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TR-01 Gas Chromatograph Mass Spectrometer for Trace Analysis

Principal of Protocol:

Gas chromatography is responsible for separating various components within a volatile mixture. A gas chromatograph consists of three basic parts: an injection port, a column, and a detector. In the case of a Gas Chromatograph/Mass Spectrometer (GCMS) the detector is a mass-selective detector (MSD). A liquid or gas sample is injected into the heated injection port where it is vaporized. The volatilized sample is then moved through the column by the carrier gas. The various compounds within the sample will be separated based on boiling point and affinity for the column. When the separated components exit the column they enter the mass-selective detector. The sample components are fragmented by electron impact (EI) into molecular and fragment ions. A mass filter sorts the fragments creating a fragmentation pattern that can be compared to the fragmentation patterns of known standards.

Associated Protocol(s):

TR-100 Analysis of Ignitable Liquids
TR-101 Ignitable Liquid Sampling Apparatus

Specimen(s) Required:

Liquid or vapor (gas) samples

Solvents:

Refer to the appropriate protocol for recommended standards.

Controls:

1. Each day casework is to be performed, an autotune and tune evaluation will be performed. This will be archived in the case file.

   The following require action before casework testing can commence, but not removal of the instrument from service:

   a. When high levels (above approximately 10% on the scale of the autotune) of O2, H2O are present in the autotune:

      1. Find and repair the leak immediately and document in the maintenance log.

   b. Failure of the instrument tune evaluation:

      1. The operator is to diagnose and correct the problem, and document in the maintenance log.

      2. If the operator is unable to determine the cause of the failure, contact the technical manager or designee to determine the best course of action.

2. An appropriate positive control will be analyzed each day casework is performed. The results of the positive control will be archived in the case file. A solvent blank will be injected before each case as a negative control and the results will be archived in the case file. The solvent blank must be the solvent used in the analysis of the sample, if a solvent was used, i.e. carbon disulfide for ignitable liquid cases.

If the controls do not meet the pre-defined criteria, the test should be repeated to determine the source of the contamination/issue. If the results of the second controls meet the pre-defined criteria, all samples should be re-run. If the results of the second test are unacceptable, the analyst will take steps to resolve the issue prior to re-sampling and/or any further analysis.

Apparatus and Materials:

1. Merlin Microseal
2. Gas chromatograph/Mass Spectrometer
3. Appropriate split/split less injection liners
4. Appropriate capillary column
5. Five (5) microliter or ten (10) microliter volume syringes compatible with Merlin Microseal
6. Appropriate Accessories

Individual Steps of Protocol:

1. Inject a diluted sample onto the GCMS. The autosampler or manual injection may be utilized.
2. The analyst will directly compare the questioned sample to a standard spectrum for interpretation (refer to the specific protocol for the proper interpretation).
3. For specific sampling techniques, consult the appropriate protocol(s).

Protocol Notes:

1. Any sampling component will undergo function verification prior to being put into service.
2. Maintenance includes cleaning and/or changing the Merlin Microseal, liner o-ring, liner, gold seal and washer, and cleaning the MSD source. Metal septum nuts are to be cleaned or replaced when the Merlin Microseal is changed. Changes and cleaning should be performed when sample selectivity or sensitivity suffers, as needed. Maintenance and/or repairs will be documented in the instrument maintenance spreadsheet, located on \\vm-fsc-files\Common\Trace\1 - Trace Instrument Maintenance.
3. Refer to www.agilent.com for instrumentation user's manual.
4. There are no calibration records for the gas chromatograph/mass spectrometer. Autotunes are documented in each casefile.

Recommended Report Wording / Interpretation of Test Data:

A comparison is made between the data obtained from the sample and an in-house standard spectra or a spectra obtained from other evidence submitted for analysis. If the two spectra are consistent, a copy of the in-house standard spectra or other known spectra shall be placed in the case file.

Recommended report wording can be found in the protocol for the type of analysis being performed.

TR-06 Fourier Transform Infrared (FTIR) for Trace Analysis

Principle of the Protocol:

Infrared spectroscopy is a nondestructive technique that allows the examiner to classify binder types and/or extenders in paint samples, identify polymer composition of man-made fibers and identify or compare other organic and some inorganic compounds.

Associated Protocol(s):

TR-160 Paint and Polymer Analysis
TR-170 Fiber Analysis

Specimen(s) Required:

1. Fibers
2. Paint
3. Other samples

Solvents:

Liquid Nitrogen

Standards:

1. Thermo Scientific 1.5 mil Polystyrene Standard

Controls:

Each day casework is to be performed, the FTIR IS50 must pass the ValPro Qualification. If the Continuum is going to be used in casework, liquid nitrogen needs to be added to the detector and a polystyrene standard must be analyzed. All documentation will be archived in the case file.

If a ValPro System Qualification of the bench fails, repeat the ValPro System Qualification. If this fails, the following actions should be taken:

1. Determine performance problem, repair, verify instrument performance by running a ValPro System Qualification and document in the maintenance log.
2. Notify the Technical Manager or Supervisor if the instrument needs repair by vendor and/or taken out of service.

If an unsatisfactory polystyrene standard is obtained, the following actions will be taken:

1. Obtain a 2nd polystyrene spectrum.
2. If the 2nd spectrum is unsatisfactory, remove the instrument from service and notify the Technical Manager or Supervisor.
3. Determine the performance problem, repair. Verify instrument performance by analyzing a polystyrene standard and document in the maintenance log.

Apparatus & Materials:

1. Thermo Fisher Nicolet IS50 Analytical FTIR Spectrometer equipped with OMNIC Software and ValPro System Qualification Software
2. Nicolet Continuum FT-IR Microscope
3. Microcompression Cells, DuraSamplIR ATR or other accessories

TR-06 Fourier Transform Infrared (FTIR) for Trace Analysis

Individual Steps of Protocol:

1. Sample Preparation – Sample preparation techniques are dependent on the type of sample to be analyzed. The most frequently used sample preparation techniques are listed below:
    a. Microcompression Cell with Diamond Windows
        1. Hand-tighten to compress/flatten the sample.
        2. A stereomicroscope should be utilized to prevent over-tightening and damage to the cells.
    b. Flattening (other)
        1. Flattening of the sample may be accomplished through various means (roller or other).
    c. KBr Pellet
2. **Sample Analysis**—the size of the sample area to be analyzed will determine if the spectra can be collected utilizing the ATR accessory or the microscope.
    a. ATR
        1. Properly install the DuraSamplIR ATR Accessory.
        2. Load the "Bench" Experiment Setup.
        3. Place the sample between the crystal and pressure tip. Flatten the sample by turning the knob clockwise.
        4. Collect the sample and background spectra.
    b. Microscope
        1. When using the Microscope, the detector must be cooled with liquid nitrogen.
        2. Align the microscope prior to analyzing each sample (See section c: Steps for alignment of the microscope).
        3. Adjust the sampling window on the microscope to the approximate size and shape of the sample to be analyzed.
        4. Collect the sample and background spectra
    c. Steps for Alignment of the Microscope
        1. Place the desired sample in the sample path.
            a. For ease of locating the sample, the 10x objective can be used.
            b. Focus on the sample using the reflachromat 15x objective, or another appropriate objective.
            c. Upon achieving the correct focus, do not move the stage/focus knobs.
        2. Select sampling mode (transmission or reflection) by pressing the Sampling Mode switch on the front panel of the microscope.
        3. Adjust the aperture and light intensities to achieve appropriate illumination.

4. Adjust the compensation ring on the objective and condenser to their appropriate values.

   Note: The compensation ring (condenser or objective) is used to adjust for the thickness of the sampling window / pellet. Usually, the compensation ring is adjusted to the thickness of the window that is used. For example, if nothing is in the path, compensation is 0. If there is a 1mm thick window **under** the sample, compensation on the **condenser** is 1. If there is a 1mm thick window **above** the sample, compensation on the **objective** is 1.

5. Place the pinhole slide in the universal slide holder. Do not allow the stage to bump the objective or condenser, as this could damage the microscope.
6. Position the pinhole directly under the objective.
   a. Look directly at the slide (not through the viewer) and use the stage focus controls to raise or lower the stage until the spot of white light on the slide is most concentrated (the smallest spot size).
   b. While looking directly at the slide, center the illuminated area on the slide pinhole (the sampling mode should be reflection for this portion of the alignment).
   c. A single image of the pinhole through the viewer should be observed when the eyepieces are positioned correctly.
7. While looking through the eyepieces, use the condenser focus knob to move the condenser up or down to bring the aperture into sharp focus.
8. Remove the pinhole slide from the stage, open the transmission and reflection field of view controls fully and turn on the Reflex aperture illuminator.
9. If the aperture image is offset, use the condenser knobs to center it.
10. The FTIR microscope is now aligned and is ready to collect spectra.

Protocol Notes:

1. Align the bench with the automatic align bench button as necessary (i.e., multiple ValPro failures). This can be done by selecting Experiment Setup under the Collect tab, then selecting Diagnostics. Choose the Align button to automatically align the bench.
2. The FTIR bench and microscope are properly aligned when the infrared energy has been focused to achieve maximum sensitivity. This is achieved when the max value on the live display is the highest possible level. The live display can be viewed by selecting Experiment Setup under the Collect tab, then selecting Diagnostics.
3. A continuous purge of the FTIR with dry instrument air is recommended at all times to maintain a low humidity environment in the instrument.
4. Preventative maintenance, performed annually, will be performed by a company authorized to service the FTIR. All preventative maintenance, maintenance or repairs will be documented in the instrument maintenance spreadsheet, located on \\vm-fsc-files\Common\Trace\1 - Trace Instrument Maintenance.

5. The instrumentation user's manuals can be found in the drawer under the instrument.
6. There are no calibration records for the FTIR.
7. Spectra can be searched using a reference library to look for potential standards. However, for items other than fibers, a standard must be run under the same operating parameters and compared to the item to be confirmed.

Recommended Report Wording/Interpretation of Test Data:

A comparison is made between the data obtained from the sample and an in-house standard spectra or a spectra obtained from other evidence submitted for analysis. For example, an analyst may be comparing spectra from a paint chip found at a crime scene with a sample of paint from a suspect's vehicle. If the two spectra are consistent, a copy of the in-house standard spectra or other known spectra shall be placed in the case file. Spectra library searches performed utilizing OMNIC software must be confirmed by visual comparison of the spectra to determine if the two spectra are consistent.

Recommended report wording can be found in the protocol for the type of analysis being performed.

TR-07 Polarized Light Microscope (PLM)

Principle of the Protocol:

The Polarized Light Microscope (PLM) allows a trained analyst the ability to observe specific properties with certain types of samples, such as fibers, paint and other microscopic particles. The PLM provides information about samples that are otherwise, not observed in analysis utilizing other instrumentation.

Associated Protocols:

TR-150 Pressure Sensitive Tape Analysis
TR-160 Paint and Polymer Analysis
TR-170 Fiber Analysis

References:

1. California Criminalistics Institute Fiber Identification and Comparison Exercise Manual

Specimen Required (Sample Handling and Preservation):

Microscopic samples of fibers, paint, etc.

Controls:

Stage micrometer (for calibrating eyepiece reticles)

Apparatus and Materials:

1. Leica CFM2 Forensic Comparison Microscope
2. Leica DMLP Polarizing Light Microscope
3. Microscope slides
4. Coverslips
5. Mounting media
6. Forceps
7. Scalpel blades

Individual Steps of Protocol:

1. **Setup**
    a. If needed, perform stage centering, objective centering, and objective calibration with a stage micrometer (If instructions are needed as a reference, they can be found in the California Criminalistics Institute Fiber Identification and Comparison Exercise Manual).

    b. If needed, establish Kohler illumination
        1. Using the coarse/fine focusing knobs, adjust the height of the sample stage until the sample comes into focus.
        2. Adjust the field diaphragm until the leaves are just within the field of view.
        3. Adjust the sub-stage condenser until the leaves come into focus.
        4. Next, center the condenser around the image of the field diaphragm (leaves).
        5. Adjust the iris diaphragm for different amounts of contrast, depending on the sample.

6. Steps 1. and 2. should be performed when changing samples of varying thickness and when changing objectives.

c. Polarizer/Analyzer Orientation

1. Rotate polarizer until the image is all black, to ensure polarizer and analyzer are 90 degrees to each other.

2. **Sample Preparation**
   a. Mount samples on a microscope slide, under a coverslip.
   b. For best optical results, always use a mounting media (such as glycerin, immersion oil, etc.)

Steps 3 through 5 are tests that can be used for sample analysis/comparison. Not all steps are required for analysis/comparison of sample(s). Completion of a step is also not required for analysis/comparison (i.e. if a discrepancy is observed in the Becke Line between known and unknown samples in parallel orientation, it is not necessary for analysis/comparison in the perpendicular orientation.)

3. **Compensator Orientation in Relation to Sample**
   a. First Order Red / Quarter Wave

1. Rotate stage to align the sample (i.e. fiber) perpendicular to First Order Red/Quarter Wave Compensation.

4. **Sign of Elongation**
   a. Obtained through the use of a compensator or Refractive Index (RI) determination.

5. **Refractive Index**
   a. Becke Line Method

1. Place the slide on the stage and focus using 40x or 63x magnification and plane polarized light (no analyzer).
2. Rotate the fiber so the length of the fiber is parallel to the plane of the polarized light.
3. Close down the condenser to prevent the formation of a false Becke Line. A false Becke Line may be observed as a second bright light, which moves in the opposite direction of the Becke Line. This is visible when the difference of the refractive index of the samples and the immersion liquid is low and the true Becke Line is faint.
4. Using the focus knob, adjust the focus while observing the edges of the fiber. When the fiber is slightly out of focus, the Becke Line will appear either inside or outside the fiber edge. <u>The Becke Line will move towards the higher refractive index when the focal distance is increased</u>. Document which medium (fiber or mounting media) has the higher refractive index.
5. Rotate the fiber so it is oriented perpendicular to the plane of polarized light.
6. Focus the edges to determine which medium has the higher refractive index.

   b. Relative RI

1. Compare the relative RI of the unknown to the known, using the Becke Line Method.

Protocol Notes:

1. Covering the microscope, when not in use, will help prevent infiltration of dust.
2. Rotate microscopes objectives by grasping the knurled knob only.
3. Microscope lenses are cleaned during use as needed; this includes condenser lenses, objective lenses and eyepieces. Avoid using abrasive materials for cleaning the lenses.
4. When changing the bulb, avoid touching the glass with bare hands.
5. There is no regularly scheduled maintenance for microscopes. Although any preventative maintenance, repairs or changing of light bulbs, will be documented in the instrument maintenance spreadsheet, located on \\vm-fsc-files\Common\Trace\1 - Trace Instrument Maintenance.
6. The microscope user's manuals can be found at \\vm-fsc-files\Common\Trace\1 - Trace Instrument Maintenance\Microscopes.
7. There are no calibration records for the microscopes.

Recommended Report Wording/Interpretation of Test Data:

The following optical properties may be used in sample comparison:

1. Color - color variances may exist when comparing with different light sources.
2. Delustrant – Examples include dull, semi-dull & bright.
3. Pleochroism – Using plane polarized light, while rotating, observe changes in color.
4. Retardation – Analyst may note either specific values or general (low, medium, high).
5. Refractive Index (RI) – Relative RI will suffice for most examinations.
6. Isotropic vs. Anisotropic – Isotropic has 1 RI whereas Anisotropic has 2 or more.
7. Cross Section – Optical sectioning may be used depending on sample.
8. Diameter – Calculation not required when using a comparison microscope.
9. Extinction – The sample appears black every 90 degrees.
10. Other – Other unique characteristics should be noted when observed.

Recommended report writing can be found in the protocol for the type of analysis being performed.

TR-08 Collection of Trace Evidence

If any work from this protocol is performed outside the Forensic Science Center, the analyst is responsible for ensuring they have access to this protocol. This can be accomplished by taking a printed copy of this protocol, having internet access to this document (https://oklahoma.gov/osbi.html) or having phone access to another analyst that has direct access to this protocol.

Principle of the Protocol:

This protocol outlines the proper procedures for general trace evidence collecting, handling and processing and storage techniques to eliminate contamination and/or cross-contamination concerns.

Associated Protocols:

TR-18 Digital Photography

TR-130 Physical Fit Comparison

Specimen Required:

Evidence such as hairs, fibers, paint, glass and/or other small items may be derived from crime scenes, but may be present on any type of evidence. If the collected specimen consists of hairs, it is preferable for root structure to be intact.

Apparatus and Materials:

1. Forceps
2. Scalpel (disposable or with replaceable blades)
3. Permanent marking pens
4. Lab bench paper (butcher paper, Benchkote®, Benchguard® or equal)
5. Scissors
6. Tape (various types)
7. Clear plastic or polyester sheets
8. Stereomicroscope
9. Microscope slides (various sizes)
10. Cover slips (various sizes)
11. Spatula or note cards (various sizes and types)
12. Petri dishes or boxes (various sizes and types)
13. Camera
14. Plastic bags (various sizes and types)
15. Post-It Notes paper
16. Slides with hole punched adhesive (labels) for storage device

Individual Steps of the Protocol:

1. Items of a size requiring a large area of space for examination (e.g., clothing, bedding, etc.) are examined on examination tables or counter-tops. This area should be equipped with a large, movable light source. In addition, this area should be restrictive to excessive air currents, traffic and static electricity.

2. Before examining the items, the table or counter-top will be cleaned and inspected to make sure there are no extraneous materials (i.e., hairs, fibers, etc.) present.
3. Spread a clean piece of butcher paper or equivalent out on the examination surface and secure with tape if necessary.
4. Before processing the evidence, photograph the condition of the outer packaging, if present. Make notes regarding whether the evidence is sealed properly. Also note anything unusual about the outer packaging.
5. Open the packaging; avoid breakage of the submitting agency's seal, if possible.
6. Remove the evidence and properly label each item of evidence with the laboratory case number, item number, date, and initials. Take digital pictures of the item (refer to Trace Evidence Protocol TR-18 Digital Photography.
7. A visual examination should be conducted at this point. Make further notes describing the evidence. This process may be assisted through the use of magnification and/or alternate light sources. The use of alternate light sources or any type of magnification should be documented in the case file.
8. If visible hairs, fibers, paint and/or glass can be easily removed, **carefully** collect using forceps. *The analyst should use caution in this step to avoid crushing or damaging the evidence with the forceps*.
9. After "Hand-Picking", tape lifting is the preferred method of recovery. Typically, taping **should not** be used to remove paint evidence. For larger or dark colored items on which evidence may be difficult to see, clear tape can be applied sticky side down to the surface of the item to be examined, pressed down, and pulled away. Hairs, fibers, glass and/or other types of evidence will adhere to the adhesive on the tape. The tape should then be secured onto a clear protective surface (i.e., transparency or page protector) for preservation. Tapes should not be stuck on paper or cardboard, which will complicate subsequent removal of the evidence. This process is repeated until the suspected area has been taped. Any given piece of tape should not be overloaded or it will not stick properly to the protective surface. It should be noted that hairs may be difficult to tape and could sustain damage and/or breakage due to taping.
10. An alternate method of taping is 1:1 taping. This is a method that recovers trace evidence without altering distribution. The area of the taping exactly represents the area on the surface that was sampled. This will increase the number of tapings to search, so appropriateness of usage should be evaluated prior to performing this method. The tapings are preserved as described above.
11. For larger items which would make individual removal difficult or where taping would not suffice, scraping, cutting or similar techniques may be used as an alternate method of recovery. Care should be taken to prevent loss and/or contamination of evidence. Suspend the large item above a large piece of clean paper. Press a clean note card or spatula against the article and use a scraping motion to remove any evidence that may be present.

12. A small known sample is cut from the items **after** collection if fiber analysis is to be done. Careful consideration should be taken prior to taking known standards from a damaged area to which a "questioned" fiber may be matched (edge of tear or cut), or an area involved with biological stains.
13. Vacuuming may also be performed on items of evidence to recover trace evidence. Vacuuming tends to collect a large amount of material which makes searching difficult and tedious. Therefore, vacuuming is generally not recommended. If this method is employed, it is recommended that this be performed after the recovery methods described above.
14. After visual examination and various recovery methods are performed, clothing or other items of evidence to be examined are shaken lightly over paper to recover any evidence that may still be adhering. This evidence should be placed into a bindle, petri dish/box, or other size appropriate container.
15. Collected evidence will be properly labeled and stored for further examinations. If the evidence itself cannot be labeled or labeling the item itself could compromise the integrity of the evidence, the proximal container will be labeled. In some instances, the analyst may deem it more beneficial to immediately mount hairs and/or fibers onto microscope slides.
16. When packaging the trace evidence, the size of the packaging should be appropriate for the evidence collected.

Protocol Notes:

1. At all times during the examination, the clothing from any suspect(s) is kept separate from that of the victim(s). Suspect(s) and victim(s) items should ALWAYS be separated by time and/or space when searched. In addition, different lab coats should be worn to prevent cross contamination. Glove changes should be performed frequently especially between articles of evidence, when appropriate. If the integrity of the evidence to be compared is ever compromised, the weight of the results is diminished.
2. If any situation or contact occurs which may contaminate or compromise the evidence in any way, the situation will be documented in the case file and communicated to the technical manager and appropriate actions taken.
3. No more than one article of clothing in an unpackaged state is allowed on any examination table at any one time unless originally packaged together. (An exception to this may be for the purposes of a physical fit examination. Proper collection of trace evidence should be performed prior to this analysis.)
4. If analysis in areas other than trace evidence is required, the examiners should consult before any work is begun. In most instances, trace evidence should be collected prior to other examinations.
5. Unless otherwise noted in the casefile, the date(s) and initials/name of the analyst performing the searching/evidence collection will be documented in photographs/PDF.
6. When collecting evidence off-site from the OSBI Laboratory, handwritten notes documenting the following information must be recorded:
    - Name(s) of analyst collecting evidence & assisting analyst

- Date & OSBI Case # (if assigned)
- Requesting agency & location collection occurred
- Evidence description, where collected from & item # (if assigned)

7. When collecting evidence off-site from the OSBI Laboratory, documentation must be recorded regarding the facilities, necessary accommodations and/or environmental conditions that may have impacted the results.
8. When a search is performed outside the lab and evidence is not collected, the photographs will be submitted as evidence. The BEAST cannot create a case without the submission of evidence.

Recommended Report Wording:

If trace evidence is collected and no instrumental analysis has been performed, the analyst will describe the items collected.

> Item 2 was searched and photographed; possible paint chips/fiber were collected. No further analysis was performed at this time.

If the analyst collecting the evidence is the same person that will be analyzing the evidence, the recommended report writing found in the protocol for the type of analysis being performed will be followed.

If the person collecting the evidence is not the person that will be analyzing the evidence, a narrative report will be generated. This report will include a general description of the items searched as well as an itemized list of what was collected.

If the evidence is collected off-site from the OSBI Laboratory, a report describing the evidence collection needs to be generated; this report will not include the accreditation symbol.

If an analyst searches and photographs evidence and no secondary evidence was collected, the report will reflect the items were searched and photographed. The report will also reflect that no secondary evidence was collected from the original evidence.

> Item 1 was photographed and searched for trace evidence. No secondary items were collected.

If an analyst opens evidence and the evidence cannot be searched due to the condition of the evidence, i.e., covered in mold, the analyst will photograph the evidence for documentation. The report will reflect the item was photographed, but not searched due to the condition of the item and should include a description of the condition, i.e., “moldy condition.”

> Item 2 was photographed, but was not searched due to the moldy condition of the item.

TR-12 Carbon Coating of SEM Specimen

Principle:

The Denton Vacuum DESK V Cold Sputter/Etch Unit with Carbon Evaporation Accessory will deposit an electrically-conductive carbon coating onto the surface of scanning electron microscope (SEM) specimens. For example, if gunshot residue (GSR) evidence is collected on a SEM sample platform ("stub") that is not produced with carbon cohesive material, carbon coating is necessary to prevent "charging" (or scattering of the electron stream) by the specimen at higher voltages when imaged on the SEM.

Associated Protocol(s):

TR-23 Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS)

Specimen Required:

SEM sample platform containing a specimen, either collected on a clear, non-electrically conductive double-sided adhesive tape material or adhesive glue

Apparatus and Materials:

1. The Denton Vacuum DESK V Cold Sputter/Etch Unit with Carbon Evaporation
2. Argon gas, 5.0 PSI maximum
3. Welder's goggles (or equivalent) to permit viewing evaporation process
4. Carbon rods

Individual Steps of Protocol:

1. File and sharpen the carbon rods according to directions in manufacturer's manual.
2. Both carbon rods should be inserted, one after another, through the outside end of the fixed rod holder. First, the pointed rod should be inserted (point trailing) and pushed through with the flattened carbon rod to move into the moving rod holder. Tighten the thumb screw holding the pointed carbon rod. Continue pushing the rods until the flat spring is deflected about one-eighth of an inch. Tighten the thumb screw holding the flattened carbon in the fixed holder.
3. Place the mounted specimens to be carbon-coated onto the chamber pedestal.
4. With the carbon source loaded, place the Pyrex cylinder on the baseplate, seat the source head, and start pump down.
5. Within two to three minutes the chamber vacuum gauge should read about fifty (50) millitorr. Push the ETCH button. An audible click will be heard as the gas solenoid opens. This will allow the chamber to be backfilled with argon gas (5.0 PSI maximum).
6. Push OFF to close the gas solenoid and to allow the chamber to pump down again. Repeat this procedure twice to reduce the level of oxygen in the chamber.
7. With the argon gas on, adjust the needle valve to obtain thirty (30) to fifty (50) millitorr.
8. **Important**! Use the protective goggles to protect the eyes from the intense light produced. Be aware of other individuals nearby that may be exposed to this hazard.

TR-12 Carbon Coating of SEM Specimen

9. Turn on the FILAMENT POWER on the Carbon Evaporation Module. Rotate powerstat clockwise to obtain fifteen (15) amps reading on the ammeter. The carbon point should be bright red and outgassing.
10. Increase filament power to twenty-eight (28) to thirty-two (32) amps until a light spark is seen. As the pointed end of the carbon rod is consumed, a fine mist-like spray of carbon material is deposited on the specimens located within the chamber.
11. **DO NOT** exceed fifty (50) amps on the meter.
12. The carbon-coated specimen is removed from the pedestal. The gold sputter head and Pyrex cylinder should be reinstalled on the baseplate.

Protocol Notes:

1. CAUTION: The intense brightness observed during carbon evaporation is harmful to eyes when viewed directly. Care must be taken not to look directly at the spark created by the carbon evaporation as well as taking the necessary precautions for observers. Welding goggles or a welding glass must be used by the analyst to visually look at the spark.
2. If after carbon coating, the analyst still sees “charging” by the specimen when imaged on the SEM, additional carbon may be applied.

TR-15 Elemental Analysis

Principle:

The scanning electron microscope filament produces a focused beam of high-energy electrons which interact with the specimen. X-ray spectroscopy (by energy dispersion) involves the identification of energy specific wavelengths for elemental analysis of the specimen by segregating these x-rays according to the energy that is given from the interaction.

Associated Protocol(s):

TR-23 Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS)

Specimen Required:

Known and/or unknown samples submitted in casework

Apparatus and Materials:

1. Scanning Electron Microscope (SEM)
2. Energy Dispersive Spectrometer (EDS)
3. Carbon Cohesive Material or Tabs
4. Sample platforms
5. SEM specimen forceps
6. Carbon Planchett

Controls and Standards:

A spectrum of positive and negative controls will be acquired each day casework is performed. These spectra will be archived in the case record.

An elemental standard of carbon will be utilized as a negative control.

An appropriate elemental standard (such as Cobalt) will be utilized as a positive control.

If the known elemental standard is misidentified or the negative control fails,

1. Notify the Supervisor and Technical Manager in charge of trace analysis.
2. Immediately take the instrument out of service.
3. Determine the performance problem, repair, verify instrument performance and document in maintenance log.
4. Do not report any results from the analysis sequence.

Individual Steps of Protocol:

1. Verify the EDS calibration by acquiring a spectrum of a positive and negative controls and archive spectra in the case record.
2. Mount the specimen on an appropriate sample platform and place the mounted specimen in the SEM's chamber.
3. Utilizing the Inca Software, acquire a spectrum of the mounted specimen. This spectrum will be included in the case record.
4. For elements that are confirmed, the characteristic energy x-ray lines will be labeled on the EDS spectra.

TR-15 Elemental Analysis

Protocol Notes:

1. Ensure the vacuum is established after placing or removing samples in the chamber.
2. Before a new lot of carbon cohesive mounting material is put into service, the material must be analyzed for the presence of interfering elements. If the cohesive material is found to contain any inorganic elements, replacement carbon cohesive material must be obtained.
3. Unless otherwise noted in the casefile, the date of sample preparation and initials/name of the analyst performing the sample preparation will be the same date & person documented on the instrumental data.

Recommended Report Wording/Interpretation of Test Data:

1. All reports will indicate what analysis was performed in addition to the results.

   Item 1 was analyzed utilizing the Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS):

2. If the sample was submitted for the analysis of red phosphorus.

   a. If it is positive for phosphorus and trace or lesser amounts of other elements are detected, the trace elements do not have to be reported.

      Item 1 contains the element Phosphorus (P).

   b. If the sample is negative for phosphorus.

      Item 1 was negative for the presence of phosphorous.

3. If the sample contains a large percentage of element(s) (X) and trace (or lesser) amounts of other element(s) (Y):

   Item 1 contains element(s) (X) and lesser amounts of element(s) (Y).

4. If the submitted known and unknown samples are compared and found to have consistent elemental composition:

   Item 1 and Item 2 contain element(s) (X) and trace amounts of element(s) (Y).

   Item 2 may have originated from Item 1 or another item with similar elemental composition.

5. If the submitted known and unknown samples are compared and found to have differing elemental compositions:

   Item 3 contains element(s) (X).

   Item 5 contains element(s) (Y).

   Therefore, Item 3 is not a source of Item 5.

6. If no inorganic elements are identified:

   The elemental composition of Item 1 was found to contain no inorganic elements.

TR-18 Digital Photography

Principle:

This protocol establishes the use of digital images within casework. Digital images are typically considered an extension of the notes.

Specimen Required:

Evidentiary samples (microscopic to macroscopic in size).

Apparatus and Materials:

1. Digital camera

Individual Steps of Protocol:

1. Digital images should be taken on all searching, physical fit, GSR with clothing and comparative cases (paints, tapes, and fibers). Cases not requiring photos include ignitable liquid analysis, elemental analysis, miscellaneous and gunshot residue (GSR) analysis when the only evidence is GSR Kits; photos can be taken of these cases at the discretion of the analyst.
2. For cases that will be documented with digital images:
    a. Container photos: if the container(s) has been compromised, then photos of all sides should be taken. The number of photos should be determined by any case requirements, condition of the container(s) and at the discretion of the analyst.
    b. For each item of evidence, digital images should be taken of the overall evidence, followed by close-up photos of areas of interest.
    c. Digital images may also be acquired using a microscope mounted camera.
    d. Digital images may be annotated to show particular areas of interest.
    e. All digital images should include the case number, date, initials, and item number, if possible. Exceptions would include crime scene images, images taken with microscope or close up images of evidence; this information should be added using a text box in the final PowerPoint.
    f. Images that are taken to document the state in which an item is received should be taken prior to any alteration or testing of the evidence.
3. The digital images may be uploaded individually or compiled into a PowerPoint and organized into files (such as .pdf) for attaching electronically to the case file located in the Laboratory Information Management System (LIMS). Additionally, the size of the individual images may be reduced when high resolution in not required.

TR-18 Digital Photography

Recommended Report Wording:

1. If photography is the only analysis requested:
   Items 1 and 2 were photographed only. No other analysis was performed at this time.
2. If another analysis is performed:
   Items 1 and 2 were photographed, or all items were photographed.

If other analysis is performed, used Recommended Report Wording from the appropriate protocol.

TR-19 Microspectrophotometer Analysis

Principle of the Protocol:

A qualitative and objective method of color analysis and comparison is an integral part of any fiber or paint comparison. Microspectrophotometry (MSP) can be used for this purpose. This protocol deals with the use of MSP where color analysis is required.

Associated Protocol(s):

TR-160 Paint and Polymer Analysis
TR-170 Fiber Analysis

Specimen Required:

1. Fiber
2. Paint
3. Other samples

Apparatus and Materials:

1. CRAIC Technologies 508 PV Microspectrophotometer
2. Mounting Media (i.e., glycerin, Permount, Meltmount, etc.)
3. Microscope slides
4. Microscope coverslips

Controls:

CRAIC Reference Filter Set includes Reference Slide, Holmium Oxide Reference Filter, Didymium Reference Filter, and Neutral Density Filters.

Each day casework is performed, run the Photometric and Wavelength Check. Verify certification check of Filter Set. The results of this will be included in the appropriate case file(s) for the proper documentation of quality controls in cases utilizing Microspectrophotometry.

If Wavelength or Photometric Check fail on any portion, rerun check. If either fails again, re-align the microscope and rerun the checks. Upon continued failing of the wavelength and/or photometric checks, the instrument will be taken out-of-service. The instrument will be repaired by an authorized service provider and the documentation updated in the maintenance log prior to the instrument being placed back in-service.

Note: The Photometric Check is a check of light intensity. The ink spot on the Reference Filter could be blocking some of the light and thus decreasing the intensity of the light through the instrument. If the Photometric Check fails, move the Reference Filter (and the ink spot) away from the scanning area and rerun the Photometric Check.

Individual Steps of Protocol:

1. Turn on the halogen and deuterium lamps using the switches on the front, and then turn on the light housing, which is the green box on top of the microspectrophotometer. Let the lights warm up for 30 minutes before opening up the Lambdafire software
2. Perform the QC:
    a. Open up the Lambdafire software

b. From the Reference Filter Set, remove the quartz slide and place it on the stage
    i. Click on the User Mode tab on the top, and check that UV-Vis is selected
c. Click on the Tools tab at the top, and select Auto-Calibration: Transmission
d. A dialog box will pop up, with several options at the bottom
    i. Select My Folder to confirm the location to save the data files
    ii. Next, select Autoset Optimize
        1. Verify the camera display is on- check that the metal rod on the right of the eyepieces is pulled out
    iii. Ensure the aperture (black box) is focused on a clean area of the slide, then select Dark and Reference
    iv. Next, select Wavelength Check
        1. A small dialog box will pop up, with instructions on which filters to measure
            a. The filters should be placed with the words facing up, on top of the lens under the stage
            b. The filters should be positioned so the light can pass through the central glass unobstructed
        2. After all of the measurements are completed, a dialog box will pop up with the check results
            a. Select Save and Print Results
    v. Lastly, select Photometric Check
        1. A small dialog box will pop up, with instructions on which filters to measure
            a. The filters should be placed with the words facing up, on top of the lens under the stage
            b. The filters should be positioned so the light can pass through the central glass unobstructed
        2. After all of the measurements are completed, a dialog box will pop up with the check results
            a. Select Save and Print Results

3. Sample analysis using transmission
    a. Check the User Mode tab to check that Glass is selected
    b. Use the buttons at the bottom of the screen
    c. Select My Folder to confirm the location of the data files
        i. Make sure in the bottom left corner the Light Source: TL and TDUV are selected
    d. Select Auto Optimize
        i. Make sure the camera display is on- check that the metal rod on the right of the eyepieces is pulled out
    e. Move the aperture (black box) and focus on a clean area of the slide next to the sample, then select Dark and Reference
    f. Move the aperture onto the sample, then select Collect Spectrum
        i. A dialog box will pop up

TR-19 Microspectrophotometer Analysis

1. Check that Absorbance is selected
2. Check that the correct objective is selected
3. Edit the sample name
4. Collect the sample by selecting Scan Image
    a. To record the location on the sample, select Scan and Take Image
5. Repeat steps 1-4, moving the aperture along the sample in different areas until a minimum of 10 spectra have been obtained. More can be collected if necessary.

4. Sample analysis using fluorescence
    a. Make sure the mercury light source is turned on for at least 15-20 minutes before use. On the halogen light source, the right switch should be set to TL (transmitted light)
    b. Open Set Parameters to set up following options for each spectrometer for data collection
        i. Wavelength Range
        ii. Number of Scans
        iii. Resolution Factor
    c. Check the User Mode tab to check that Fluorescence is selected
    d. Use the buttons at the bottom of the screen
    e. Select My Folder to confirm the location of the data files
        i. Verify that Transmission light source (TL) is selected.
    f. Move aperture to desired sampling area
    g. Press Auto-Set Optimize for the best integration time (in milliseconds)
        i. If successful, a happy face message will appear.
        ii. If unsuccessful, a frowning face message will appear, indicating that it may be necessary to re-check alignment or adjust light intensity.

    At times, it may be necessary to manually set the integration time, instead of using Auto-Set Optimize. This can be typical for low-level fluorescence samples. Use Realtime Mode to help chose an integration time.

        1. The best integration time is 10 ms, which can be manually set by selecting the gears icon.

    h. Focus on sample.
    It may be helpful to 1st focus the specimen in transmission prior to engaging the fluorescence filter as low intensity Fluorescence may require longer exposure times on the camera that make focusing difficult.
        i. The 40x objective will focus the fluorescence signal better.
    i. Verify desired excitation cube is selected using the filter turret below the oculars. The excitation cubes correspond to the numbers 2, 3, and 4, and the wavelengths 365 nm, 420 nm, and 546 nm respectively.
    j. Open aperture and field diaphragm entirely, along with any diffusers.
    k. Select the camera icon, and adjust the exposure and gain settings to improve the image. The fiber should be visible.

l. On the base of the microscope, the wheel should be set to the circle to block out any transmitted light.
m. Press Collect Dark and Reference Scan
    i. The aperture should be focused on the fiber.
    ii. The system will sample the electronic (dark) noise of the system and reference scan.
    iii. As the reference scan will not be used in emission calculations, it will provide a preview of the collected Fluorescence spectra.
n. Press Collect Sample
    i. The reference and sample can be collected in the same area.
o. An additional window will appear at this time to select the following:
    i. Type of measurement desired (i.e., transmission, reflectance, fluorescence, etc.)
        1. Emission should be selected
    ii. Objective magnification
        1. Select the objective used
    iii. Name of spectral file
        1. Include the excitation filter used
p. Click Save Spectrum and Image, repeat as necessary for additional sampling areas

Repeat measurements will automatically increment file names by +1.

For both transmission and fluorescence, the analyst will directly compare the results from two separate samples (i.e., known and questioned) to determine if they are consistent.

Protocol Notes:

1. Analysis using transmission can be used on paints, fibers and tapes. Fluorescence can be used on fiber and paint samples
2. Samples with no color (i.e., clear coat in paint samples), white and black samples are not appropriate for MSP analysis. MSP analysis may render little information with darker fibers, i.e., dark brown fibers.
3. The Wavelength check verifies that peaks are being detected at the correct wavelengths on particular known filters. The Photometric check verifies that the peaks are at the correct intensity.
4. The Neutral Density filters are coated with a film that is very fragile. Care should be taken to avoid scratching the film on these filters. Therefore, these filters should never be wiped to clean them. If they become soiled, it will be necessary to replace them. Also, when placing the neutral density filters on the field diaphragm ensure that the filter's edge is parallel to the front of the microscope. This measurement is sensitive to position of the filter.
5. Once measurements are taken of the sample especially in fluorescence mode, the sample should be moved out of the path of the light to protect the sample from degradation.
6. There is no regularly scheduled maintenance for the MSP. Although any preventative maintenance, repairs or changing of light bulbs, will be documented in the instrument

maintenance spreadsheet, located on \\vm-fsc-files\Common\Trace\1 - Trace Instrument Maintenance.

7. The MSP user's manuals can be found in the drawer next to the instrument.
8. The certification records can be found in the maintenance spreadsheet or near the instrument. There are no calibration records for the MSP.

Recommended Report Wording/Interpretation of Test Data:

Recommended report wording can be found in the protocol for the type of analysis being performed.

TR-23 Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS)

Principle of the Protocol:

Scanning Electron Microscopy with Energy Dispersive Spectrometry (SEM/EDS) is a nondestructive technique that allows the examiner to obtain magnified images of samples and elemental information based upon the interactions of the sample with the electron beam created by the SEM. The secondary and backscatter electrons that are generated by the sample are detected by the SEM and make up the images that can be viewed. The X-rays that are generated by the sample are detected by the EDS and processed for elemental composition.

The Scanning Electron Microscope (SEM) can accurately measure the size of mounted specimens and is a function of the viewed magnification versus the working distance (WD). On the SEM's monitor, a "micron bar" is displayed as well as the associated metric measurement. However, this laboratory does not reflect any size measurements in the examination reports.

The Oxford Inca Energy Dispersive Spectrometer (EDS), integrated with the Tescan VEGA3 Scanning Electron Microscope (SEM), can qualitatively or semi-quantitatively identify the elements (by their unique energy signatures) contained within mounted specimens. Quantitative results are not reported by this laboratory.

References:

1. VEGA3 SEM Instruction for Use Manual, Tescan

Associated Protocol(s):

TR-12 Carbon Coating of SEM Specimens
TR-15 Elemental Analysis
TR-110 Primer Gunshot Residue Analysis by SEM/EDS

Specimen Required:

1. Paint
2. GSR
3. Unknown substances or other samples

Apparatus and Materials:

1. Tescan VEGA3 Scanning Electron Microscope (SEM)
2. Oxford Energy Dispersive Spectrometer (EDS)
3. Elemental Standards (Nickel, Cobalt, Rhodium, or others as needed)
4. MRS-3 Magnification Reference Standard & Stage Micrometer
5. Stereomicroscope
6. Sample Preparation tools: scalpel, forceps, tungsten probes, rollers, etc.
7. Adhesive such as Duro-Tak©, Carbon Planchette, Carbon Cohesive Tabs, Aluminum SEM Stubs

Controls and Standards:

A spectrum of positive and negative controls will be acquired each day casework is performed. These spectra will be archived in the case record.

An elemental standard of carbon will be utilized as a negative control.

TR-23 Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS)

An appropriate elemental standard (such as Cobalt) will be utilized as a positive control.

If the known elemental standard is misidentified or the negative control fails,

- Determine the performance problem.
  - If needed, make appropriate adjustments and verify instrument performance.
  - If repairs are necessary, make repairs, verify instrument performance, and document in maintenance log.
- Do not report any results from the analysis sequence.

Any instrument removed from service will be documented in the maintenance log and reported immediately to the Supervisor and Technical Manager.

Individual Steps of Protocols:

1. **Sample Preparation**

   Samples are mounted onto sample stubs using conductive adhesive material or placing on a carbon planchette. A stereomicroscope may be used when mounting samples. Microtomy, polishing or other suitable means may be used to ensure the sample surface is flat.

2. **SEM/EDS Operating Condition and Parameters**

   The working distance should be fixed at 15mm. If used for imaging only, this value may be adjusted. Other conditions may also be adjusted as necessary. The beam voltage may be adjusted for element specific analysis. Light elements may require lower voltage and heavy elements may require higher voltage. 20kV should be used as a starting point for most examinations.

   New filaments should be "seasoned" to obtain maximum lifetime. When heating a filament for the first time, ensure the new filament box is selected. A new filament should be heated by using the steps listed in the VEGA3 manual as a guide.

   The Degauss feature should be used as needed. Using Degauss "clears" the noise in the electromagnetic lenses.

   Focus wobble and stigmation should also be checked and adjusted.

3. **Sample Analysis**
   a. Verify the EDS calibration by acquiring the spectra of an appropriate elemental standard (archive spectra in the case record.)
   b. Manually confirm elements.
   c. Take precautions to ensure the interaction volume does not comingle with nearby samples.

Protocol Notes:

1. A filament generally lasts approximately 500 hours or longer. The filament is replaced when it burns out or when it is getting close to burning out.
2. When the INCA control boxes need to be reset due to software hang-ups, etc., initiate the 'tidy up' software located on the desktop of the EDS computer. Follow the directions listed on the monitor. Note: Inca analysis software must be closed any time another Inca software program such as 'tidy up' is in use.

TR-23 Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS)

3. Preventative maintenance, performed annually, will be performed by a company authorized to service the SEM/EDS. All preventative maintenance, maintenance or repairs will be documented in the instrument maintenance spreadsheet, located on \\vm-fsc-files\Common\Trace\1 - Trace Instrument Maintenance.
4. The SEM/EDS user's manuals can be found on the instrument in the "Help" screens.
5. There are no calibration records for the SEM/EDS

Recommended Report Wording / Interpretation of Test Data:

1. An element is identified when:
    a. The peak(s) has the characteristic energy for that element.
    b. A peak is statistically significant (only peaks which are statistically significant should be considered for identification). The minimum size of the peak (P) should be three times the standard deviation of the background at the peak position. ($P>3(N_B)^{1/2}$). This peak height can be estimated directly on the EDS display from the statistical scatter in the background SUBTRACTION on either side of the peak. The "thickness" of the background trace due to statistical fluctuations in the counts is a measure of $(N_B)^{1/2}$. The peak, then, should be approximately three times this thickness. If it is difficult because of statistical fluctuations in the count to decide whether a peak exists above the continuum, then more counts should be accumulated. Any calculations performed should be included in the notes in the case record.
2. The vertical (counts) scale should be present in the spectrum and can be adjusted to reveal detail.
3. The presence of an element can be identified only when a distinctive, unique set of lines is produced or when a single peak occurs at an energy where it cannot be mistaken for another element.
4. If an automatic identification application is used, peak identification must be confirmed by the analyst.
5. For documentation, spectra must be displayed on a scale that clearly demonstrates the peaks identified; an overall spectrum to 20 keV should be included. Multiple scales may be necessary.

Recommended report wording can be found in the protocol for the type of analysis being performed.

TR-100 Analysis of Ignitable Liquids

Principle of Protocol:

Liquids or residues which fit into the classifications of ignitable liquids are routinely identified by the laboratory through gas chromatography / mass selective detector (GC/MSD) instrumentation. A comparison of the chromatograms of known standards and a questioned sample is performed. This analysis can only be accomplished once the analyst has established an internal library of ignitable liquid standards, common pyrolysis products and miscellaneous liquids encountered in the samples routinely submitted. Because many products have the same or similar formulations, ignitable liquid analysis is able to provide information only about the range and class of the ignitable liquid such as heavy petroleum distillate, light aromatic, medium isoparrafinic, etc. Individualized information such as brand name, if the ignitable liquid came from a specific bottle, or if two samples came from a single source cannot be determined from this analysis. Ignitable liquid analysis is able to distinguish gasoline from other medium aromatic compounds, but no information can be provided on where the gasoline came from (i.e., Texaco, 7-11, Shell, etc.) or if two samples came from the same source.

References:

American Society for Testing and Materials (ASTM), E1618, Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.

Associated Protocols:

TR-01 Gas Chromatograph Mass Spectrometer for Trace Analysis
TR-101 Ignitable Liquid Sampling Apparatus

Specimen(s) Required:

Fire debris and/or other appropriate evidence samples.

Solvents:

1. Carbon Disulfide, 99+% or other appropriate solvent

Apparatus and Materials:

1. Gas chromatograph/mass selective detector (GC/MSD) and appropriate accessories
2. Sampling apparatus (see associated protocol)
3. Hewlett Packard HP-5 column (30-meter length x 0.25 millimeter x 0.25 micrometer film thickness, cross-linked 5% Phenyl Methyl Silicone (or equivalent)
4. Clean unused metal (lined or unlined) cans with double seal, friction-fit lids (various sizes)
5. Nylon Heat Seal Arson Evidence Collection Bags

Standards and Controls:

1. Ignitable liquids consist of all brands that fit within the classification system established by ASTM, E1618. Some examples include, but are not limited to: Hydrocarbons C6 to C30, toluene, 3-ethyltoluene, 1,2,4-trimethylbenzene, naphthalene, gasoline, camping fuel, charcoal starter, kerosene, diesel, etc. Included in this list are all chemicals that will be used to identify a class of ignitable liquids. These may be purchased locally as well as from laboratory or

chemical supplier/manufacturers.

2. Standards/chemicals used in the identification of a class of ignitable liquids must have retention time differences which do not exceed plus or minus 2% of the sample and must be analyzed on the column the sample was analyzed on. If the column is cut or changed, the standards/chemicals must be reanalyzed on the column before they can be used in casework.
3. The supplies and materials used in the analysis of ignitable liquids must be checked prior to use. All materials and solvents must be free of contaminants that may interfere with analysis.
4. The positive control will be a standard accelerant mixture (SAM). If the SAM is created in-house, it is recommended the SAM be a 1:1:1 light petroleum distillate:gasoline:heavy petroleum distillate mixture. Alternatively, a commercially prepared ignitable liquid test mixture can be obtained from Cerilliant.
    a. The SAM must be analyzed as a liquid sample if the case(s) being analyzed contain liquid samples.
    b. The SAM must be analyzed as a heated sample (utilizing a carbon strip) if the case(s) being analyzed contain samples which were heated in the oven. The SAM must be heated at the same time as the case(s).
    c. If samples are being re-analyzed on a day following the original instrumental analysis, a liquid SAM can be used as a positive control for that day. The original SAM must be included in the casefile to demonstrate the analysis and instrument were working properly on the date of original analysis.
5. The expected results for the positive control are as follows
    a. If the SAM is created in house, the following components must be in the correct ratio and identified:
        1. Two hydrocarbons from the light petroleum distillate
        2. The following eight compounds from gasoline: ethylbenzene, p/m-xylene, o-xylene, propylbenzene, 3-ethyltoluene, mesitylene, 2-ethyltoluene, 1,2,4-trimethylbenzene
        3. Five contiguous hydrocarbons from the heavy petroleum distillate
    b. If a commercially prepared ignitable liquid test mixture is used, each component in the mixture will be identified.
6. Carbon Disulfide (or other appropriate solvent) is the solvent used as the blank. The blank will be free of ignitable liquid contamination.

    A daily blank will be run before casework, typically before the SAM. A blank will be run before each case using the same method used for case samples. A blank will also be run after fourth injection in a case.
7. If the expected results from the positive control or blank are not acquired:
    a. Notify the Supervisor and Technical Manager of trace analysis.
    b. Determine the performance problem, repair, verify instrument performance and document in maintenance log.

c. Do not report any results from the analysis sequence.

8. A system blank will be prepared and analyzed each time samples are heated in an oven. The system blank should be placed into the oven along with the evidence containers and heated for the same amount of time utilized on the evidence. A system blank is a lined can/nylon bag/etc. containing a carbon strip; the system blank will be made from the same type of container(s) that is used to contain the evidence being analyzed.
9. If an ignitable liquid is suspected in the system blank, the following actions will be taken:
    a. Notify the Supervisor and Technical Manager of trace analysis.
    b. The results of the analysis will be considered suspect. The results may be reported but the analyst must consult with the Technical Manager of trace analysis.
10. The positive control, blanks and system blank will be archived in the casefile. The autotune will also be archived in the casefile (see TR-01).

Approved Methods:

The current approved methods being utilized for ignitable liquids are the IL20 method, the LIGHT20 method, and the SOLVENT method.

1. The IL20 method is the method that will be utilized unless the analyst has reason to be suspect the presence of a compound that is smaller than C7. The parameters for the IL20 method are:
    a. Initial temperature: 50° C
    b. Initial time: 2.50 min
    c. Final time: 2.20 min
    d. Total run time: 18.50 min
    e. Transfer line: 300° C
    f. Ramp rate: 7° C/min to 100° C, 30° C/min to 300° C
    g. Injection volume: 1.0 µL
    h. Split ratio 20:1
    i. Injection port temperature: 300° C
    j. Solvent Delay: 1.75 min

2. The LIGHT20 method would be used in cases where the analyst suspects a compound lighter than C7 and the analyst has diluted the sample before analysis. The parameters for the LIGHT20 method are:
    a. Initial temperature: 50° C
    b. Initial time: 2.50 min
    c. Final time: 2.20 min
    d. Total run time: 18.50 min
    e. Transfer line: 300° C
    f. Ramp rate: 7° C/min to 100° C, 30° C/min to 300° C
    g. Injection volume: 1.0 µL
    h. Split ratio 20:1
    i. Injection port temperature: 300° C
    j. Solvent Delay: 0.00 min

3. The SOLVENT method will only be used if the analyst is sampling a liquid suspected of being smaller than C7 and no dilution will be made. The parameters for the Solvent method are:
    a. Initial temperature: 50° C
    b. Initial time: 1.00 min
    c. Final time: 0.00 min
    d. Total run time: 3.50 min
    e. Transfer line: 290° C
    f. Ramp rate: 20° C/min to 100° C
    g. Injection volume: 0.10 µL
    h. Split ratio 500:1
    i. Injection port temperature: 290° C
    j. Solvent delay: 0.00 min

Individual Steps of Protocol:

Sampling using a carbon-strip:

1. Open the evidence container and make observations and notes of the enclosed fire debris. The analyst may open and inspect the evidence container either before or after the container is heated.
2. Close the evidence container (tightly replace the lid if evidence container is a can).
3. Insert the sampling apparatus. Avoid contact between the c-strips and the fire debris.
4. Prepare a system blank by placing a sampling apparatus into a similar evidence container. If a can is used for the evidence, a can should be used as the container or if a nylon bag is utilized for the evidence, a nylon bag should be used as the container for the system blank).
5. Place the system blank and the evidence in the oven for sixteen hours at 60°C +/- 5°C for most routine samples. At the analyst's discretion and depending upon the type of evidence encountered, the length of time in the oven may be less.
6. Place the c-strips from the evidence into a vial, add solvent and seal with a cap.
7. Place the c-strip from the system blank into a vial, add approximately 1 ml of solvent and seal with a cap.
8. Prepare a blank utilizing the same solvent as used to prepare the sample.
9. The sample, system blank, and blank are now ready for injection on the GC/MSD.
10. After analysis, the vial containing the carbon-strip will be returned to the submitting agency with the evidence.

TR-100 Analysis of Ignitable Liquids

Sampling using the solvent wash method:

1. Open the evidence container and make observations and notes of the enclosed fire debris.
2. Pour the solvent (i.e.: carbon disulfide, pentane or hexane) over the fire debris within a laboratory fume hood. The amount of solvent will depend on the size of the sample and the size of the container.
3. Swirl the solvent over and around the fire debris for desired interval of time.
4. Filter the solute through a funnel containing filter paper media.
5. Collect the liquid in a clean glass beaker.
6. Allow the liquid to evaporate to a volume necessary to fill an autosampler vial (~1 to 2 milliliters). This will concentrate any collected or dissolved ignitable liquids.
7. Place the remaining liquid into a sample vial and seal with a cap.
8. Prepare a blank utilizing the same solvent as used to prepare the sample.
9. The sample and blank are now ready for injection on the GC/MSD.

Sampling liquid evidence

1. Open the evidence container and make observations and notes of the contents.
2. Remove an aliquot of the unknown liquid and place into a properly labeled sample vial. This aliquot can be diluted with a solvent, if needed, so that an acceptable chromatogram can be obtained (approximately 1:1000 dilution, sample: solvent).
3. Properly cap the vial.
4. Prepare a blank utilizing the same solvent as used to prepare the sample. If no solvent was used to prepare the sample, a blank of carbon disulfide will be used.
5. The sample and blank are now ready for injection on the GC/MSD.

Performing the flame test on a liquid sample:

1. A small amount of the liquid should be poured into a watch glass, drawn into a glass pipette, or collected into other reservoir.
2. A heat source is then used to attempt ignition. A flame should be observed to report a positive flame test.

Gas Chromatography/Mass Spectrometer

1. Using the above prepped blanks, SAM and samples place the samples in the sample tray.
2. Enter appropriate vial information into the Chemstation sample log table, including case number, sample item number, method to be used for analysis, data file name, analyst's name, and vial barcode.
3. Inject a portion of the sample onto the GCMS utilizing one of the approved methods for ignitable liquid analysis.

Sampling refers to testing a portion of the evidence and is not a statistical sampling method.

TR-100 Analysis of Ignitable Liquids

Evaluation Criteria

The following data/information are required for evaluation and classification and must be included in the casefile:

Class	Light (C4-C9)	Medium (C8-C13)	Heavy (C9-C20+)
Gasoline	Ethylbenzene, m/p-xylenes, o-xylenes, propylbenzene, 3-ethyltoluene, mesitylene, 2-ethyltoluene, 1,2,4-trimethylbenzene		
Petroleum Distillate	All hydrocarbons identifiable, in addition to 2-3 other peaks	5 hydrocarbons in a row or as many as are identifiable (bell curve should be observed)	At least 5 hydrocarbons in a row or as many as are identifiable (bell curve should be observed)
Isoparaffinic Product (branched chain aliphatic compounds)	5 branched chain aliphatics or as many as identifiable	5 branched chain aliphatics or as many as identifiable	5 branched chain aliphatics or as many as identifiable
Aromatic	5 aromatic compounds or as many which are identifiable	5 aromatic compounds or as many which are identifiable	5 aromatic compounds or as many which are identifiable
Naphthenic-Paraffinic (branched chains or cyclic alkanes)	All branched chains or cyclic alkanes	5 branched chains or cyclic alkanes, or as many as identifiable	5 branched chains or cyclic alkanes, or as many as identifiable
Normal Alkanes	All identifiable hydrocarbons	All identifiable hydrocarbons	All identifiable hydrocarbons
Oxygenated (the major oxygenated compounds should be in large excess-at least 10x the matrix peaks)	All major oxygenated compounds	All major oxygenated compounds	All major oxygenated compounds
Other-Misc.	Single component – mass spec hit to library created on instrument and retention time match. Other – 3 to 5 compounds	Terpenes-3 or more identified with hit to library created on instrument. Other – 3 to 5 compounds	At least 5 compounds

A reference standard which has the same compounds at approximately the same relative concentration/abundance as the unknown will be included in the casefile. The reference standard should include the same mass spectral data as listed above. The reference standard must be run on the same instrument and using similar conditions.

The chromatographic pattern is very important. The GC pattern of the unknown and standard should be similar. In comparing the two, the mass spectral data, at the peaks with corresponding retention times, should match. There may be times when a mass spectral hit/match is made to NIST or another library, this will give the analyst information if a branched chain aliphatic or straight chain hydrocarbon is present; this will be sufficient for mass spectral data comparison.

Styrene – when styrene is at a concentration significantly higher than toluene or xylenes, the finding of toluene or xylenes are not of significance as they are by-products of styrene.

Protocol Notes:

1. 'Ignitable liquid residue' may be used where appropriate. For example, when identifying minute amounts of gasoline in debris, it could be desired to use 'ignitable liquid residue' instead of 'ignitable liquid'. This is because, when looking at the debris, the gasoline may not be present in a visible liquid form, just as a residue.
2. A flame test may be performed whenever additional data is needed (such as when a good GC library match does not exist) to identify an ignitable liquid.
3. In some cases, the analyst may encounter compounds referred to as terpenes. Terpenes are compounds which are naturally occurring in some types of woods. Terpenes are also found in turpentines. This lab does not determine whether the terpenes are due to wood or turpentine. (See *Recommended Report Wording* below)
4. Samples containing soil and/or vegetation should be analyzed promptly, as microbial degradation can degrade ignitable liquids that may be present in the sample.
5. Microbial degradation of gasoline has been known to occur in certain samples containing bacteria, such as soil and vegetation. If microbial degradation is suspected, an ignitable liquid cannot be conclusively identified.
6. Unless otherwise noted, the date of sample preparation will begin on the date the analyst assigns the case to themselves in the BEAST. Also unless noted, the assigned analyst will be the analyst performing the sample preparation.
7. The analyst's name and date(s) of instrumental analysis of samples and standards will be documented on instrumental data. It is not required for the sample and standard to be analyzed by the same analyst.
8. If a carbon strip is used in the analysis, the sample vial containing the carbon strip will be closed with a crimp top cap, placed in a plastic bag and returned to the container. The sample vial will be added to the inventory in the BEAST as a sub-item of the container; the container will be labeled with the item number of the container and sub-item number of the sample vial.
9. Documentation to be uploaded into the BEAST casefile will include (as applicable):
    - Daily Autotune and Tune Evaluation
    - Daily carbon disulfide blank
    - SAM (liquid, heated or both)
    - System blank
    - Carbon disulfide blank for the case and between case samples
    - Sample data
    - Standard(s) data

TR-100 Analysis of Ignitable Liquids

Recommended Report Wording/Interpretation of Test Data:

The current revision of E1618 (See Reference Section of this protocol) will be used as a guide for data interpretation.

1. All reports will indicate what analysis was performed in addition to the results. For example:

   Item 1 was analyzed using Gas Chromatography/Mass Spectrometry (GC/MS)

2. If the sample vial containing the carbon strip extract is retained in the evidence container, wording will be added to the results to indicate it was retained. For example:

   The evidence, including the sample used in analysis, will be returned to the submitting agency.

3. If an ignitable liquid (IL) is identified: (*Within the listed classes*...). List the class and range if available and give examples where appropriate.

   Item 1 was analyzed using Gas Chromatography/Mass Spectrometry (GC/MS). This sample contains an ignitable liquid in the medium isoparaffinic class. Examples of products in the medium isoparaffinic class include some charcoal starters, some paint thinners, and some copier toners.

   Item 1 was analyzed using Gas Chromatography/Mass Spectrometry (GC/MS). This sample contains an ignitable liquid in the light petroleum distillate class. Examples of products in the light petroleum distillate class include some cigarette lighter fluids and some camping fuels.

   Item 1 was analyzed using Gas Chromatography/Mass Spectrometry (GC/MS). This sample contains an ignitable liquid in the gasoline class.

4. If no ignitable liquids are identified:

   Item 1 was analyzed using Gas Chromatography/Mass Spectrometry (GC/MS). No ignitable liquids were detected in this sample.

   Item 1 was analyzed using Gas Chromatography/Mass Spectrometry (GC/MS). No ignitable liquids were identified. It should be noted that ignitable liquids may evaporate or can be totally consumed during a fire. A negative finding of ignitable liquids does not preclude its presence during a fire.

   Item 1 was analyzed using Gas Chromatography/Mass Spectrometry (GC/MS). No ignitable liquids were identified. This does not preclude the possibility that ignitable liquids were present.

5. If single solvent peaks are identified:

   Item 1 was analyzed using Gas Chromatography/Mass Spectrometry (GC/MS). This sample contains an ignitable liquid identified as toluene.

6. If a mixture of ignitable liquids is identified:

   Item 1 was analyzed using Gas Chromatography/Mass Spectrometry (GC/MS). This sample contains a mixture of an ignitable liquid in the gasoline class and an ignitable liquid in the heavy petroleum distillate class. Examples of products in the heavy petroleum distillate class include kerosene, diesel fuel, some jet fuels, and some charcoal starters.

TR-100 Analysis of Ignitable Liquids

7. If the examination is inconclusive for an ignitable liquid:

   Item 1 was analyzed using Gas Chromatography/Mass Spectrometry (GC/MS). Although this sample has a chemical profile associated with a light petroleum distillate, no comparison standard could be located. Therefore, this sample is considered inconclusive for the presence of a light petroleum distillate. Examples of products in the light petroleum distillate class include some cigarette lighter fluids and some camping fuels.

8. If terpenes are identified:

   Item 1 was analyzed using Gas Chromatography/Mass Spectrometry (GC/MS). This sample contains an ignitable liquid (in the miscellaneous class) known as terpenes, which are naturally occurring in some types of wood. An example of a product containing terpenes is turpentine.

9. If a positive flame test is performed but no instrumental data exists to identify the liquid:

   Item 1 was analyzed using a flame test and Gas Chromatography/Mass Spectrometry (GC/MS). This sample was flammable based on a flame test; however, the type of ignitable liquid was unable to be identified by instrumental analysis.

10. If microbial degradation is suspected and there are indications that gasoline is present in the sample:

    Item 1 was analyzed using a flame test and Gas Chromatography/Mass Spectrometry (GC/MS). This item has a chemical profile associated with gasoline that has undergone microbial degradation. Microbial degradation has been known to occur in certain samples containing bacteria, such as soil and vegetation. Not all chemical components are present in sufficient amounts for confirmation of gasoline. Therefore, this sample is considered inconclusive for the presence of gasoline.

TR-101 Ignitable Liquid Sampling Apparatus

Principle of the Protocol:

This protocol describes how to assemble the carbon strip apparatus used in fire debris sampling techniques.

Associated Protocols:

TR-1 Gas Chromatograph Mass Spectrometer for Trace Analysis

TR-100 Analysis of Ignitable Liquid Residues

References:

1. Dietz W.R., Pro-Tek Charcoal Strips for Accelerant Recovery. Paper provided by the Bureau of Alcohol, Tobacco and Firearms, from the Arson Ignitable Liquids Detection Course.
2. James R., Arson Debris Vapor Sampling System. ATF Laboratory Interface. 1984 Summer, 1,1:3.

Controls:

The supplies and materials used in the analysis of ignitable liquids must be checked prior to use.

All materials must be free of contaminants which may interfere with analysis.

If materials are not free from contaminants, test results cannot be reported.

Apparatus and Materials:

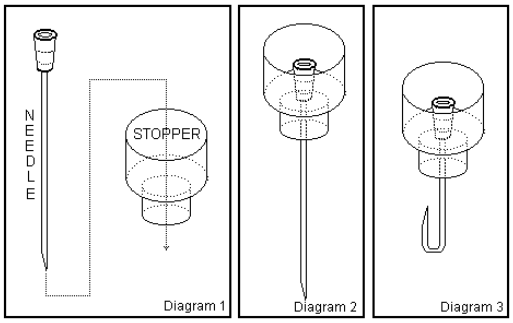
1. C-Strips (Albrayco, Part No: ACS-150) or equivalent
2. Sterile needles
3. Natural red rubber stoppers
4. Vacuum desiccator for storage of supplies and tools.
5. Needle-nosed pliers
6. Forceps
7. Paper clips
8. Magnets

Individual Steps of Protocol:

How to assemble the c-strip sampling apparatus using red stoppers:

1. Insert one needle through one red stopper (see *Diagram 2*).
2. Insert the needle through one c-strip.
3. Bend the needle into a hook-shape with a pair of needle-nosed pliers (see *Diagram 3*).
4. The c-strip hangs on the now-formed “fish-hook.”
5. The sampling apparatus is now ready for use and should be stored in a manner to avoid contamination.

TR-101 Ignitable Liquid Sampling Apparatus



How to assemble the c-strip sampling apparatus using paperclip and magnet:

1. Bend the paperclip into a hook-shape.
2. The c-strip hangs on the now-formed "hook."
3. The magnet attaches to the outside of the evidence can lid, while holding the paperclip on the inside.

Sampling refers to testing a portion of the evidence and is not a statistical sampling method.

Proper handling of carbon-strips (c-strips):

1. The analyst must remember that the c-strips are a highly adsorptive medium for volatile compounds and chemicals. The analyst is charged with the responsibility that the integrity of the c-strips is not compromised.
2. Prior to use, quality control and quality assurance steps must be performed. This is done by placing a c-strip (picked at random from the lot) into an autosampler vial, filling the vial with appropriate solvent, capping and injecting the solution on the GC/MSD. If the chromatogram is of a satisfactory result, the strips may be used.
3. The c-strips will be stored in an area where they will not be subjected to contaminants. This is best accomplished by placing the c-strips in a clean glass jar with a screw-lid. Do not handle the c-strips with one's hands. Handling should be done by the use of clean forceps. The jar (and loosely attached lid) with the c-strips is placed in a vacuum desiccator at room temperature and under vacuum to prevent any possible contamination. The tools used to assemble the sampling apparatus should be stored in the same desiccator.

Protocol Notes:

1. This protocol does not cover all available sampling methods. The type of sampling apparatus employed is at the discretion of the analyst depending on the form of evidence.
2. Any sampling component will be checked for function and purity prior to being put into service.

TR-110 Primer Gunshot Residue Analysis by SEM/EDS

Principle of The Protocol:

Primer gunshot residue (GSR) particles can be detected and identified based on morphology (shape) and elemental composition using a Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS). This method of GSR analysis provides the most definitive identification of GSR particles currently available.

Associated Protocols:

TR-23 Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS)

Specimen(s) Required:

OSBI Gunshot Residue evidence collection kit or other properly collected sample on a SEM sample platform (referred to as a "stub").

Apparatus and Materials:

1. Scanning Electron Microscope (SEM)
2. Energy Dispersive Spectrometer (EDS)
3. Elemental standard (Cobalt/Rhodium or other elements as required)
4. Positive GSR Sample
5. Blank environmental stub
6. SEM stub forceps

Standards and Controls:

1. The SEM stub forceps should be rinsed with deionized water between handling the sample sets and environmental blank controls.
2. If GSR particles are detected on the environmental blank, the following actions will be taken:
    a. Notify the Supervisor and Technical Manager in charge of trace analysis.
    b. The results of the analysis will be considered suspect. The results may be reported but a qualifying statement must be included in the report for each case analyzed during that run.
    c. Thoroughly clean the SEM chamber and analyze a new blank stub to verify that the chamber and surrounding areas are free from any suspect particles.
    d. If GSR particles are detected on the new blank stub
        1. Instrument will immediately be taken out of service
        2. Determine the performance problem, repair, verify instrument performance, and document in the maintenance log.
3. If at least one particle from the positive GSR sample cannot be manually confirmed,
    a. Notify the Supervisor and Technical Manager in charge of trace analysis.
    b. Immediately take the instrument out of service
    c. Determine the performance problem, repair, verify instrument performance and document in maintenance log.

d. Do not report any results from the analysis sequence.

Individual Steps of Protocol:

1. The underside of each stub should be labeled with the case and item number if the stub does not have a unique identifier provided by the manufacturer. Where a unique identifier is present, this number will be recorded in the case notes. The orientation of the stub will be marked on the stub; the mark will indicate which side of the stub is facing the chamber door.
2. Visually inspect each stub for the possibility of overload. Overload is when the abundance of particles on the stub is so great, that not all of them adhere to the stub. If overload is suspected, analyze this case separate from any other case. After the case where overload is suspected, a new environmental blank will be analyzed to verify the chamber and surrounding areas are free from any suspect particles.
3. An environmental control stub (blank) will be placed in the sample handling area while sample stubs are being labeled and transferred to the SEM chamber. An environmental control will be analyzed during each automated run.
4. The z-axis will be checked for each stub, prior to initiation of the GSR run. This will be documented in the case record.
5. Initiate the automated GSR software on the EDS ensuring that analysis of a selected area of the positive known GSR sample is performed at the beginning of each run. The positive known GSR sample will be analyzed between cases and at the end of each run.
6. At least one particle from the first known GSR sample will be manually confirmed. The image and spectra of these particles will be placed in the case record. GSR particles do not need to be manually confirmed from subsequent known GSR samples analyzed throughout the run.
7. GSR particles reported from casework samples will be manually confirmed and a record containing an image and spectra of each confirmed particle will be placed in the case record.
8. The individual PDFs of reacquired particles will include a vertical scale, the conclusion of the analyst, the analyst initials and the date of conclusion. Examples of wording:

    a. Particle morphology correct & Ba, Pb and Sb confirmed:
    Ba + Pb + Sb + Morphology = GSR *(May use commas in place of "+")*

    b. If morphology correct and one of elements unconfirmed or absent:
    Unconfirmed Ba + Pb + Sb + Morphology = Associated GSR

    c. If morphology correct and 2 elements unconfirmed and/or absent:
    Ba + Unconfirmed Pb + Unconfirmed Sb + Morphology = No GSR
    Ba + Unconfirmed Sb + Morphology = No GSR

    d. Particle morphology incorrect:
    Incorrect Morphology = No GSR

    e. If the particle has high levels of Fe:
    High Fe = No GSR

    .

9. Samples that do not contain any 3 component particles initially identified by the instrument, but do contain 2 component particles, shall be reacquired for confirmation. A minimum of 10 particles (if present) must be reviewed for morphology and composition per set of stubs analyzed; scans will be included in the casefile.
10. For clothing or other items submitted for GSR, if applicable, the following instructions should be followed; there may be exceptions/circumstances which require stubbing of different areas.
    a. The garment/item in question should be placed side-up on a clean work bench covered with butcher paper. SEM stub(s) are then dabbed over the questioned area until the tackiness of the adhesive dissipates. Areas of interest to be searched can be sectioned off using a visible colored marker. A description explaining the areas sampled will be documented in the case notes.
    b. The following are example areas and may vary from case-to-case or otherwise requested by the submitting agency:
        i. Long Sleeve Shirt
            1. Front and back right cuff to mid-sleeve area
            2. Front and back left cuff to mid-sleeve area
            3. Front right chest area
            4. Front left chest area
        ii. Short Sleeve Shirt
            1. Front right chest and partial sleeve area
            2. Front left chest and partial sleeve area
        iii. Pants and shorts
            1. Front right pocket and upper thigh area
            2. Front left pocket and upper thigh area
        iv. Other types of garments or inanimate objects will be treated on a case-by-case basis
11. For stubs with possible blood, the stubs may not be analyzed.

Protocol Notes:

1. Samples taken from active living individuals up to 10 hours after the shooting incident will be analyzed unless there is sufficient cause not to analyze these samples. Samples taken more than 10 hours after the shooting incident from an active living individual will not be analyzed unless there is sufficient cause.
2. A shooting victim's clothing will not be analyzed.
3. If a GSR Kit and clothing are submitted on the same person, the clothing may not be analyzed if the GSR Kit is positive for the presence of GSR.
4. If a control stub is present in the GSR kit it will be given an item number and properly labeled but it may not be analyzed.
5. If the automated GSR software detects 20 Characteristic GSR particles on a single stub, the analysis of that stub will cease. An analyst may choose to continue analysis, if needed.
6. If the automated GSR software detects one particle in multiple fields, noted by its coordinates, the duplicate data may be deleted and is not considered rejected data.

7. An analyst may group stubs from the same GSR kit into a single sample set. If the automated GSR software detects 20 Characteristic GSR particles on a single sample set, the analysis of that set will cease.
8. Unless otherwise noted in the casefile, the date of sample preparation and initials/name of the analyst performing the sample preparation will be the same date and person documented on the initial line of chain of custody for the specific sub-item.
9. Required PDFs for the case file include the Calibration and Thresholds report, the Marked Features for GSR report (convert the Cobalt spectrum to line draw and include a vertical scale), the Marked Features report for each kit analyzed and the Marked Features report for the environmental blank.

Interpretation of Test Data:

1. *Morphology*:
   The majority of GSR consists of spherical or molten particles between 0.5 micron and 5.0 microns in diameter; the remaining particles are irregular in shape and vary dramatically from 1 to 100+ microns in size. In general, it is not consistent with the mechanisms of GSR formation to commonly find particles with crystalline morphology. Since morphology can vary greatly, it should never be considered the only criterion for identification of GSR.
2. *Elemental Composition*:
   Three component GSR particles are composed of Lead, Barium and Antimony. Two component GSR particles are composed of any combination of two of the above-mentioned elements. Particles with elevated levels of additional elements that indicate a source such as fireworks or primer-based construction tools would not be considered gunshot residue. Ammunition produced and marketed to be non-toxic or environmentally safe can produce particles consisting of elements not listed previously such as Strontium or Titanium and Zinc.

Recommended Report Wording:

1. All reports will indicate what analysis was performed in addition to the results. For example:

   The following items were analyzed utilizing the Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS):

   The following items were sampled for analysis:

2. If GSR particle(s) of interest are detected and confirmed on the evidence collection stubs:

   1A Microscopic particles containing lead, barium, and antimony were confirmed.

3. If the limit of 20 three component particles was reached on a single sample set, causing the run to terminate prior to analysis of all the evidence collection stubs in the GSR Kit, evidence collection stubs that are not analyzed:

   1B The detection limit of primer gunshot residue particles was reached during the analysis of item 1A, therefore this item was not analyzed.

4. If no primer gunshot residue particles are detected:

1A No primer gunshot residue particles were detected.

5. If the living subject was sampled more than 10 hours after the incident:

   1A Not analyzed due to the samples being collected more than 10 hours after the incident.

6. If the GSR kit control stub is unopened (intact factory seal) and unused:

   1A The control stub was not analyzed.

7. If clothing is not analyzed because a GSR Kit, submitted on the same person, has been analyzed and is positive:

   1A The shirt/pants/hat/jacket/clothing was not analyzed.

8. If clothing is stubbed for analysis, the description of the evidence will include the stubbing and the results for the clothing will be reported. For example:

   Description of Evidence:
   2 One sealed brown paper sack containing one black shirt
   2A Stubbing of right side of shirt
   Results and Conclusions:
   The following items were analyzed utilizing…
   2 Details of the examination of the sampling stubs for this item are as follows:
   2A Microscopic particles containing….

9. If three component or two component particles are detected on the environmental blank, the analyst will analyze each particle to confirm the presence of characteristic or associated particles. If these particles are confirmed, the report will include a qualifying statement detailing the type of particles confirmed on the environmental stub. For example:

   Microscopic particles containing lead, barium and antimony were detected and confirmed on the environmental control stub. The results obtained on the evidence stubs may have been contaminated with such particles during the processing of the evidence.

10. All reports, in which the results are positive for three component GSR particles, will include a statement stating particles are molten in appearance. For example:

    All reported particles are molten in appearance.

    Microscopic particles containing all three elements (lead, barium and antimony) with a molten appearance are products of the discharge of a firearm.

11. All reports will include a statement providing interpretation of the significance of all reported results. For example:

    The presence of primer gunshot residue on a person is consistent with that person having discharged a firearm, having been in the vicinity of a firearm when it was discharged, or having handled an item with gunshot residue on it.

    The absence of primer gunshot residue on a person does not eliminate that person from having discharged a firearm.

    The presence of primer gunshot residue on an object is consistent with that object having been in the vicinity of a firearm when it was discharged or having otherwise come in

contact with a person or object bearing gunshot residue.

12. If the stub has possible blood, use the following report wording:

1A Item not analyzed due to presence of possible biological substance.

TR-130 Physical Fit Comparison

Principle of the Protocol:

Physical fit comparisons, also known as fracture match comparisons, are performed in an attempt to uniquely associate a piece of material with an item from which it is thought to have originated. They are applicable to situations in which an item has been broken, torn, cut or otherwise separated into two or more pieces. If the separation involves random processes, the particular separation would not be expected to occur again in exactly the same way. It is therefore possible that the edges of the separate pieces could be fit together in a unique fashion, much like the pieces of a jigsaw puzzle, to demonstrate a common origin.

Associated Protocol(s):

TR-8 Collection of Trace Evidence

TR-18 Digital Photography

Specimen Required (Sample Handling and Preservation):

Evidence submitted for physical fit comparisons should be composed of a material that is stable after separation. Very soft or elastic materials may not be suitable because they may become stretched or deformed at the separation line. Examples of evidence suitable for physical fit comparisons are glass, plastic, wood, metal and paint chips.

Apparatus and Materials:

1. Stereomicroscope
2. Tweezers or forceps
3. Digital Camera

Individual Steps of Protocol:

Physical Comparisons Which Involve Fractured Edges

1. Perform a visual examination (macroscopic/microscopic) of each of the items.
2. Observations should be made regarding the overall characteristics exhibited on each of the items. Examples of overall characteristics are the type of material (glass, plastic, etc.) color, texture, curvature, and thickness. If the items exhibit similar overall characteristics, then the examiner should proceed by examining and comparing edges of one item to the edges of other items.
3. Once the examiner has found similarly shaped edges, the pieces are then tested by carefully holding them close together and in different positions. If a physical fit is found, the pieces will "jigsaw" together.
4. The examiner should examine the separation line (macroscopic/microscopic) for continuity of surface markings. (This portion of the examination is especially important for items that have smooth (straight, featureless) separation lines). As an option, the match may be confirmed and the results documented by a second examiner, document initials of the second examiner in the notes of the case file).

5. The examiner must document the results of his/her examination in the form of written notes and photographs. In cases where a physical fit has been made and the items are of sufficient size, the examiner may draw a line with an indelible marker on both of the physically matched items and mark the same letter designation (A, B, C, etc.) on each side of the line. This is particularly helpful in cases involving multiple physical fits, especially if the items will be reconstructed at a later date.

Physical Comparisons Which Do Not Involve Fractured Edges

1. Sometimes the examiner is asked to conduct a physical comparison of items without any fractured surfaces (i.e.: buttons in a set).
2. This type of examination involves the comparison of the overall characteristics of the items submitted. This would include such things as the composition of the item, color, texture, and dimension. The examiner should follow the procedure outlined for physical fit comparisons. However, the examination would not include any comparison of fractured edges.

Protocol Notes:

1. If any significant trace materials are disclosed (fibers, hair, blood, etc.), the item should be processed for the trace material prior to any physical comparison of that item. This will prevent any loss or cross transfer of material. The examiner should recognize that the trace material may also form part of the physical fit (a blood smear on a broken item may form part of the match).
2. There are three important considerations when packaging evidence for physical comparison examinations. They are as follows:
    a. The separated edges should be protected from any further damage.
    b. Questioned and known items need to be packaged separately.
    c. Caution needs to be taken when allowing known and questioned samples to come in contact. Possibilities such as transfer of fibrous material or actual damage and subsequent change of the edges could occur.
3. In cases involving a large number of questioned and known submissions, it is often easier to first reconstruct all the known items and all the questioned items prior to conducting any questioned to known comparisons.
4. Unless otherwise noted, the date of comparison and initials/name of the analyst performing the comparison will be documented in photos.

TR-130 Physical Fit Comparison

Recommended Report Wording/Interpretation of Test Data:

The following are examples of possible report wording:

1. If a physical fit exists:

   Physical comparison of the piece of questioned plastic

   The broken grille piece recovered at the scene (Item 1) and the portion of known grille (Item 2) were found to be a physical fit (*or* found to form a physical fit) and conclusively established that the broken grill piece originated from the known grille.

   Physical comparison of the piece of questioned glass

   The broken glass recovered from the pants (Item 1) has edges that physically match (or form a physical fit) with the piece of known glass from the store's window (Item 2). Therefore, these pieces were once joined to form a single item.

2. If a physical fit does not exist and the class characteristics of the items are different:

   Physical comparison of the piece of questioned plastic

   No physical fit exists between Item 1 and Item 2. These items were found to be different _____ (i.e., colors, thicknesses). Therefore, Item 1 and Item 2 did not originate from a common source.

   Physical comparison of the piece of questioned glass

   The broken glass recovered from the pants (Item 1) and the piece of known glass (Item 2) were found to be different in color. The piece of questioned glass could not have originated from the piece of known glass.

3. If a physical fit does not exist, but the class characteristics of the items are consistent:

   Physical comparison of the piece of questioned plastic

   The broken grille piece recovered at the scene (Item 1) and the portion of known grille (Item 2) do not form a physical fit. However, the overall characteristics (physical and chemical) of Item 1 are consistent with Item 2. Therefore, Item 1 could have originated from Item 2 or another item with the same characteristics (physical and chemical).

4. If any other analysis was performed in an effort to associate or disassociate the compared items, then the examiner should follow the report writing guidelines for that specific analysis type.

TR-150 Pressure Sensitive Tape Analysis

Principle:

Forensic pressure sensitive tape analysis typically concerns the comparison of tape samples, typically recovered from a crime scene or victim to a roll or piece of tape that are associated with a suspect.

Pressure sensitive tape can consist of components such as release coat, paper or film backing, scrim fabric, and adhesive, among others. Comparison of the backing color, texture, width, thread count, weave uniformity, the characteristics and composition of the warp and fill fibers within the fabric (if present), and the color and composition of the adhesive can be used to assist the examiner in formulating a conclusion.

Before conducting a tape comparison, consider the need to preserve any latent prints, DNA, or trace evidence that may be on the tape.

Associated Protocol(s):

TR-06 Fourier Transform Infrared (FTIR) for Trace Analysis
TR-07 Polarized Light Microscopy (PLM)
TR-18 Digital Photography
TR-19 Microspectrophotometer Analysis
TR-23 Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS)
TR-130 Physical Fit Comparison
TR-170 Fiber Analysis

Specimen(s) Required (Sample Handling and Preservation):

1. Questioned tape sample(s)
2. Known tape roll or sample

Apparatus and Materials:

1. Polarized Light Microscope (PLM)
2. Stereomicroscopes
3. Comparison Microscope
4. Fourier Transform Infrared Spectrometer (FTIR)
5. Microspectrophotometer (MSP)
6. Scanning Electron Microscope (SEM)
7. Energy Dispersive Spectrometer (EDS)

TR-150 Pressure Sensitive Tape Analysis

Individual Steps of Protocol:

1. Examine each sample separately, documenting the following characteristics, as applicable:
    a. Width of tape
    b. Color of backing
    c. Presence and shape of any markings on the backing
    d. Overall thickness and thickness of backing
    e. Uniformity of the fabric weave
    f. Number of threads per inch in the warp and fill directions
    g. Color of the adhesive
    h. Polymer orientation
    i. Number of layers

2. Perform a physical comparison, if adequate samples are submitted and the physical characteristics of the questioned and known tapes are similar.

    a. If a physical fit exists between the known and questioned tape, it is not usually necessary to continue with tape analysis.

    b. Heat, liquid nitrogen and other solvents (i.e. methanol or hexanes) can be used to release the adhesive and allow the ends of the tape to be compared.

3. Perform sample analysis

    At any point where analytical or physical properties differ, analysis can be concluded. Analysis does not have to be performed in the order listed below.

    a. If threads are present within the tape:

        i. Cut small separate sections (not on the torn edge) of thread from the warp and from the fill directions

        ii. Remove the adhesive from the backing to expose threads using a solvent and separately mount the warp and fill thread fibers.

        iii. On the threads, perform microscopic examination, cross sectioning and FTIR, as necessary, for comparison. Warp and fill fibers of the scrim can be examined using a UV light source, a forensic light source, and/or fluorescence microscopy.

    b. A small portion of the adhesive may be removed and compared using FTIR. Analysis for comparison can also be accomplished using the FTIR-ATR; place the tape directly on the ATR for analysis of the backing and adhesive.

    c. If tape backings have color, MSP can be used for color comparison.

    d. Make a cross section of multilayered backed tape by freezing the tape with liquid nitrogen or compressed air to make the tape more rigid and cutting the edge of the tape. Examine the cross sections microscopically for comparison.

    e. The polymer orientation of polypropylene tapes can be determined by using polarizing light microscopy.

        i. Observe interference colors, patterns and variation of the tape when rotating the stage.

ii. Monoaxially oriented polymers will have one point of extinction, every 90°.

iii. Biaxially oriented polymers will have two points of extinction, every 90°.

iv. The angles of the crosshatches and extinction angles should be consistent within a single roll of tape.

v. Some tapes may not exhibit total extinction. Irregularities should be noted.

4. Compare the analytical data for the known and questioned tape evidence to evaluate the possible association between the two

Protocol Notes:

1. Questioned and known tape samples which exhibit meaningful differences in the observed characteristics such as color, thickness, material composition, texture of the backing, width, thread count, uniformity of the fabric weave, adhesive color, adhesive composition or fiber composition leads to the conclusion that the questioned tape and known tape are from different rolls of tape.
2. Questioned and known tape samples which exhibit no meaningful differences in the observed characteristics such as color, thickness, material composition, texture of the backing, width, thread count, uniformity of the fabric weave, adhesive color, adhesive composition or fiber composition leads to the conclusion that the questioned tape and known tape could have come from a common source.
3. Questioned and known tape samples with a physical fit lead to the conclusion that the questioned tape was once joined to, and subsequently removed from, the known tape.
4. Tape may not be in its original state due to weathering, stretching, chemicals, etc. These changes may prevent a full range of examinations from being performed and, therefore, may limit the information obtained from the analyses.
5. Unless otherwise noted, analysis will be performed by the assigned analyst.
6. Unless otherwise noted in the casefile, dates of analysis will be documented in the following manner:

    a. Polarized Light Microscopy – photographs/PDF
    b. Fluorescence – photographs/PDF
    c. FTIR – on instrumental data
    d. Light Microscopy – photographs/PDF
    e. Microspectrophotometry – on instrumental data
    f. Comparison Microscopy – photographs/PDF
    g. SEM/EDS – on instrumental data

Recommended Report Wording/Interpretation of Test Data:

1. If the sample could not be analyzed due to its conditions or sample size, the report may be worded as follows:

    Item 1: No analysis could be performed due to insufficient sample size.

    Item 1: No analysis could be performed due to the condition of the sample.

    No comparison could be made between tape (Item 1) and the roll of tape (Item 2) due to the sample condition of Item 1.

2. If a physical fit is determined between the known and questioned tape evidence and no further analysis is performed, the report wording found in TR-130 "Physical Fit Comparison" shall be followed.

3. The report should indicate what analysis was performed in the case:

   The following were utilized during the analysis of this case: Light Microscopy, Fourier Transform Infrared Spectrometry (FTIR), and Microspectrophotometry (MSP).

4. If the questioned sample has been eliminated as originating from the known sample, or if the questioned and known samples are eliminated as originating from the same source, the report should indicate how the samples are different and a statement clearly communicating the elimination. Some examples follow:

   Item 1 and 2 could not have originated from the same source due to differences in color of adhesive/backing (or any other criteria: width of tape, fabric weave, chemical composition, etc.)

   Item 2 can be eliminated as being the source for Item 1 due to differences in color of adhesive/backing (or any other criteria: width of tape, fabric weave, chemical composition, etc.)

5. If the known and questioned samples are consistent and an association is made between the two, the report should indicate all areas where the samples are consistent. The significance of the association shall be communicated clearly and qualified properly in the report. Some examples follow:

   Item 1 (green duct tape recovered from the victim) and item 2 (roll of green duct tape from the suspect's vehicle) were consistent in colors of tape backing and adhesive, width, fabric weave and chemical properties. The tape recovered from the victim could have originated from the green duct tape from the suspect's vehicle or another source.

   Item 1 (green duct tape) was consistent in colors of tape backing and adhesive, width, fabric weave and chemical properties to item 2 (roll of green duct tape). The green tape on the victim could have originated from the roll of green duct tape or from another source of tape of the same colors, width, fabric weave, and chemical composition.

6. If no definitive conclusions can be reached, the report should indicate how the samples are consistent or different and a statement clearly communicating the reason the analysis is inconclusive.

   Item 1 and 2 were consistent in backing colors and width, but their chemical compositions were slightly different. Therefore, the results of this examination were inconclusive.

   Item 1 and 2 were consistent in fabric weave, width, and chemical composition; however, a slight difference was noted in the backing colors. This may be the result of weathering of the tape or the question tape may be from a different tape of a similar type. Therefore, the results of this comparison were inconclusive.

TR-160 Paint and Polymer Analysis

Principle:

Forensic paint analysis typically concerns the recognition, identification, and comparison of questioned and known paint samples. An examiner may also be requested to obtain information on an individual paint sample with no known paint evidence available.

Paint can be characterized by a number of physical and chemical properties. Limited sample size and sample preservation requirements dictate that the comparative tests must be selected and applied in a reasonable sequence in order to maximize the discriminating power of the test results without undue consumption of the sample. Sample size, condition of evidence, type of sample (e.g. smears, transfer patterns, multi-layered or mixed samples) are factors that affect the sequence of tests that will be conducted.

This protocol is intended primarily for the examination and comparison of paint evidence, but it may also be applied to polymers and plastics.

Associated Protocol(s):

TR-06 Fourier Transform Infrared (FTIR) for Trace Analysis
TR-07 Polarized Light Microscopy (PLM)
TR-15 Elemental Analysis
TR-18 Digital Photography
TR-19 Microspectrophotometer Analysis
TR-23 Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS)
TR-130 Physical Fit Comparison

Specimen Required (Sample Handling and Preservation):

1. Question paint evidence
2. Known paint evidence, which is comprised of all layers, from the outermost layer to the substrate, preferably collected near the area where the questioned paint is suspected of originating.

Apparatus and Materials:

1. Polarized Light Microscope (PLM)
2. Stereomicroscopes
3. Fourier Transform Infrared Spectrometer (FTIR)
4. Microspectrophotometer (MSP)
5. Scanning Electron Microscope (SEM)
6. Energy Dispersive Spectrometer (EDS)
7. Microtome

Individual Steps of Protocol:

1. Examine each sample separately.
2. If practical, perform a physical fit comparison between the known and questioned paint samples. Paint samples should be taken before a physical fit is performed. If a physical fit is determined between the known and questioned paint evidence, it is not necessary to continue with paint analysis.

3. Utilizing a stereomicroscope, comparison microscope, or polarized light microscope, examine representative layers of the paint evidence and record the descriptive information for each sample. This shall include, as applicable:
    a. Possible Repaint (presence of Bondo, sanding mark, excessively thick and/or uneven layers, etc.)
    b. Approximate amount or size of paint sample
    c. Type (vehicular/structure; enamel/lacquer; with or without decorative flake)
    d. Physical characteristics (soft, hard, pliable, etc.)
    e. Surface or texture features (dull, glossy, pitted, rough, weathered, etc.) and/or
    f. Number of paint layers, relative color, sequence, and thickness.
4. If the sample is of sufficient size, prepare cross-sections in order to detect all layers. The microtome may be used when necessary to obtain satisfactory cross-sections.
5. In the instance that known samples are unavailable, the Paint Data Query (PDQ) may be used to gain vehicle information when it is determined that a questioned paint sample may be automotive paint. To perform a PDQ Search, the following should be done:
    a. Determine the physical and chemical characteristics of the sample and ensure that an Original Equipment Manufacturer (OEM) sequence is present.
    b. If an OEM sequence is present, each layer will be analyzed by FTIR and designated appropriately (OT2, OT1, OU1, OU2, etc.)
    c. Each layer will be evaluated for individual chemical components and this information will be entered into the chemical and color properties section of the PDQ program and a search of the database will be conducted.
    d. Using the information gathered, a spectral search of each layer will be performed against the PDQ spectral database and conclusions will be drawn based on the information gathered.
6. Compare the known samples macroscopically and microscopically. The questioned and known paint samples may be excluded as originating from the same source of paint if significant differences are observed in the color or layer structure. NOTE: Before excluding the samples, the examiner should first confirm that the difference is not attributed to improper collection. For instance, if the known sample was collected from an area of the car not close to the damaged area and had been previously repainted, then further known samples should be requested.
7. The analyst should now perform a combination of analyses that extract the greatest potential for discrimination between the samples. Multi-layered samples must also be consistent in layer structure. Sample size, condition, and layer structure complexity should be considered when determining which techniques to use. Following layer structure determination, FTIR analysis usually provides the most discrimination of paint samples and is typically conducted first. The number of analytical tests performed is left to the discretion of the analyst. The following is a list of available techniques:
    a. Solubility/Microchemical Tests
    b. Polarized Light Microscopy
    c. Fluorescence

d. FTIR
e. SEM/EDS
f. Light Microscopy
g. Microspectrophotometry

8. Compare the analytical data for the known and questioned paint evidence to evaluate the possible association between the two.
9. Samples that are neither constrained by amount nor condition shall be subjected to analysis by FTIR, MSP, and SEM/EDS before a questioned and known sample are determined to originate from the same source.

Protocol Notes:

1. Under most circumstances, for a known and questioned paint sample to have originated from the same source, all physical and chemical characteristics that are compared between the known and the questioned samples must be consistent. Consistency does not mean that; for example, since the questioned sample is a lighter shade of blue than the known sample, then they are consistent. Consistency in this case means the two samples cannot be distinguished apart.
2. Automotive paint is typically found as smears or multi-layer fragments, and can usually be recognized by a characteristic layer sequence and chemical composition.
3. Architectural paint fragments may be recognized by their particulate texture and /or rough surfaces, and may have wood or masonry material as a substrate.
4. Unless otherwise noted, analysis will be performed by the assigned analyst.
5. Unless otherwise noted in the casefile, dates of analysis will be documented in the following manner:
    a. Solubility/Microchemical Tests – in casefile notes
    b. Polarized Light Microscopy – photographs/PDF
    c. Fluorescence – photographs/PDF
    d. FTIR – on instrumental data
    e. SEM/EDS – on instrumental data
    f. Light Microscopy – photographs/PDF
    g. Microspectrophotometry – on instrumental data

Recommended Report Wording/Interpretation of Test Data:

1. If the sample could not be analyzed due to its conditions or sample size, the report may be worded as follows:

    Item 1: No analysis could be performed due to insufficient sample size.

    Item 1: No analysis could be performed due to the condition of the sample.

    No comparison could be made between paint recovered from the hammer (Item 1) and the paint recovered from the wall (Item 2) due to the sample condition of Item 1.

2. If a physical fit is determined between the known and questioned paint evidence and no further analysis is performed, the report wording found in TR-130 Physical Fit Comparison shall be followed.

3. If a physical fit is not determined between the known and questioned paint evidence, the report wording found in TR-130 Physical Fit Comparison shall be followed.

4. The report should indicate what analysis was performed in the case:

   The following were utilized during the analysis of this case: Light Microscopy, Fourier Transform Infrared Spectrometry (FTIR), Microspectrophotometry (MSP) and Scanning Electron Microscopy with Energy Dispersive Spectrometer (SEM/EDS).

5. If the questioned sample has been eliminated as originating from the known sample, or if the questioned and known samples are eliminated as originating from the same source, the report should indicate how the samples are different and a statement clearly communicating the elimination. Some examples follow:

   Item 1 and 2 could not have originated from the same source due to differences in color (or any other criteria: layer sequence, chemical composition, etc.)

   Item 2 can be eliminated as being the source for Item 1 due to differences in color (or any other criteria: layer sequence, chemical composition, etc.)

6. If the known and questioned samples are consistent and an association is made between the two, the report should indicate all areas where the samples are consistent. The significance of the association shall be communicated clearly and qualified properly in the report. Some examples follow:

   Item 1 (green metallic multi-layer paint recovered from the victim's clothing) and item 2 (paint from the suspect's vehicle) were consistent in color, layer sequence, physical and chemical properties. The paint recovered from the victim's clothing could have originated from the suspect's vehicle or a vehicle painted in the same manner.

   Item 1 (green paint from hammer) was consistent in color, texture, and chemical composition to item 2 (green paint from wall). The green paint found on the hammer could have originated from the green paint on the wall or from another source of paint of the same color, texture, and chemical composition.

7. If no definitive conclusions can be reached, the report should indicate how the samples are consistent or different and a statement clearly communicating the reason the analysis is inconclusive.

   Item 1 and 2 were consistent in color and texture, but their chemical compositions were slightly different. Therefore, the results of this examination were inconclusive.

   Item 1 and 2 were consistent in texture, layer structure, and chemical composition; however, a slight difference was noted in color. This may be the result of inadequate sampling of the reference paint or the question paint may be from a different vehicle of a similar type. Therefore, the results of this comparison were inconclusive.

TR-160 Paint and Polymer Analysis

8. For samples where a search of PDQ was successful:

   Item 1 was consistent with an original paint finish on a motor vehicle. A search of the automotive paint database query (PDQ) indicated that Item 1 (multi-layered red metallic paint) was most similar to a GMC paint utilized as original finish on 1978-1981 Buick Skylark manufactured in the Detroit Plant. The search for a suspect vehicle should be focused on, but not limited to, a 1978-1981 Buick Skylark. It should be noted that not all makes/models/years of vehicles produced by each manufacturer are present in the paint database. Sufficient sample from item 1 remains for comparison to known paint from a suspect vehicle.

9. For samples where a search of PDQ was unsuccessful:

   Item 1 consisted of a multi-layered black paint sequence on a black substrate. A search of the automotive paint database (PDQ) was conducted. The results of the search did not indicate a possible make/model of origin. It should be noted that painted plastic substrates are not well represented in the database.

   Item 1 (red paint from victim's clothing) was indicative of an automotive repaint; therefore, no make/model determination could be made. The red paint is suitable for comparison purposes. If a possible source of paint is located, resubmit item 1 along with the possible paint source for comparison.

TR-170 Fiber Analysis

Principle of the Protocol:

This protocol is for the identification and comparison of natural and synthetic fibers. Fibers collected from a victim's clothing may be compared to fibers from a suspect's vehicle, for example, to determine the possibility of contact between the victim and suspect. The absence of fibers; however, should not imply that contact was never made. An examiner may be requested to obtain information from questioned fibers when known fibers are not available. Unless a physical fit is determined or other unique circumstances exist, a comparison examination can never prove the exact source of a questioned fiber.

Yarn, thread, cordage, and fabric evidence submitted may also be examined utilizing this protocol.

Associated Protocols:

TR-06 Fourier Transform Infrared (FTIR) for Trace Analysis
TR-07 Polarized Light Microscopy (PLM)
TR-15 Elemental Analysis
TR-18 Digital Photography
TR-19 Microspectrophotometer Analysis
TR-23 Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS)
TR-130 Physical Fit Comparison

Specimen(s) Required (Sample handling and preservation):

1. Questioned Fibers
2. Known Fibers

Standards

Known fibers (Synthetic and Natural)

Apparatus and Materials:

1. Polarized Light Microscope (PLM)
2. Stereomicroscopes
3. Comparison Microscope
4. Fourier Transform Infrared Spectrometer (FTIR)
5. Microspectrophotometer (MSP)
6. Scanning Electron Microscope (SEM)
7. Energy Dispersive Spectrometry (EDS)

Individual Steps of Protocol

1. Examine questioned and known samples in separate areas and/or at different times.
2. If practical, perform a physical fit comparison between the known and questioned samples. Fiber samples should be taken before a physical fit comparison is performed. If a physical fit is determined between the known and questioned fiber evidence, it is not necessary to continue with fiber analysis.

3. The fiber evidence should be examined macroscopically and microscopically. This can be done by utilizing a stereomicroscope, comparison microscope, or polarized light microscope. Document physical characteristics of each sample such as: Crimp, straight, visible color, any variation in color, relative diameter, damage, fineness, coarseness, and optical cross-section. If the sample contains yarns, threads, or sections of fabric, record the construction and composition (blend, woven, twist, braided, etc.).

4. If a comparison is requested, compare the samples macroscopically and microscopically. A side-by-side examination using the comparison microscope is a valuable tool for comparing fiber widths. The questioned and known fiber samples may be excluded as originating from the same source if significant differences are observed in class, physical characteristics, or color. NOTE: Before excluding the samples, the examiner should first confirm that the difference is not attributed to improper collection. For instance, if known and questioned carpet fibers appear consistent in all areas except color; the questioned fibers may have come from an area exposed to sunlight from a window, while the known sample was taken from an area that was shielded from sunlight. Further known samples may need to be requested.

5. The analyst should perform a combination of analyses that extract the greatest potential for discrimination between the samples. The analyst must do at a minimum a macroscopic examination.

   When possible, a full characterization of the questioned and known fibers should be performed. Characterizing fibers generally includes using a stereomicroscope, polarized light microscope, FTIR and MSP. During the analysis, the analyst should identify the generic fiber class, describe physical and optical properties, and compare the fiber color.

6. If a comparison has been requested, compare the analytical data for the known and questioned fiber evidence to evaluate the possible association between the two. In most instances, an analyst must utilize at a minimum, polarized light microscope, FTIR, and MSP to be able to determine two samples are consistent.

7. Cotton identification and comparison requires only microscopic observation and when required for color, microspectrophotometry. Cotton fibers are transparent, colorless tubes which have collapsed into irregularly to regularly twisted ribbons. A notable identifying characteristic is the lack of extinction between crossed polars. Proper notation of observed characteristics must be recorded in the examination documentation.

Protocol Notes

1. Under most circumstances, for a known and question fiber to have originated from the same source, the known and the questioned samples must be consistent in class, physical and optical characteristics, and color. Consistency means the two samples cannot be distinguished apart.

2. Unless otherwise noted, analysis will be performed by the assigned analyst.

3. Unless otherwise noted in the casefile, dates of analysis will be documented in the following manner:
   a. Polarized Light Microscopy – photographs/PDF
   b. Fluorescence – photographs/PDF
   c. FTIR – on instrumental data
   d. SEM/EDS – on instrumental data

e. Light Microscopy – photographs/PDF
f. Microspectrophotometry – on instrumental data

Recommended Report Wording / Interpretation of Test Data:

1. If the sample could not be analyzed due to its conditions or sample size, the report may be worded as follows:

   Item 1: No analysis could be performed due to insufficient sample size.

   Item 1: No analysis could be performed due to the condition of the sample.

   No comparison could be made between the fiber recovered from the victim's clothing (Item 1) and the fiber recovered from the suspect's vehicle (Item 2) due to the sample size of Item 1.

2. If a physical fit is determined between the known and questioned fiber evidence and no further analysis is performed, the report wording found in TR-130 "Physical Fit Comparison" shall be followed.

3. If class and/or color of a fiber is determined, the report should indicate what analysis was performed and the class and/or color of the fiber. The report may be worded as follows:

   Item 1 was examined microscopically and macroscopically. Item 1 is a brown nylon fiber.

4. The report should indicate what analysis was performed in the case:

   The following were utilized during the analysis of this case: Light Microscopy, Fourier Transform Infrared Spectrometry (FTIR), Microspectrophotometry (MSP) and Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM/EDS).

5. If the questioned sample has been eliminated as originating from the known sample, or if the questioned and known samples are eliminated as originating from the same source, the report should indicate how the samples are different and a statement clearly communicating the elimination. Some examples follow:

   Item 1 and 2 could not have originated from the same source due to differences in color (or any other criteria: class or physical characteristic)

   Item 2 can be eliminated as being the source for Item 1 due to differences in color (or any other criteria: class or physical characteristic)

6. If the known and questioned samples are consistent and an association is made between the two, the report should indicate all areas where the samples are consistent. The significance of the association shall be communicated clearly and qualified properly in the report. An example follows:

   Item 1 (fiber from blanket) is consistent in class, physical characteristics, and color to item 2 (fiber recovered from suspect's shirt). The fiber recovered from suspect's shirt could have originated from the blanket or from another source of the same class and color with the same physical characteristics.

7. If no definitive conclusions can be reached, the report should indicate how the samples are consistent or different and a statement clearly communicating the reason the analysis is inconclusive. An example follows:

   Item 1 and 2 are consistent in class and physical characteristics, but their color is slightly different. Therefore, the results of this examination are inconclusive.

   Items 1 and 2 are brown nylon fibers, and are consistent in class and physical characteristics. However, a slight difference was noted in the color of the fibers. This may be the result of inadequate sampling of the reference carpet or the question fiber may be from a different carpet of a similar type. Therefore, the results of this comparison are inconclusive.

TR-190 Examination, Identification and Comparison of Irregular, Non-Standard or Infrequently Encountered Forensic Samples

Principle of Protocol

The intent of this protocol is to provide guidance to analysts dealing with samples not specifically covered in the below protocols. These types of samples would not necessarily fit into conventional trace evidence categories such as fiber, paint, soil, glass, gunshot residue, or ignitable liquid. Some examples of these types of evidence are: pressure sensitive tape, sheetrock, food condiments, plastic bag/sheeting sequencing and fingernail tear comparisons. The analysis of this irregular evidence has been defined through the use of the instruments and techniques employed in typical trace evidence.

Specimen(s) Required:

Known and Unknown samples

Individual Steps of Protocol:

1. Form a written plan for all type(s) of analysis possible for the sample(s) in question. The plan will be documented in the case file.
2. Determine if the total quantity of sample required exceeds the quantity of existing sample.
3. If there is not enough sample, determine a priority of analyses based on which provides the most investigative information and is least destructive.
4. Perform the analysis based upon the current OSBI Trace and/or Controlled Substances protocols.
5. If a needed technical procedure does not exist, one must be able to develop and validate the new procedure prior to testing the case samples. OSBI CSD QP 21.2 describes the process for validation that will be followed.

Protocol Notes:

1. Due to the circumstances of working with samples that are not routinely seen, extensive documentation of the procedures performed become a critical step that is sometimes not required in a standard procedure. Keep in mind that during the analysis of the irregular samples, all important information must be documented which would allow a second analyst to reproduce the same results.
2. Unless otherwise noted, analysis will be performed by the assigned analyst.
3. Unless otherwise noted in the casefile, dates of analysis will be documented in the following manner:
    a. Polarized Light Microscopy – photographs/PDF
    b. Fluorescence – photographs/PDF
    c. FTIR – on instrumental data
    d. SEM/EDS – on instrumental data
    e. Light Microscopy – photographs/PDF
    f. Microspectrophotometry – on instrumental data

g. GC/MS – on instrumental data

Recommended Report Wording / Interpretation of Test Data:

If the questioned sample has been eliminated as originating from the known sample, or if the questioned and known samples are eliminated as originating from the same source, the report should indicate what analysis was performed, how the samples are different, and a statement clearly communicating the elimination.

If the known and questioned samples are consistent and an association is made between the two, the report should indicate what analysis was performed and all areas where the samples are consistent. The significance of the association shall be communicated clearly and qualified properly in the report.

If no definitive conclusions can be reached, the report should indicate what analysis was performed, how the samples are consistent or different, and a statement clearly communicating the reason the analysis is inconclusive.

Author(s) and Approvals

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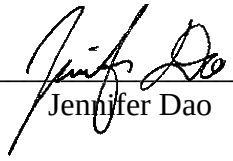
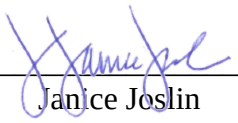
Approval:

Technical Manager  Jennifer Dao Date 10-30-25

Laboratory Director  Janice Joslin Date 10/30/2025

History

Rev. #	Issue Date	History
0	12-31-2017	Original Issue of Merged Protocols; Protocols formatted the same **TR-01 (Rev 11):** Added Associated Protocols. Frequency & Tolerance paragraph merged into Controls. Controls – removed "See Frequency..." statement. Reworded/changed section regarding actions if autotune fails. Protocols – removed second paragraph referring to knowns/blanks/autotunes, redundant to Controls. **TR-02 (Rev 07):** Rescinded. Content merged into TR-07. **TR-06 (Rev 03):** Associated Protocols- added section. Frequency & Tolerance – Moved to Controls. Controls – removed requirement to follow "qualification report" recommendations and changed the actions to be taken if ValPro fails. **TR-07 (Rev 03):** Content from TR-02 merged. Associated Protocols – removed TR-02. References – section was added. Apparatus – added microscopes and numbered items. Individual Steps of Protocol – moved Setup step "Rotate objectives…" to Protocol Notes. 3. Compensator – added specific instructions. 5. Refractive Index – added steps for Becke Line Method and Relative RI. Protocol Notes – added wording to 1. "when not in use". Interpretation of Test Data and Recommended Report Wording – combined the two sections. Reworded the first line regarding optical properties. **TR-08 (Rev 12):** Associated Protocols – added TR-130. Renumbered Individual Steps of Protocol - #10 was missing. Recommended Report Wording – added statement regarding off-site evidence collection reports. **TR-12 (Rev 07)** – Dropped the "s" from the title **Specimens**. Combined Special Supplies and Apparatus and Materials. Combined Frequency and Tolerance… and Protocol Notes. **TR-15 (Rev 12):** Combined Materials and Special Supplies and Apparatus. Controls & Standards - added requirements if negative control fails and requirements for running & archiving controls. **TR-16 (Rev 07):** Combined Frequency and Tolerance… and Standards. **TR-18 (Rev 04):** Added Apparatus and Materials and included camera. Individual Steps of Protocol – 2.a and 2.b combined and reworded, removing requirement for photos of all sides. 2.d. added mounted camera to end.3. added "compiled into a PowerPoint" **TR-19 (Rev 05):** Associated Protocols – new section with protocols. Controls – new section. Moved CRAIC Reference Filter Set from Apparatus to Controls. Moved Frequency… to Controls. Individual Steps of Protocol –added "Verify certification check of Filter Set." **TR-23 (Rev 05):** Combined Materials and Special Supplies and Apparatus into Apparatus and Materials. Controls and Standards – changed to match TR-15

History

TR-100 (Rev 12): Combined Materials and Special Supplies and Apparatus into Apparatus and Materials. Standards and Controls – added 9. regarding archiving controls. Individual Steps of Protocol – Step 6 added +/-5°C. Recommended Report Wording…- Added "for data interpretation" to E1618 statement.
TR-101 (Rev 07): Controls – removed reference to solvents. Combined Frequency and Tolerance… and Controls. Apparatus and Materials – added paperclips and magnets. Individual Steps of Protocol – added "using red stoppers" to first assembly instructions. Added assembly instructions for paperclip/magnet apparatus.
TR-130 (Rev 06): Renames Supplies to Apparatus and Material to match other protocols. Apparatus and Materials – added "digital" to camera.
TR-160 (Rev 05): No changes, other than formatting.
TR-170 (Rev 10): No changes, other than formatting.
TR-190 (Rev 09): No changes, other than formatting.
TR-500 (Rev 14): Rescinding as TR-500 and making it stand alone Quality Manual.

1 05-31-2018

Corrected document name in various headers throughout document
TR-01: Principle of Protocol – Added more description to mass spectrometer.
Renamed Reagent section to Solvents.
Controls – renamed reagent blank to solvent blank. Added specific requirement to solvent blanks.
Apparatus & Materials – added GC/MS.
Protocol Notes – changed wording from "be checked for function and purity" to "undergo function verification prior."
TR-06: Reagents renamed to Solvents.
TR-07: no changes.
TR-08: Protocol Notes – added 5. regarding dates and analyst of searching/evidence collection
TR-12: Reagents – removed section.
Apparatus & Materials – added carbon rods
TR-15: Protocol Notes – added 3. regarding date and analyst
TR-16: Individual Steps of Protocol – modified 6. To indicate calibration results will be archived in the casefile.
Protocol Notes – added 7. regarding date and analyst
TR-18: no changes
TR-19: no changes
TR-23: no changes
TR-100: removed the word "reagent" from "reagent blank" throughout protocol. Reagents renamed to Solvents. Removed 2. regarding standard analysis mixture.
Standards & Controls – added "class" to what is identified "a class of ignitable liquids." Added 2. requiring standards to be analyzed

History

		within 24 months of casework or if column is cut or changed. 8. defined "system blank". Individual Steps of Protocol – 8. Added volume to amount of solvent added. 9 & 10 removed "reagent" from "reagent blank." Added GC/MS section and instructions. 11. added "After analysis." Protocol Notes – added 5. regarding dates of sampling & analyst and 6. regarding instrumental analysis dates & analyst for standards and samples. Recommended Report Wording – 6. Removed wording for inconclusive gasoline. **TR-101:** no changes **TR-110 (Rev 17):** Added to Merged Trace Protocols. Protocol Notes – added 6. Regarding date and initials. Recommended Report Wording – 1. Added wording for reporting the sampling for GSR collection. **TR-130:** Protocol Notes – added 4. Regarding date & initials of comparison. **TR-160:** Fixed section number/name in header. Protocol notes – added 4. regarding assigned analyst and 5. regarding dates of analysis. **TR-170:** Protocol Notes – added numbering and added sections 2. Regarding assigned analyst and 3. regarding dates of analysis. **TR-190:** Fixed section number/name in header. Protocol notes – added 2. regarding assigned analyst and 3. regarding dates of analysis.
2	08-03-18	Change Lab Director's last name. **TR-01:** Protocol Notes – 2. Added reference to location of instrument maintenance spreadsheet. 3. Added location of user's manual. 4. Added reference to no calibration records and location of autotunes. **TR-06:** Protocol Notes – Added sections 4, 5 & 6 regarding maintenance & documentation of maintenance, user's manuals and calibration records. **TR-07:** Individual Steps of Protocol - 1.a. Clarified California Criminalistics …Manual as a reference only. Protocol Notes – Moved section 4 Regarding using knurled knobs to section 2. Moved what was section 2 to section 3 (cleaning) and added when lenses are to be cleaned and which lenses are included. Added section 5 regarding maintenance and where it is to be documented. Added sections 6 regarding user manuals and 7. regarding calibration records. **TR-08:** No changes. **TR-12:** No changes. **TR-15:** No changes. **TR-16:** Standards - Added requirements for vendor repairing calipers and calibration documentation.

History

		Protocol Notes – Combined section 7 into section 1, removing section 7. Section 5 added location of user's manual. **TR-18:** No changes. **TR-19:** Protocol Notes – Added section 4 regarding maintenance and documentation of maintenance, section 5 regarding user's manuals and section 6 regarding certification records. **TR-23:** Protocol Notes – Added section 4 regarding maintenance and documentation of maintenance, section 5 regarding user's manuals and section 6 regarding certification records. **TR-100:** Recommended Report Wording – Added section 2 and renumbered other sections, section 2 adds wording for retained sample vial containing a carbon strip extract. **TR-101:** No changes. **TR-110:** No changes. **TR-130:** No changes. **TR-160:** No changes. **TR-170:** No changes. **TR-190:** No changes.
3	08-31-19	**TR-08:** Added statement at beginning of protocol regarding accessing protocol when work is being performed outside of FSC. Protocol Notes – Added point 7. Regarding performing work outside of FSC and documenting accommodations/conditions. **TR-15:** Apparatus & Materials - Removed instrument model from SEM **TR-100:** Approved Methods - Added method parameters. **TR-110:** Apparatus & Materials - Removed instrument model from SEM
4	08-21-2020	**TR-01:** No changes. **TR-06:** No changes. **TR-07:** No changes. **TR-08:** No changes. **TR-12:** No changes. **TGR-15:** Apparatus and Materials – 1. Added "(SEM)", 2. Removed "Oxford Inca x Max" and added "(EDS)" **TR-18:** No changes. **TR-19:** No changes. **TR-23:** Principle of the Protocol – Replaced "LEO 1450 VPSE" with "Tescan VEGA 3." Added a "References" section with "1. VEGA 3 SEM Instruction for Use Manual, Tescan." Apparatus and Materials – Replaced "LEO 1450 VPSE" with "Tescan VEGA 3." Individual Steps of Protocols – Paragraph 2. Replaced the specific instructions for heating a new filament with a reference to the VEGA 3 manual as a guide. Removed reference to the hysteresis button and added reference to the Degauss feature for clearing noise. Protocol Notes – Deleted paragraph 1, referencing the chiller fluid and vacuum fluid and renumbered subsequent paragraphs. New

History

		paragraph 1. Changed filament hours from "150 hours" to "300 hours or longer." **TR-100:** Protocol Notes – Inserted new paragraph 4. regarding analysis of soil samples and renumbered subsequent paragraphs. **TR-101:** No changes. **TR-110:** Apparatus and Materials – Paragraph 2. Removed "Oxford." Recommended Report Writing – Paragraph 9. removed references to two component particles. Removed last paragraph in the examples, referencing particles with 2 of the elements. **TR-130:** No changes. **TR-160:** Apparatus and Materials – Split paragraph 5. into 5. and 6. Add "(SEM)" to paragraph 5. In paragraph 6. change "Spectrometry" to Spectrometer and remove "SEM" from the abbreviation that follows. Renumber "Microtome" to paragraph 7. Recommended Report Wording/Interpretation of Test Data – Paragraph 3. change "Scanning Electron Microscopy – Energy Dispersive X-Ray Spectrometry" to "Scanning Electron Microscopy with Energy Dispersive Spectrometer." **TR-170:** Apparatus and Materials – Split paragraph 6. into 6. and 7. Add "(SEM)" to paragraph 6. Remove "SEM" from "(SEM/EDS)." Recommended Report Wording/Interpretation of Test Data – Paragraph 4. change "Scanning Electron Microscopy – Energy Dispersive X-Ray Spectrometry" to "Scanning Electron Microscopy with Energy Dispersive Spectrometer." **TR-190:** No changes.
5	09-30-2021	**TR-01**: No changes. **TR-06**: Apparatus & Materials – 1. Updated the FTIR model to current instrument. Protocol Notes – Paragraph 3. Removed nitrogen gas. **TR-07**: Associated Protocols – Added TR-150 **TR-08**: Apparatus & Materials – Added Post-It Notes & Slides as 15 & 16. **TR-12**: No changes. **TR-15**: No changes. **TR-16**: No changes. **TR-18**: No changes. **TR-19**: No changes. **TR-23**: Protocol Notes – Changed 300 to 500 for the filament hours. **TR-100**: No changes. **TR-101**: No changes. **TR-110**: Individual Steps of Protocol – Added paragraph 8 regarding confirmation of stubs with only 2 component particles. Recommended Report Writing – Inserted new paragraph 8, regarding report wording for stubbed clothing; renumbered paragraphs 8-11. Paragraph 11 added example for presence of primer gunshot residue on an object.

History

TR-130: No changes.
TR-150: New written protocol for tape analysis; tape analysis is not a new protocol. Formerly, the use/combination of current protocols was used for tape analysis; this "addition" is not really a method addition, but more of an administrative improvement to have the protocol documented in one place together.
TR-160: No changes.
TR-170: No changes.
TR-190: Individual Steps of Protocol – Paragraph 4. changed "Forensic Chemistry" to "Trace and/or Controlled Substances".

6	12-31-2022	Lab Director change. **TR-01**: No changes. **TR-06**: Standards – Removed Glass Standard. Controls – Added liquid nitrogen instructions and added instructions for unsatisfactory standard. Individual Steps – Cleaned up language. **TR-07**: Individual Steps – Removed quartz wedge. **TR-08**: Protocol Notes – Added 8. Regarding submitting photos as evidence. Recommended Report Wording – Added wording regarding reporting of evidence collected (first paragraph) and last two paragraphs regarding searches with no evidence collection and photography only and evidence in condition not suitable for searching. **TR-12**: No changes. **TR-15**: No changes. **TR-16**: Rescinded. **TR-18**: Individual Steps – 1. Clarified when photos should be taken. 2. Removed requirement to photograph all containers and restructured paragraph 2a. Paragraph 2d added "Digital" Paragraph 2e. Added exceptions to when case information does not have to be in the digital image. Added Recommended Report Wording section and included wording for when photography is the only analysis. **TR-19**: Individual Steps – Moved step for controls/checks to Controls section. Added steps for using MSP software and instrument for transmission and fluorescence. Protocol Notes – Added new paragraphs 1 & 2 regarding analysis by transmission and fluorescence and sample colors. **TR-23**: No changes. **TR-100**: Incorporated deviation from 04-20-22 for standards and SAM. Incorporated deviation from 09-22-22 adding Evaluation Criteria section. Standards – Added to paragraph 6 giving more direction regarding blanks and adding blank after every fourth injection in a case. Approved Methods – Corrected IL20 to Light20 in 2. Individual Steps – Removed 3. Regarding punching a hole in the can and renumbered. Protocol Notes – added paragraphs 8 & 9 for carbon strips and Beast documentation requirements. Recommended Report Wording – changed recommended wording in paragraph 2.

History

		TR-101: No changes. **TR-110**: Individual Steps – Added marking the stub in paragraph 1. Protocol Notes – Added 2. Regarding victims' clothing not being analyzed. Moved paragraph 5 to 3, to correspond with new paragraph 2. Added new paragraph 6, regarding duplicate data not being considered rejected data. Recommended Report Wording – incorporated deviation from 01-05-22. **TR-130**: No changes. **TR-150**: No changes. **TR-160**: Protocol Notes – Word smithing paragraph 1. **TR-170**: Protocol Notes – Word smithing paragraph 1. **TR-190**: Individual Steps – Paragraph 1 clarified requirements for a plan.
7	12-31-2023	Changed Technical Manager, added line for Quality Manager approval. **TR-01**: Controls – Removed "corrective" and changed "before case work can be reported" to "before casework testing can commence". Recommended Report Wording – changed "writing" to "wording" in the second paragraph. **TR-06**: Controls – added "should be". Protocol Notes – Added 7. Regarding use of reference library for items other than fibers. Recommended Report Wording – changed "writing" to "wording" in the second paragraph. **TR-07**: Recommended Report Wording – changed "writing" to "wording" in the second paragraph. **TR-08**: Individual Steps – 4. Removed unnecessary "and" from "photograph and the condition". **TR-12**: Principle – Updated model number from II to V. Apparatus & Materials – 1. Updated model number from II to V. **TR-19**: Principle – Corrected spelling of qualitative. Controls – changed "Reference Filter" to "Reference Slide". Individual Steps – 4.a. changed "RL" to "TL" and "reflected light" to "transmitted light". 4.e.i. changed "Reflectance" to "Transmission" and "RL" to "TL" and removed italicized statement regarding transmission light. Protocol Notes – 1. Added "paint samples" to fluorescence statement. Recommended Report Wording – changed "writing" to "wording". **TR-23**: Controls and Standards – removed "user setting". Recommended Report Wording – added clarification to documentation and changed "writing" to "wording" in the last paragraph. Protocol Notes – 2. Changed ";" to ",". Recommended Report Wording – changed "writing" to "wording" in last paragraph. **TR-100**: Individual Steps – Added statement regarding sampling. **TR-101**: Individual Steps – Proper handling of carbon-strips 2. changed "picked by random" to "picked at random". Added statement regarding sampling.

History

		TR-110: Individual Steps – 5. Added clarification on when the positive known sample will be analyzed. 6. – Added clarification regarding confirmation of subsequent analysis of the known sample. Added 8. Regarding conclusions on PDFs. 9. – Section renumbered. Added 10. Regarding analysis of clothing. Added 11. Regarding stubs with possible blood. Protocol Notes – Added 9. Regarding required PDFs for case files. Interpretation of Test Data – 2. Changed "above mentioned" to "above-mentioned" and changed "primer based" to "primer-based". Header Recommended Report Writing changed to Recommended Report Wording to be consistent throughout manual. 2. Reworded recommended wording. 3. Changed to reflect not analyzed because limit of detection was reached. Added 12. Regarding stub with possible blood. **TR-150:** Individual Steps – 3. Statements added at the beginning to address process of analysis. 3.a added "within the tape:" and reformatted to include 3.a.i. 3.b. renumbered to 3.a.ii. 3.c. renumbered to 3.a.iii. 3.d. renumbered to 3.b. 3.e. renumbered to 3.c. 3.f. renumbered to 3.d. and added "backed" for clarification. 3.g. renumbered to 3.e. 3.e.ii. corrected extinction angle from "180" to "90". **TR-190**: Protocol Notes – Added 3.g. GC/MS.
8	11-30-2024	Annual review. **TOC:** Removed "Cotton and Manufactured" from Fiber Analysis. **TR-06:** Associated Protocols – edited title of TR-170 to be consistent with Table of Contents. **TR-07:** Associated Protocols – edited title of TR-170 to be consistent with Table of Contents. **TR-08:** Updated the bureau web address and hyperlink. **TR-15:** Corrected title of header on the second page from TR-16 to TR-15 Elemental Analysis. **TR-19:** Associated Protocols – edited title of TR-170 to be consistent with Table of Contents. **TR-101:** Associated Protocols – edited title of TR-1 to be consistent with Table of Contents. **TR-150:** Associated Protocols – edited titles of TR-06, TR-07, TR-19 and TR-170 to be consistent with Table of Contents. **TR-160:** Associated Protocols – edited titles of TR-06, TR-07 and TR-19 to be consistent with Table of Contents. **TR-170:** Removed "Cotton and Manufactured" from title, updated headers throughout. Principle of the Protocol: replaced "cotton" with natural and "manufactured" with synthetic in the first sentence. Associated Protocols – edited titles of TR-07 and TR-19 to be consistent with Table of Contents. **Approval:** Removed Quality Manager.
9	12-31-2025	Annual review. Changed Supervisor/Technical Manager. **TOC:** Changed "Physical Match" to Physical Fit.

History

TR-08: Associated Protocols – changed "Physical Match" to Physical Fit and changed throughout protocol.
TR-15: Recommended Report Wording: removed mention of iodine as it is unable to be analyzed on our instruments.
TR-18: Individual Steps – changed "physical match" to physical fit. Added "tapes" to comparative case types. Recommended Report Wording: added wording options for when other analysis is performed. Corrected header for pg. 20 to the correct protocol name and manual information.
TR-23: Protocol Notes: 2. Removed extra up from sentence.
TR-100: References – corrected title case of ASTM E1618, removed "Committee E-30".
TR-110: Recommended Report Wording: 6. Removed "per OSBI Trace protocol". 7. Removed "per OSBI Trace protocol".
TR-130: Changed "Physical Match" to Physical Fit in title, headers and changed throughout protocol.
TR-150: Associated Protocols – added TR-23 and changed "Physical Match" to Physical Fit and changed throughout protocol.
TR-160: Changed "Physical Match" to Physical Fit and changed throughout protocol. Individual Steps – 5. Corrected "Database" to "Data". Recommended Report Wording: Added 2. Regarding report wording for when a physical match is not found. All subsequent steps were renumbered.
TR-170: Principle of the Protocol – changed "physical match" to physical fit and changed throughout protocol.