on the case only proceeds if quality control measures are acceptable. If the quality control data is outside the acceptable criteria, case work is not initiated or continued. The problem should be determined and corrected with passing quality control before case work proceeds.

All probative hair associations must be verified by another qualified analyst.

Proficiency Testing
Each analyst engaged in testing shall be proficiency tested at least once per year. If the analyst conducts testing in more than one category, the analyst will be tested in each category in which they perform testing at least once during each five year accreditation cycle.

The categories of proficiency testing for Trace Evidence include fire debris, gunshot residue (stubs), paint, fibers, glass, hair, tapes, lamp filaments, and general physical and chemical analysis.

At least one of the proficiency tests for Trace Evidence each year must be from an external provider.

Case Review
Every report generated by the Trace Evidence Unit is reviewed technically and administratively.

The review form TR-FORM-01A is used for cases in which one analyst will be reviewing the case. If the case involves multiple categories of testing which require more than one analyst to review, form TR-FORM-01B may be used.

If the reviewer discovers an error in the case record, the reviewer must document the error on the review form and inform the analyst. If the analyst and the reviewer cannot reach consensus, then both the analyst and reviewer must meet with the section chief (or designee) for resolution.

Technical reviews must be conducted by individuals having expertise gained through training and experience in the category of testing being reviewed. An individual conducting the technical review does not have to be an active examiner or currently proficiency tested but must have sufficient knowledge of the discipline to verify compliance with the unit's technical procedures and that the conclusions reached are supported with the examination documentation. Anyone not currently competent in the discipline must be authorized to perform technical reviews by the section chief. This authorization documentation will be maintained in the individual's employee

history binder. If the review is conducted by a qualified analyst who is not an employee of the Arkansas State Crime Laboratory, the reviewer must be from an accredited laboratory. The accreditation certificate for the laboratory and a CV for the individual conducting the review will be maintained on file (S:\Technical Reviewers).

Administrative reviews may be conducted by any laboratory analyst or other individual qualified to perform technical review. The administrative reviewer of a case that has been technically reviewed by an outside agency will push the technical review in the LIMS before proceeding with the administrative review. The administrative reviewer will ensure that the completed review form has been scanned into the case file.

Technical and administrative reviews may not be conducted by the author of the report.

Refer to the labwide quality manual for instructions on testimony review.

5.10 Reporting the Results

When analytical conclusions and/or opinions are made on evidence submitted for analysis, a "Report of Laboratory Analysis" will be issued to the investigating agency. The results shall be reported accurately, clearly, unambiguously, and objectively. Each analyst will proofread and sign their reports ensuring the report is accurate. LIMS allows the analyst to electronically sign reports.

A laboratory report is not required if an internal request is made. (e.g. Determining if there is tissue present on the root of a hair for DNA to proceed with analysis.) However, if the examination results in gaining additional probative information or additional samples are needed, a report should be issued. See labwide quality manual for additional exceptions.

See the labwide quality manual for minimum laboratory reporting requirements. In addition, Trace Evidence reports will contain the types of analysis used in the testing of the evidence. See section 5.4 for examples of reporting for each category of testing.

Analysts testifying based on the examination records generated by another analyst shall complete a Court Case Review Form (ASCL-FORM-57) on the particular case prior to testifying. This form will be scanned into the case file.

If testing results are obtained from a subcontractor (i.e. FBI, FBI regional laboratory, etc.), a report will be issued by the crime laboratory stating that the report is attached. The original report will be scanned into the case file and the report will be sent to the agency requesting the testing.

ASCL reports are generated using the LIMS and will be formatted in a manner to accommodate the types of tests conducted and to minimize the possibility of misunderstanding or misuse. Section chiefs should ensure that discipline report designs are optimized for the clear presentation of test results.

See labwide quality manual for information on supplemental and amended reports.