

Department of Forensic Science

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TRACE EVIDENCE PROCEDURES MANUAL OF FORENSIC SCIENCE

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1 INTRODUCTION

- 1.1** Trace Evidence examinations comprise a wide variety of evidence types. This manual consists of the procedures most routinely used to analyze evidence submitted to the Trace Evidence (TE) Section (the Section) of the Virginia Department of Forensic Science (the Department, DFS). This manual, in combination with the Section Training Manual, provides the basis for effective quality management of analyses. The Department's Quality Manual (QM) and Safety Manual provide additional guidelines.
- 1.2** This manual is not all-inclusive. Its purpose is to provide a framework of analytical procedures for the Trace Evidence Examiner. Every effort has been made to document the routine procedures used by the Section and to provide appropriate references. Many procedures have been adapted from standard laboratory practices and, therefore, no specific reference may be available. For examinations that involve the comparison of an unknown to a known, the unknown item(s) shall be evaluated to identify general characteristics suitable for comparison prior to examination of the known item(s). The examiner's case notes must document the extent to which this evaluation was performed.
- 1.3** Every case is unique and must be evaluated by the individual examiner. Not all possible analyses that may be encountered in casework can be appropriately covered in a procedures manual nor can all possible variations to a described procedure be included. It is always the examiner's responsibility to choose the best analytical scheme for each individual case, particularly for evidence not routinely encountered. It is expected that Supervisors or the Chemistry Program Manager will be consulted for extraordinary procedures and that deviations from existing procedures shall occur in accordance with the Department's QM.
- 1.3.1** New methods must be validated before use. Published methods must be verified to work in each Regional Laboratory before use. Prior to beginning a validation process, consult the Chemistry Program Manager for an appropriate validation plan.
- 1.4** Reference collections of data or materials used for identification, comparison or interpretation shall be fully documented, uniquely identified and properly controlled.
- 1.4.1** Purchased reference collections of data or materials are obtained with documentation and unique identification of the data or materials.
- 1.4.1.1** Examples of these types of reference collections include, but are not limited to:
- GC-MS Libraries, both electronic and hard copy (e.g., NIST, Wiley, EPA/NIH)
 - FTIR Libraries, both electronic and hard copy (e.g., Sadtler, Aldrich, Hummel, Georgia State)
 - Microtrace Forensic Fiber Reference Collection
 - Collaborative Testing Services, Inc. Reference Collection of Synthetic Fibers
 - Scientific Polymer Products, Polymer Sample Kits
 - Locke Certified Glass Standards
 - Supelco Inc. or Alltech/Applied Science Hydrocarbon and Alcohol Kits
 - Collaborative Testing Automotive Paints
 - PDQ Paint Data Query paint database (not purchased, obtained from the RCMP)
 - Automotive Refinish Color Collection
 - NCFS Ignitable Liquids Reference Collection
- 1.4.2** In-house reference collections of materials shall be uniquely identified.
- 1.4.2.1** Purchased or donated materials may be identified by name and a laboratory identification number. New entries shall have the name, laboratory identification number, source of material and the date and initials of the person who obtained the material.

Current in-house reference collections of materials include:

- Ignitable Liquids (Statewide)
- Tapes (Central)
- Pigment Powder (Central)
- Explosives (Central)
- Fibers (Central)

1.4.2.1.1 Data generated and retained from these products must be correlated to the specific product with the laboratory identification number.

1.4.2.1.2 A spreadsheet inventory of the contents of these reference collections will be maintained on the Trace Evidence Shared Folders.

1.4.2.1.3 All examiners in the Section are responsible for properly labeling and entering new reference collection items into the spreadsheet. See ¶ 1.4.2.2.1 for the procedure for statewide collections.

1.4.2.2 All collections distributed and used by examiners statewide (e.g., the Ignitable Liquids Reference Collection), shall have the same unique identifiers.

1.4.2.2.1 The Central Laboratory Section Supervisor shall designate an examiner to be responsible for a statewide reference collection. The examiner will be responsible for assigning laboratory identification numbers, entering new items into the spreadsheet and distributing aliquots of properly labeled reference material (and associated data) to each regional laboratory in a timely fashion.

1.4.2.2.2 Regional laboratory staff shall request laboratory identification numbers from the designated examiner and promptly forward new items for inclusion into the statewide reference collection to the designee.

1.4.2.3 These collections are properly controlled by limiting their access to Trace Evidence personnel.

1.4.3 In-house data libraries are considered reference collections and are found on the following instruments:

- GC/MS
- FTIR
- XRD

1.4.3.1 The following information, if applicable, shall be included with each new entry into in-house data libraries:

- Compound/Product name
- Lot number or laboratory identification number
- Date
- Initials of person entering data

1.4.3.2 For in-house libraries, each entry is automatically identified by a unique tracking number generated by the instrument software.

1.4.3.3 In-house libraries shall be generated or modified either by an instrument operator or by a designee of the Section Supervisor.

1.4.4 A list of Trace Evidence reference collections is maintained and is available to Section personnel on the network drive in Trace Evidence Shared Folders.

- 1.4.5 Materials used in casework for comparison shall be documented in the case notes.
- 1.4.6 Materials that are not a part of a reference collection but that are retained for training, display or other purposes must be clearly marked as such.
- 1.5** Worksheets are provided as controlled forms and should be used as designed. Examiners are reminded to take appropriate notes which will allow for another examiner/supervisor to evaluate the data, interpret the results and come to the same conclusion; and also be able to repeat the analysis under conditions as close as possible to the original. There may be times where plain paper may be useful for note taking. This is an acceptable practice as long as the evidence description and pertinent information regarding tests performed are recorded.
- 1.6** Evidence sample selection is very much dependent upon the type and amount of evidence received and the examination(s) requested. If no specific selection guideline is mentioned in this manual, it is the examiner's responsibility to ensure that the case file notes explain how/why samples were selected for analysis, when appropriate. Reported conclusions shall be based upon the samples selected and examined.
- 1.7** Internet references included in case file documentation will at a minimum contain the website address and the date accessed/printed.
- 1.8** The case file notes will contain documentation as to what types of materials were generated during the analysis (e.g., 16 Post-it type notes used to recover hairs/fibers from Item 21) and how they are being packaged for return. (e.g., 3 round plastic boxes containing debris from Item 5 placed in a sealed plastic bag and returned to Container 5.)
- 1.9** If a test result or observation is rejected, the reason for the rejection shall be documented. In addition, the identity of the individual taking the action and the date shall be documented.
- 1.10** Report (CoA) wording examples are included in the individual sections of this manual. When drafting report wording for evidence types not listed or when specific examples do not appear for a particular type of evidence, look first to existing wording in the other sections that may be applied to the current situation.
- 1.10.1 Reports may not use non-specific terms (e.g., "consistent with", "highly specific", "similar to", "indicative of", or "characteristic of") without qualification (to include, for example, "*physically* and *chemically* indicative of...") or stating shared properties.
- 1.10.2 The laboratory analysis conducted and/or instrumentation used for examination will be included in the report. This information may be summarized and will appear only once if all items are examined using the same methods and/or instrumentation. Otherwise, mention those used for each item separately.
- Item ____ was examined visually and using [stereomicroscopy, polarized light microscopy (PLM), compound microscopy, comparison microscopy, fluorescence microscopy, a pH meter, a balance, a digital caliper, an ignition test, a multimeter, microsolvability tests, microchemical tests, polarizing films, ultraviolet light (UV), Microspectrophotometry (MSP), Fourier Transform Infrared Spectrophotometry (FTIR), Gas Chromatography (GC), Gas Chromatography-Mass Spectrometry (GC-MS), Ion Chromatography (IC), X-Ray Diffraction (XRD), Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS), Laser Induced Breakdown Spectroscopy (LIBS), and Glass Refractive Index Measurement System (GRIM3)].
- 1.11** Disagreements, including documentation requirements, shall be reconciled first by discussion between the parties involved. The Section/Group Supervisor may be called upon to review the situation and make a decision at that time. The Chemistry Program Manager would become involved if resolution is still pending. The Technical Services Director would be the final decision maker in the process, if necessary.
- 1.12** Electronic data of examination and QA/QC documentation which also exists as printed, hard copies will be copied periodically from the hard drives to external storage.

- 1.13** The Department's laboratory facilities provide sufficient environmental conditions to conduct all tests listed in this Procedures Manual with no further consideration required.
- 1.14** Effective separation is maintained between the SEM-EDS lab and the Firearms and Toolmarks (FX) Section with separate air handling systems for each Section and by not permitting Firearms and Toolmarks personnel in the SEM-EDS lab, by using a sticky mat at the entry to the SEM-EDS lab and changing it as needed, and by being cautious when a Trace Examiner must be present in the FX Section.
- 1.15** Good housekeeping is important when analyzing evidence particularly when the evidence is present at a "trace" or microscopic level.
- 1.15.1 Ensure that cleanliness is maintained throughout the laboratory areas.
- 1.15.2 Ensure that work areas are cleaned prior to evidence being retrieved and/or opened.

1.16 Instrumentation and Equipment – General Guidelines

- 1.16.1 After an instrument/equipment has been shut down or significant maintenance has been performed, verify that the instrument/equipment is fit for use by analyzing QA samples appropriate for that instrument. Ensure the instrument performs as expected before returning the instrument to service for casework.
- 1.16.1.1 Retain instrument verification documentation.
- 1.16.2 Any instrument or equipment that has been subjected to overloading or mishandling, gives suspect results, or has been shown to be defective or outside specified limits, shall be taken out of service.
- 1.16.2.1 The instrument or equipment shall be clearly labeled as out of service.
- 1.16.2.2 The effect of the defect or departure from specified limits on previously analyzed case samples will be examined and any non-conforming work will be addressed following the Department's QM.
- 1.16.2.3 Once the issue has been addressed, verify that the instrument/equipment performs as expected by analyzing QA samples appropriate for that instrument before returning the instrument/equipment to service for casework.
- 1.16.2.4 Retain instrument verification documentation.
- 1.16.3 Replacement Instrument Installation
- 1.16.3.1 Obtain documentation from the instrument service representative that demonstrates that the instrument performs to the manufacturer's specification.
- 1.16.3.2 Load methods, macros, libraries or other software as appropriate and test their functionality.
- 1.16.3.3 Verify that the instrument is fit for use by analyzing QA samples appropriate for that instrument. Ensure the instrument response is appropriate before placing the instrument in service for casework.
- 1.16.3.3.1 A summary of the verification shall be sent to the Chemistry Program Manager for approval prior to placing the replacement instrument into service.
- 1.16.3.3.2 Copy methods, macros and other information, as appropriate, to suitable long-term storage media as a back-up to the system.
- 1.16.3.3.3 Retain instrument verification documentation.

1.16.3.4 Ensure that examiners using the replacement instrument have been trained and authorized to use the instrumentation prior to use in casework.

1.16.3.4.1 The Chemistry Program Manager will either devise the training and competency check or the examiner responsible for the instrumentation will draft this for the Chemistry Program Manager's approval prior to proceeding.

1.16.4 New Instrument Installation and Validation

1.16.4.1 Obtain documentation from the instrument service representative that demonstrates that the instrument performs to the manufacturer's specification.

1.16.4.2 The new instrument must be validated for use with samples similar to evidentiary material examined by Trace Evidence personnel.

1.16.4.3 The new instrument validation procedure will be determined in conjunction with the Chemistry Program Manager and will not proceed without the Chemistry Program Manager's approval.

1.16.5 Documentation

1.16.5.1 A log shall be maintained for each item of instrumentation and equipment (and its software) significant to the tests performed. These records shall include at a minimum:

- The identity of the item of equipment and its software
- The manufacturer's name, type identification and serial number or other unique identification (include DFS inventory number)
- Checks that the equipment complies with laboratory specifications
- The current location, where appropriate
- The manufacturer's instructions, if available, or reference their location
- Dates, results and copies of reports and certificates of all calibration, adjustments, acceptance criteria, and the due date of next calibration
- The maintenance plan, where appropriate, and maintenance carried out to date
- Any damage, malfunction, modification or repair to the equipment

1.17 Evidence Storage

1.17.1 Evidence in Section storage in Section Administrative Custody (e.g., X/TE/ADM) is considered to be in long-term storage and must be stored properly sealed.

1.17.2 Evidence in an examiner's personal custody may either be stored in the examiner's personal evidence locker(s), in a room with restricted access (e.g., Scraping Room) or in Section storage.

1.17.2.1 Personal custody evidence stored in a Section storage area must be placed in an area clearly labeled as the personal custody of the particular examiner.

1.17.2.2 Whenever evidence is not actively being analyzed it must be sealed.

1.17.2.2.1 The time frame for evidence considered to be actively being analyzed should generally not exceed 30 days.

1.17.2.2.2 Evidence for a case waiting for instrument support results is considered to be actively being analyzed.

1.17.2.3 Evidence stored in a room with limited access does not have to be sealed.

1.17.2.3.1 Evidence does not need to be returned to a container but it must be protected.

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2 **EXPLOSIVES****2.1 Analytical Approach**

- 2.1.1 Perform a detailed physical exam. Based upon these observations, the examiner will decide which analytical procedures will be used.
- 2.1.2 Record observations using one or more Explosives Worksheets, as applicable.
- 2.1.3 If unconsumed low explosives powder (black powder, black powder substitute, smokeless powder) is present, proceed to Section 2.5, Analysis of Unconsumed Low Explosives Powder.
- 2.1.4 If unconsumed flash powder is present, follow the procedure for the analysis of unconsumed low explosives powder but add SEM-EDS.
- 2.1.5 If unconsumed pyrotechnic (fireworks) powder is present follow the procedure for the analysis of unconsumed low explosives powder but add SEM-EDS. IC may be added if necessary.
- 2.1.6 If no unconsumed powder is present/identified, proceed with extractions in the following order:
- 2.1.6.1 Methanol extracts monomethylamine nitrate (MMAN). If water gel explosives are not suspected, this step may be omitted.
 - 2.1.6.2 Ether extracts nitroglycerin (NG). Use only when nitroglycerin identification or comparison is needed for suspected double-base smokeless powders or NG-containing dynamite.
 - 2.1.6.3 Acetone extracts most organic explosives to include nitrocellulose (NC).
 - 2.1.6.4 Water extracts inorganic explosives.
 - 2.1.6.5 Carbon disulfide (CS₂) or toluene extracts free sulfur. (Optional)
 - 2.1.6.6 Allow the material being extracted to dry thoroughly between extractions.
 - 2.1.6.7 Remove a portion of the water extract for IC analysis, if appropriate.
 - 2.1.6.8 Perform microchemical and instrumental analysis on the extracts themselves or the dried residue from the extractions.
- 2.1.7 If a large amount of consumed powder residue is present, the examiner may choose to omit extractions and perform microchemical and appropriate instrumental analysis on the residue directly.
- 2.1.8 Any remaining extract and/or extracted residue is retained and returned in the item container corresponding to that particular item, unless otherwise indicated.
- 2.1.9 Microchemical results generally are noted on the Explosives Worksheet. Instrumental results are compared to known standards and/or standard data files, and both are retained in the case file.
- 2.1.10 Chemical reaction devices are a unique subset of improvised devices which are discussed in a separate section. (Section 2.3)
- 2.1.11 **Safety Considerations: The utmost caution is required when handling explosive materials and devices.**
- 2.1.11.1 Explosive devices must be dismantled or rendered safe before submission. If a live device is encountered, safely secure it and the laboratory area, notify the appropriate supervisory staff and call the submitting agency for immediate removal from the laboratory.

- 2.1.11.2 If there are any questions regarding the stability of an explosive or its safety, notify the appropriate supervisory staff who may need to contact the nearest bomb disposal unit.
- 2.1.11.3 Analyze only small quantities of material.
- 2.1.11.4 Do not work near sources of heat or fire.
- 2.1.11.5 Do not subject material to violent shocks.
- 2.1.11.6 Store material away from heat, fire and other explosives or accelerants.
- 2.1.12 Minimum Standards and Controls
 - 2.1.12.1 Reagent reliability checks will be performed using the known materials specified on the Reagent Log and recorded on the Explosives Worksheet.
 - 2.1.12.2 Solvents used for extractions should be a high quality, low residue solvent (e.g., HPLC grade, OMNISOLV, OPTIMA).
 - 2.1.12.3 Solvent blanks must be run with each case extracted.

2.2 Physical Examination

2.2.1 Purpose

The purpose of the physical examination is to observe, document and recover materials for analysis. In addition to other materials, the search includes recovery of any unconsumed explosives powder. The information derived from this examination is necessary for determining the appropriate analytical procedure. It may also be beneficial to the submitting agency in providing investigative direction.

2.2.2 Analytical Procedures

- 2.2.2.1 If a fingerprint examination is requested, handle evidence appropriately.
- 2.2.2.2 Note type of material present, e.g., cardboard, plastic, paper, pipe, metal, etc.
- 2.2.2.3 Record approximate measurements of any devices.
- 2.2.2.4 Examine evidence for damage by violent force. If fragments are present, note size and shape. Note the presence of threaded fragments and end caps.
- 2.2.2.5 Determine the means of ignition, if possible, by examining evidence for the presence of fuse holes, fuse material, blasting caps and leg wires or electrical circuitry.
 - 2.2.2.5.1 If unconsumed fuse is present, follow the procedure as in Section 2.4.
- 2.2.2.6 Using the stereomicroscope, and/or illuminated magnifier, search the device and/or fragments for particulate or powder material and preserve for further analysis.
- 2.2.2.7 Note any paint or markings. This can include letters, designs, or color-coded marks.
- 2.2.2.8 Record observations on worksheet or in notes.

2.2.3 References

- 2.2.3.1 Yallop, H.J., Explosion Investigation, The Forensic Science Society & Scottish Academic Press, Harrogate, England and Edinburgh, Scotland, 1980.

- 2.2.3.2 Saferstein, Richard, Criminalistics: An Introduction to Forensic Science, ed. 5, Prentice-Hall, Inc.: Englewood Cliffs, NJ, 1995.

2.3 Analysis of Chemical Reaction Devices

2.3.1 Purpose

- 2.3.1.1 The purpose of analyzing chemical reaction devices is to determine the composition of the components of what is most commonly referred to as a bottle bomb.
- 2.3.1.2 Physical indications of a device that functioned in the absence of any chemical residue may indicate a dry ice (carbon dioxide) or liquid nitrogen bottle bomb.

2.3.2 Analytical Procedures

- 2.3.2.1 Note condition of the container, e.g., ruptured, distorted, capped, uncapped, and the amount and condition (e.g., reacted and/or unreacted strips, balls, twists) of any foil present.
- 2.3.2.2 Follow the General Chemical Procedure for Acids and Bases in Section 6.3.
- 2.3.2.3 Analyze recovered liquid or water extracts by IC and SEM-EDS.
- 2.3.2.3.1 Alternatively, the precipitate from the silver nitrate or barium chloride microchemical test may be dried and used for SEM-EDS. Also, include a silver nitrate or barium chloride reagent blank.
- 2.3.2.4 Analyze intact or partially consumed “aluminum” foil by SEM-EDS.

2.4 Analysis of Unconsumed Pyrotechnic Safety Fuse

2.4.1 Purpose

The purpose of analyzing unconsumed pyrotechnic safety fuse is to observe its physical properties and determine its functionality.

2.4.2 Analytical Procedures

- 2.4.2.1 Describe the physical appearance of the fuse, noting the color of its core, the twist of its fibers and the presence of an outer plastic layer and its color.
- 2.4.2.2 Measure and record the diameter of the fuse.
- 2.4.2.3 Perform an ignition test on the fuse (see Section 2.9).

2.5 Analysis of Unconsumed Low Explosive Powder

2.5.1 Purpose

The purpose of analyzing unconsumed low explosive powder is to determine its chemical composition for identification or classification purposes. This is generally applicable to black powder, black powder substitutes, smokeless powder, flash powders and pyrotechnic powders.

2.5.2 Analytical Procedures

- 2.5.2.1 If any unconsumed powder is recovered from an expended device, no extractions will be performed unless there is an indication of a mixture of filler materials from the examiner’s observations or information derived from the RFLE.

- 2.5.2.2 Weigh the powder, unless trace amount. Alternatively, if there are only a few particles of unconsumed powder present, instead of weighing, count the particles and make a note of how many particles are consumed during analysis.
- 2.5.2.3 Describe the physical appearance of the powder.
- 2.5.2.4 Perform an ignition test on the powder, Section 2.9.
- 2.5.2.5 Perform microchemical tests on the powder, Section 2.8.

As a minimum:

- If suspected smokeless powder- DPA
- If suspected black powder or black powder substitute- DPA, Triphenylselenium chloride (while supplies last), Ascorbic Acid Test Strip
- If suspected flash powder or pyrotechnic mixture- DPA, Triphenylselenium Chloride (while supplies last)

- 2.5.2.6 Perform FTIR instrumental analysis on the powder, utilizing one of the following sample preparation techniques:

- KBr
- The micro compression cell with diamond windows
- From acetone on KBr in transmittance mode
- ATR

- 2.5.2.7 In lieu of FTIR, XRD may be used for powders other than smokeless powder.

- 2.5.2.8 If the powder is smokeless powder and a comparison has been requested, determine if it is single or double base.

- 2.5.2.8.1 Take an amount of the powder particles (determined by examiner discretion, particle size, and/or total sample amount) and extract it with approximately 200 μL of methylene chloride or acetone for approximately 90 minutes, with occasional vortexing.

- 2.5.2.8.1.1 The amount of solvent used for extraction may be adjusted accordingly for small amounts of powder. For example, a volume of 50 μL of solvent may be more appropriate for the extraction of one or two particles.

- 2.5.2.8.2 Analyze the extract using GC-MS.

- 2.5.2.8.2.1 The **absence** of nitroglycerin in the extract combined with an FTIR spectrum consistent with smokeless powder identifies the powder as **single base**.

- 2.5.2.8.2.2 The **presence** of nitroglycerin in the extract combined with an FTIR spectrum consistent with smokeless powder identifies the powder as **double base**.

- 2.5.2.9 If a comparison of powders is requested, additional steps are necessary.

- 2.5.2.9.1 Measurements of the dimensions of smokeless powders will be recorded. For other powders, determine the approximate grain size.

- 2.5.2.9.1.1 Measurements should be taken with a digital caliper.
- 2.5.2.9.1.2 Sample selection for measurement of disk and cylinder smokeless powders:
- 2.5.2.9.1.2.1 Measure the diameter and the length or thickness of at least 10 randomly chosen particles, if available.
 - 2.5.2.9.1.2.2 The visually largest and smallest particles should be selectively measured in comparison to the average diameter or length (cylinders only) to establish a range of values.
 - 2.5.2.9.1.2.3 The range of values established should be used for the comparison.
 - 2.5.2.9.1.2.4 Questioned smokeless powder particles with an observed physical dimension range that overlaps with the observed physical dimension range of the known smokeless powder sample(s) are considered to be consistent in physical dimensions.
- 2.5.2.9.1.3 Sample selection for measurement of ball, flattened-ball, and other types of smokeless powders:
- 2.5.2.9.1.3.1 Diameter and thickness of ball-type smokeless powders will vary somewhat within a similar lot. Ball-type powders include spherical, partially flattened, or mostly flattened particles. The diameter and thickness of other types of powders may vary considerably.
 - 2.5.2.9.1.3.2 Measure the diameter and thickness (except for spherical balls) of at least 20 randomly selected particles, if available.
 - 2.5.2.9.1.3.3 The visually largest and smallest particles should be selectively measured in comparison to the average diameter to establish a range of values.
 - 2.5.2.9.1.3.4 The range of values established should be used for the comparison.
 - 2.5.2.9.1.3.5 Questioned smokeless powder particles with an observed physical dimension range that overlaps with the observed physical dimension range of the known smokeless powder sample(s) are considered to be consistent in physical dimensions.
- 2.5.2.9.2 The comparison will include minimal microchemical tests and the ignition test as described in the above procedure.

- 2.5.2.9.3 If similar, GC-MS shall be used to compare patterns and the mass spectra of the components.
- 2.5.2.9.4 For other powders, a comparison of the results of IC, SEM-EDS and XRD testing should be conducted.

2.5.2.10 When smokeless powder particles are recovered from a disrupted device and a disruptor round is also submitted, use procedure as in Section 2.5.2.9.

2.5.3 References

- 2.5.3.1 Parker, R.G., McOwen, J.M., and Cherolis, J.A., "Analysis of Explosives and Explosive Residues. Part 2: "Thin-Layer Chromatography", Journal of Forensic Sciences, Vol. 20, No. 1, April 1975, pp. 254-256.

2.6 Analysis of Intact High Explosives

2.6.1 Analytical Approach

2.6.1.1 While the Section receives some explosives evidence types on a fairly routine basis, others may require method development at the time the case is being worked. This section provides analysis schemes for some of those that the Section has analyzed with some regularity.

2.6.1.2 Good scientific principles and a logical analysis scheme are applied to those high explosives that have not been encountered before. The examiner, in consultation with the Section Supervisor, will devise an analytical approach that will take into consideration the type and quantity of evidence submitted and the case information received. Generally speaking, a visual macroscopic/microscopic examination will be performed (stereo, compound, comparison and/or polarizing microscopes) for color, texture, viscosity, morphology, optical properties and homogeneity.

2.6.1.3 The amount of sample will determine how the examiner proceeds. With a limited amount of sample, nondestructive testing is performed first. Solubility/miscibility tests with organic/inorganic and polar/nonpolar solvents (e.g., water, carbon disulfide, pentane, acetone, chloroform) should be conducted. The pH of water soluble/miscible samples should be taken using pH paper or a pH meter. Microchemical tests should be performed as indicators of a class of compounds present (e.g., for sulfates, chlorides, nitrates, sugars).

2.6.1.4 Necessary instrumentation is used to identify or to compare samples.

2.6.2 Emulsion Explosives

2.6.2.1 Purpose

The purpose of analyzing emulsion explosives is to determine their physical and chemical composition for identification purposes. Emulsion explosives are typically composed of inorganic oxidizers (ammonium nitrate, sodium nitrate, and/or sodium perchlorate), hydrocarbon oils or waxes, glass or plastic microballoon sensitizers, and an emulsifier. They can also contain NG, NC, EGDN, or aluminum.

2.6.2.2 Analytical Procedures

2.6.2.2.1 Weigh the material.

2.6.2.2.2 Describe the physical appearance of the material.

2.6.2.2.3 As emulsion explosives typically contain an oil, rinse a small amount of the material with pentane, collecting both the pentane and the remaining material.

2.6.2.2.3.1 The pentane extract is analyzed via GC-MS.

2.6.2.2.4 Remaining material can be analyzed via microscope, microchemical tests, FTIR, XRD, SEM-EDS, and/or IC.

2.6.2.2.5 Any microballoons present are viewed on the polarized light microscope to determine if they are composed of glass or plastic.

2.6.2.2.6 If the presence of NG or NC is suspected, rinse a small amount of the material with ether or acetone, respectively.

2.6.2.3 References

2.6.2.3.1 Nakamura, J., et al., The instrumental analysis of emulsion explosive residues. In: Midkiff, C. R., editor. Proceedings of the 5th International Symposium on the Analysis and Detection of Explosives; 1995 Dec 4-8; Washington, D.C. Washington, D.C.: Bureau of Alcohol, Tobacco, Firearms and Explosives; 1997.

2.6.2.3.2 Lau, K. L. M., et al., Pre-blast and post-blast examination of emulsion explosives. In: Midkiff, C. R., editor. Proceedings of the 5th International Symposium on the Analysis and Detection of Explosives; 1995 Dec 4-8; Washington, D.C. Washington, D.C.: Bureau of Alcohol, Tobacco, Firearms and Explosives; 1997.

2.6.2.3.3 Yinon, J. and Zitrin, S., Modern Methods & Applications In Analysis of Explosives, John Wiley & Sons, Inc.: NY, NY, 1993.

2.6.3 Composition C-4

2.6.3.1 Purpose

The purpose of analyzing Composition C-4 is to determine its chemical composition for identification purposes. C-4 is a white putty-like explosive used solely by the military. It contains the high explosive RDX, a plasticizer, a rubber binder, and motor oil.

2.6.3.2 Analytical Procedures

2.6.3.2.1 Weigh the material.

2.6.3.2.2 Describe the physical appearance of the material.

2.6.3.2.3 Follow the extraction procedure outlined in Keto, 1986.

2.6.3.2.4 Examine extracts using appropriate instrumentation to include, but not limited to, GC-MS, XRD, and/or FTIR.

2.6.3.3 References

2.6.3.3.1 Keto, R. O., "Improved Method for the Analysis of the Military Explosive Composition C-4," *Journal of Forensic Sciences*, JFSCA, Vol. 31, No. 1, Jan. 1986, pp. 241-249.

2.6.4 PETN (Pentaerythritol Tetranitrate)

2.6.4.1 Purpose

The purpose of analyzing PETN is to determine its chemical composition for identification purposes. PETN is a white crystalline solid having many military and some nonmilitary applications such as booster charges, demolition charges, and detonating cord, and as the main charge in blasting caps. PETN can be combined with plasticizers to form moldable sheets and blocks. The explosive Semtex is a plasticized mixture of PETN and RDX; Pentalite is a mixture of PETN and TNT.

2.6.4.2 Analytical Procedures

2.6.4.2.1 Weigh the powder, unless a trace amount or if contained in detonating cord.

2.6.4.2.2 Describe the physical appearance of the powder.

2.6.4.2.3 Perform microchemical tests on the powder. As a minimum:

- DPA
- Brucine

2.6.4.2.4 Perform FTIR instrumental analysis on the powder.

2.6.4.2.5 In lieu of FTIR, XRD may be used on unconsumed powder.

2.6.4.3 References

2.6.4.3.1 Yinon, Jehuda and Zitrin, Shimuel, Modern Methods & Applications In Analysis of Explosives, John Wiley & Sons, Inc.: NY, NY, 1993.

2.6.4.3.2 Beveridge, A. D. "Development in the Detection and Identification of Explosives Residues" ; Forensic Science Review, Volume 4, Number 1; June 1992.

2.6.4.3.3 Anger, V., and Feigl, F., Spot Tests in Inorganic Analysis, ed.6., Elsevier Publishing Company: Amsterdam, The Netherlands, 1972.

2.6.4.3.4 Jungreis, Ervin, Spot Tests Analysis, John Wiley and Sons, Inc.:New York, New York, 1985.

2.6.4.3.5 Parker, R.G., Stephenson, M.O., McOwen, J.M., and Cherolis, J.A., "Analysis of Explosives and Explosive Residues. Part I: Chemical Tests", Journal of Forensic Sciences, Vol. 20, No. 1, January 1975, pp. 133-140.

2.7 Extractions

2.7.1 Methanol Extraction

2.7.1.1 Purpose

The purpose of a methanol extraction is to remove any monomethylamine nitrate present in the material being extracted.

2.7.1.2 Minimum Standards and Controls

2.7.1.2.1 Previously cleaned glassware is rinsed with methanol.

- 2.7.1.2.2 A methanol blank must be run at the same time as the debris extract. The same volume of methanol used for extraction of the debris should be used for the blank and the blank should be reduced to the same volume. This blank is compared to the extract from the debris.
- 2.7.1.2.3 If the sample extract is to be filtered, the blank will be filtered in a like manner.
- 2.7.1.2.4 If the sample is to be evaporated, the blank is evaporated a like amount.
- 2.7.1.3 Analytical Procedures
- 2.7.1.3.1 Rinse the material being extracted with methanol in the areas where residue is observed/suspected.
- 2.7.1.3.2 Filter the methanol extract, if necessary, using filter paper or a Pasteur pipette with a plug of glass wool inserted into it.
- 2.7.1.3.3 Decant the methanol into an evaporating dish or beaker and concentrate to several milliliters. Sodium sulfate can be used to remove water from the sample if necessary.
- 2.7.1.3.4 Transfer to a labeled sample vial.
- 2.7.1.3.5 Analyze extract using the appropriate instrumentation.
- 2.7.2 Ether Extraction
- 2.7.2.1 Purpose
- The purpose of an ether extraction is to remove any nitroglycerin from the material being extracted.
- 2.7.2.2 Safety Considerations
- 2.7.2.2.1 Ethyl ether is a very volatile and highly flammable liquid. Do not use near a flame.
- 2.7.2.3 Minimum Standards and Controls
- 2.7.2.3.1 Ether is "washed" with deionized water (to remove any alcohol).
- 2.7.2.3.2 Previously cleaned glassware is rinsed with ether prior to use.
- 2.7.2.3.3 A solvent blank must be run at the same time as the debris extract. The same volume of ether used for extraction of the debris should be used for the blank and the blank should be reduced to the same volume. This blank is compared to the extract from the debris.
- 2.7.2.3.4 If the sample extract is to be filtered, the blank will be filtered in a like manner.
- 2.7.2.3.5 If the sample extract is to be evaporated, the blank will be evaporated a like amount.
- 2.7.2.4 Analytical Procedure
- 2.7.2.4.1 Rinse the material being extracted with ether in the areas where residue is observed/suspected.

- 2.7.2.4.2 Filter the ether extract, if necessary, using filter paper or a Pasteur pipette with a plug of glass wool inserted into it.
- 2.7.2.4.3 Decant the ether into an evaporating dish or beaker and concentrate to several milliliters.
- 2.7.2.4.4 Transfer to a labeled sample vial.
- 2.7.2.4.5 Analyze extract using appropriate instrumentation.

2.7.3 Acetone Extraction

2.7.3.1 Purpose

The purpose of an acetone extraction is to remove any organic explosives, to include nitrocellulose, from the material being extracted. Note: Chloroform can be substituted for acetone on wet samples.

2.7.3.2 Safety Considerations

2.7.3.2.1 Acetone is a very volatile and highly flammable liquid. Do not use near a flame.

2.7.3.3 Minimum Standards and Controls

2.7.3.3.1 If necessary, filter through a drying agent (such as sodium sulfate) to remove excess water.

2.7.3.3.2 Previously cleaned glassware is rinsed with acetone prior to use.

2.7.3.3.3 A solvent blank must be run at the same time as the debris extract. The same volume of acetone used for extraction of the debris should be used for the blank and the blank should be reduced to the same volume. This blank is compared to the extract from the debris. (Note: It is not necessary to analyze this blank on FTIR unless the FTIR spectrum of the sample extract is consistent with containing explosives or explosive residue.)

2.7.3.3.4 If the sample extract is to be filtered, the blank will be filtered in a like manner.

2.7.3.3.5 If the sample extract is to be evaporated, the blank will be evaporated a like amount.

2.7.3.4 Analytical Procedures

2.7.3.4.1 Rinse the material being extracted with acetone in the areas where residue is observed/suspected.

2.7.3.4.2 Filter the acetone extract, if necessary, using filter paper or a Pasteur pipette with a plug of glass wool inserted into it.

2.7.3.4.3 Decant the acetone into an evaporating dish or beaker and concentrate to several milliliters. Sodium sulfate can be used to remove water from the sample if necessary.

2.7.3.4.4 Transfer to a labeled sample vial.

- 2.7.3.4.5 Perform microchemical analysis on acetone extract using the following reagents as a minimum:
- Diphenylamine (extract must be taken to dryness)
 - Acetone and 2N NaOH
 - Brucine (extract must be taken to dryness)
- 2.7.3.4.6 See Section 21.3, Chemicals and Reagents for reagent recipes.
- 2.7.3.4.7 Analyze extract using the appropriate instrumentation. If microchemical tests are negative, no further instrumental analysis is required.

2.7.3.5 Water Extraction

2.7.3.5.1 Purpose

The purpose of a water extraction is to remove any inorganic explosives or explosives residues or sugar(s) from the material being extracted.

2.7.3.5.2 Minimum Standards and Controls

2.7.3.5.2.1 Previously cleaned glassware is rinsed with deionized water prior to use.

2.7.3.5.2.2 A solvent blank must be run at the same time as the debris water extract. The same volume of water used for extraction of the debris should be used for the blank and the blank should be reduced to the same volume. This blank is compared to the extract from the debris.

2.7.3.5.2.3 If the sample extract is to be filtered, the blank will be filtered in a like manner.

2.7.3.5.2.4 If the sample extract is to be evaporated, the blank will be evaporated a like amount.

2.7.3.5.3 Analytical Procedures

2.7.3.5.3.1 Rinse the material being extracted with DI water in the areas where residue is observed/suspected.

2.7.3.5.3.2 Filter the DI water extract, if necessary, using filter paper or a Pasteur pipette with a plug of glass wool inserted into it.

2.7.3.5.3.3 Concentrate sample and blank, if necessary.

2.7.3.5.3.4 A portion of the extract and an equal amount of the blank is evaporated to dryness.

2.7.3.5.3.5 Transfer the liquid extract to a labeled sample vial and any dried residue to a suitable container.

2.7.3.5.3.6 Perform microchemical analysis on extract using the following reagents as a minimum:

- Diphenylamine (extract must be taken to dryness)
- Silver Nitrate plus NH₄OH

- Triphenylselenium chloride (while supplies last)
- Barium Chloride plus HOAc
- Nessler's
- 1-Naphthol

2.7.3.5.3.7 See Section 21.3, Chemicals and Reagents for reagent recipes.

2.7.3.5.3.8 Analyze extracts using the appropriate instrumentation. If all microchemical tests are negative, the extracts may be analyzed by IC and SEM-EDS. If any microchemical tests are positive, the samples should be analyzed by IC, SEM-EDS, and XRD. If there is not sufficient dried residue from the extract for XRD analysis, the residue may be analyzed by FTIR.

2.7.3.6 Carbon Disulfide or Toluene Extraction

2.7.3.6.1 Purpose

The purpose of a carbon disulfide or toluene extraction is to remove free sulfur present in the material being extracted.

2.7.3.6.2 Minimum Standards and Controls

2.7.3.6.2.1 Previously cleaned glassware is rinsed with carbon disulfide or toluene prior to use.

2.7.3.6.2.2 If the sample extract is to be filtered, the blank will be filtered in a like manner.

2.7.3.6.2.3 A solvent blank of the same volume as the sample extract must also be evaporated to dryness. Residue present in the evaporated blank should be compared with the sample residue.

2.7.3.6.3 Analytical Procedures

2.7.3.6.3.1 Rinse the material being extracted with carbon disulfide or toluene in the areas where residue is observed/suspected.

2.7.3.6.3.2 Filter the carbon disulfide or toluene extract, if necessary, using filter paper or a Pasteur pipette with a plug of glass wool inserted into it.

2.7.3.6.3.3 Evaporate to dryness.

2.7.3.6.3.4 Perform microchemical analysis on a portion of the residue using pyridine and NaOH.

2.7.3.6.3.5 If the microchemical test is positive, analyze the extract using SEM-EDS or XRD.

2.7.3.7 References

2.7.3.7.1 Saferstein, Richard, *Criminalistics: An Introduction to Forensic Science*, ed. 5, Prentice-Hall, Inc.: Englewood Cliffs, NJ, 1995.

2.7.3.7.2 Yinon, Jehuda and Zitrin, Shimuel, *Modern Methods & Applications In Analysis of Explosives*, John Wiley & Sons, Inc.: NY, NY, 1993.

- 2.7.3.7.3 Parker, R.G., "Analysis of Explosives and Explosive Residues. Part 3: "Monomethylamine Nitrate", *Journal of Forensic Sciences*, Vol. 20, No. 2, April 1975, pp. 257-260.
- 2.7.3.7.4 Midkiff, Charles R., "Arson and Explosive Investigation", *Forensic Science Handbook*, Volume 1, Second Edition, Pearson Education, Inc., 2002, pp. 510-524.

2.8 Microchemical Spot Tests

2.8.1 Purpose

Microchemical spot tests are used as a screening method for compounds or ions commonly found in explosives or explosives residues.

2.8.2 Safety Considerations

2.8.2.1 Care should be taken to minimize exposure to these reagents. The process may be carried out in a well-ventilated area or by using a "Nederman" point-of-use vent, if one is available.

2.8.2.2 Keep the quantity of reagent used to a minimum.

2.8.3 Minimum Standards and Controls

2.8.3.1 Reagents are tested with appropriate controls when being used for casework.

2.8.3.2 Reagent blanks are run using those microchemical spot tests that gave a positive result for the sample.

2.8.3.2.1 If a color develops in the reagent blank, the test should be repeated.

2.8.3.2.1.1 If the results of the second blank are acceptable, all samples should be re-run.

2.8.3.2.1.2 If the results of the second blank are unacceptable the examiner should take steps to resolve the issue (e.g., replacing the solvent in the bottle, checking the reagents) prior to re-sampling and any further analysis.

2.8.4 Analytical Procedures

2.8.4.1 The test reagent should be added to a spot plate or test tube first, and then the sample added. This practice determines if the plate or tube was clean before the analysis.

2.8.4.1.1 If a reaction occurs prior to the addition of the sample, the plate or tube shall be discarded or cleaned before testing the sample.

2.8.4.2 Add the sample to be tested to the spot plate or test tube.

2.8.4.3 Record any reaction, or lack thereof, using a stereo microscope to observe any changes as necessary.

2.8.5 References

2.8.5.1 Anger, V., and Feigl, F., *Spot Tests in Inorganic Analysis*, ed.6., Elsevier Publishing Company: Amsterdam, The Netherlands, 1972.

- 2.8.5.2 Jungreis, Ervin, Spot Tests Analysis, John Wiley and Sons, Inc.:New York, New York, 1985.
- 2.8.5.3 Parker, R.G., Stephenson, M.O., McOwen, J.M., and Cherolis, J.A., "Analysis of Explosives and Explosive Residues. Part I: Chemical Tests", *Journal of Forensic Sciences*, Vol. 20, No. 1, January 1975, pp. 133-140.
- 2.8.5.4 Yinon, Jehuda and Zitrin, Shimuel, Modern Methods & Applications In Analysis of Explosives, John Wiley & Sons, Inc.: NY, NY, 1993.

2.9 Ignition Test

2.9.1 Purpose

The purpose of the ignition test is to determine how the suspected explosive powder or unconsumed fuse behaves when ignited.

2.9.2 Safety Considerations

2.9.2.1 Only use a small amount of material.

2.9.3 Minimum Standards and Controls

2.9.3.1 The examiner should have observed ignition tests of known explosives and fuses prior to conducting the test on the case sample(s).

2.9.4 Analytical Procedures

2.9.4.1 For suspected explosive powder, a small portion of the sample may be held over a flame using a spatula or placed in a glass container and then ignited.

2.9.4.1.1 Alternatively, some of the sample may be placed in a Kimwipe and ignited in a glass container.

2.9.4.1.2 If trace amounts of sample are present, then the examiner may elect to not conduct this test.

2.9.4.2 For unconsumed fuse, a small portion of the fuse may be held in a flame with forceps or ignited in a glass container.

2.9.4.2.1 If very small lengths of fuse are present, then the examiner may elect to not conduct this test.

2.9.4.3 The reaction as the flame contacts the sample is noted and recorded.

2.9.5 References

2.9.5.1 Yallop, H.J., Explosion Investigation, The Forensic Science Society & Scottish Academic Press, Harrogate, England and Edinburgh, Scotland, 1980.

2.9.5.2 Yinon, Jehuda and Zitrin, Shimuel, Modern Methods & Applications In Analysis of Explosives, John Wiley & Sons, Inc.: NY, NY, 1993.

2.10 Instrumental Analysis

2.10.1 FTIR

The FTIR is used alone or in combination with other analytical techniques to identify chemical compounds and/or functional groups present in the explosives or explosives residues.

2.10.2 IC

The IC is used in combination with other analytical techniques to identify the sample cations and anions in solution.

2.10.3 XRD

The XRD may be used alone or in combination with other analytical techniques to identify chemical compounds present in powders exhibiting crystalline structures.

2.10.4 SEM-EDS

The SEM-EDS is used to identify the elemental composition of solid samples or dried extracts.

2.10.5 GC-MS

The GC-MS is used for the identification of compounds from their chemical structures.

2.10.6 References

- 2.10.6.1 Weiss, Joachim, Handbook of Ion Chromatography, Dionex Corporation: Sunnyvale, CA, 1986.
- 2.10.6.2 Yinon, Jehuda and Zitrin, Shimuel, Modern Methods & Applications In Analysis of Explosives, John Wiley & Sons, Inc.: NY, NY, 1993.
- 2.10.6.3 Yinon, Jehuda and Zitrin, Shimuel, The Analysis of Explosives, Pergamon Press Ltd., Oxford, England, 1981.
- 2.10.6.4 Gabriel, B.L., SEM: A User's Manual for Materials Science, American Society for Metals, Metals Park, OH, 1985.

2.11 Documentation

As a minimum, each case file will include an Explosives Worksheet(s), appropriate instrument condition sheets and hard copies of instrumental data with associated standards and/or references.

- 2.11.1 Hard copies of IC data must note if sample has been diluted and by how much.

2.12 Report Wording

The following information should be included in the body of the report whenever possible:

- 2.12.1 A description of the (reconstructed) device. Include measurements, the initiating mechanism, any labeling or markings, the container's composition, any anti-personnel material, and a description of any other material attached or associated with the device.
- 2.12.1.1 The Item 1 (reconstructed) device consisted of a metal pipe approximately 10 inches in length with an outer diameter of approximately 1 inch. Black electrical tape was wrapped around the

pipe's attached endcaps. An approximately 1/8 inch hole in the center of one of the endcaps contained an approximately 3 inch piece of green pyrotechnic safety fuse.

2.12.1.2 The Item 1 expended device contained an approximately 1/8 inch hole in the side of the length of the pipe which may have served _____.

2.12.1.2.1 If there is evidence of a fuse, complete the sentence with: may have served as a fuse hole.

2.12.1.2.2 If there is no indication of a fuse, complete the sentence with: may have served to accommodate the source of ignition.

2.12.1.3 Other device measurement examples:

2.12.1.3.1 Approximately 3 inches in length and 1/2 inch outer diameter.

2.12.1.3.2 The approximate measurements of the pipe were 3 inches in length and 1/2 inch in diameter.

2.12.1.3.3 The expended device was constructed of a 3/4 inch pipe approximately 12 inches in length.

2.12.2 A list of the tests used in the analysis.

2.12.2.1 Solid material contained in Item ___ was examined visually and using stereomicroscopy and Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS).

2.12.2.2 An extract of Item ___ was examined using a pH meter, microchemical tests, Ion Chromatography (IC) and Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS).

2.12.2.3 Item ___ was examined using stereomicroscopy, solubility tests, microchemical tests, Fourier Transform Infrared Spectrophotometry (FTIR), Ion Chromatography (IC), Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS) and X-Ray Diffraction (XRD).

2.12.2.4 The Item 1 and 2 bottles were examined visually.

2.12.3 A description of the explosive filler:

2.12.3.1 The Item _____ silver-grey powder was identified as flash powder.

2.12.3.2 Particles recovered from Item _____ were identified as a black powder substitute physically and chemically consistent with Pyrodex®.

2.12.3.3 Particles recovered from Item _____ were identified as (single/double base) smokeless powder.

2.12.3.4 Powder recovered from Item _____ was physically and chemically consistent with a pyrotechnic powder (a pyrotechnic mixture).

2.12.3.5 An extract of the Item _____ pipe was chemically consistent with that expected from deflagrated black powder.

2.12.3.6 An extract of Item _____ was chemically indicative of (black powder, black powder substitute, flash powder, smokeless powder, etc.). Indicative means that some of the data corresponds to

an explosive or explosive residue but, due to low concentration, sample condition, or other factors, a more definitive conclusion could not be reached.

2.12.4 A description of how the explosive filler would react if properly confined and/or ignited. **This statement is not necessary when the device has functioned or when the evidence submitted is intact ammunition from a convicted felon.**

2.12.4.1 Properly confined and ignited _____ (black powder, black powder substitute, flash powder, smokeless powder, etc.) will explode.

2.12.5 When no explosives are identified, the following statement should be used:

2.12.5.1 No explosives or explosives residues were identified in the Item ____ extracts.

2.12.6 For chemical reaction devices:

2.12.6.1 Unexpended device:

- Item ____ consisted of an intact, capped plastic bottle commercially labeled _____ containing liquid. The liquid was physically and chemically consistent with containing hydrochloric acid and aluminum (or hydrochloric acid and aluminum foil).
- Item ____ consisted of a disrupted, capped plastic bottle commercially labeled _____. (The liquid was...The extract was...)
- It should be noted that in the correct proportions, a mixture of hydrochloric acid and aluminum foil properly confined in a plastic bottle can produce a sufficient amount of gas to cause the bottle to explode.

2.12.6.2 Expended device:

- Item ____ consisted of a capped, ruptured, and distorted plastic bottle commercially labeled _____.
- The Item ____ materials were physically and chemically consistent with those expected from an expended chemical reaction device employing a capped plastic bottle containing hydrochloric acid and aluminum (or hydrochloric acid and aluminum foil).

2.12.6.3 Uncapped device:

- The Item ____ materials were physically and chemically consistent with those expected from an uncapped chemical reaction device employing a plastic bottle containing hydrochloric acid and aluminum (aluminum foil). It should be noted that in the correct proportions, a mixture of hydrochloric acid and aluminum (aluminum foil) properly confined in a plastic bottle can produce a sufficient amount of gas to cause the bottle to explode.

2.12.6.4 Physical and chemical evidence of a chemical reaction bomb with no bottle submitted:

- The Item ____ material was physically and chemically consistent with that expected from an expended chemical reaction device containing hydrochloric acid and aluminum (aluminum foil). It should be noted that a mixture of hydrochloric acid and aluminum (aluminum foil) properly confined in a plastic bottle can produce a sufficient amount of gas to cause the bottle to explode.

2.12.6.5 For non-aqueous chemicals such as “crystal” drain cleaners:

- It should be noted that a properly confined mixture of sodium hydroxide, water, and aluminum (aluminum foil) can produce a sufficient amount of gas to cause the bottle to explode.

2.12.6.6 For mixtures indicative of containing pool chlorinators:

- White residue present in the Item _____ bottle was chemically indicative of, but may not be limited to, the expected reaction product of a mixture of a calcium hypochlorite-based granular chlorinator, sugar, and water.
- It should be noted that a properly confined mixture of a calcium hypochlorite-based granular chlorinator, sugar, and water can produce a sufficient amount of gas to cause the bottle to explode.

2.12.7 Hoax devices:

2.12.7.1 Item _____ was physically consistent with a (disrupted) hoax explosive device.

2.12.8 For dry ice or liquid nitrogen bombs:

- The Item 1 expended device was physically and chemically indicative of, but may not be limited to, that expected from a dry ice or liquid nitrogen physical reaction device.
- It should be noted that properly confined dry ice, a solid form of carbon dioxide, or liquid nitrogen can produce a sufficient amount of gas to cause the bottle to explode.

2.12.9 1/16 inch, 3/32 inch or 1/8 inch typical green or red fuse material will be referred to as: pyrotechnic safety fuse.

2.12.9.1 For unexpended fuse:

Item _____ consisted of green pyrotechnic safety fuse, approximately 4 ½ inches in length.

2.12.9.2 For expended fuse:

Item _____ was consistent in physical properties with an expended (green, red) pyrotechnic safety fuse and was approximately ¾ inch in length.

2.12.9.3 For a fuse that does not function:

Item _____ was physically consistent with pyrotechnic safety fuse but did not function.

2.12.10 When a smokeless powder comparison has been performed:

- Particles removed from Item _ could not be associated with the Item _ smokeless powder particles due to differences in physical (or chemical) properties.
- The Item _ powder was consistent in physical and chemical properties with the Item _ powder. It was concluded that Item _ could have originated from Item _ or another source with the same physical and chemical properties.

2.12.11 When specifying commercial labeling, include at a minimum the product name and size of container, when possible.

2.12.12 If specifying a brand of black powder substitute in a report, the following superscripts are to be used:

Pyrodex®
 Clean Shot Powder™
 Black Canyon Powder™
 Clear Shot Powder™
 Golden Powder™
 Black Mag Powder™
 Triple Se7en®

2.12.13 Full Report Wording Examples – Explosives:

2.12.13.1 Example 1

Item 1	Distorted plastic bottle
Item 2	Foil
Item 3	Distorted plastic bottle
Item 4	Distorted plastic bottle

RESULTS

The Item 1 bottle was examined visually and an extract of the bottle was examined using a pH meter, microchemical tests, Ion Chromatography (IC), and Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS). The Item 2 foil was examined visually and using SEM-EDS.

Item 1 consisted of a capped, ruptured, and distorted plastic bottle commercially labeled “Nestle® Pure Life® PURIFIED WATER...”, “.5 L...”.

The Item 1 and 2 materials were physically and chemically consistent with those expected from an expended chemical reaction device employing a capped plastic bottle containing hydrochloric acid and aluminum foil.

The Item 3 and 4 bottles were examined visually and each was physically consistent with that expected from an expended chemical reaction device.

The disposition of the evidence and the results of other requested examinations are the subject of another report.

2.12.13.2 Example 2

Item 1	Box of forty-two 9mm rounds
--------	-----------------------------

RESULTS

Item 1 was examined visually and powder removed from one of each of the two different cartridge types was examined using stereomicroscopy, an ignition test, microchemical tests, and Fourier Transform Infrared Spectrophotometry (FTIR).

Item 1 consisted of a box of ammunition commercially labeled “INDEPENDENCE™...9mm LUGER...” containing 42 intact cartridges, of which there were two different commercially labeled types: “*I*” and “WIN”. The powder removed from each of the two different cartridge types was identified as smokeless gun powder.

The evidence is being returned to the Eastern Laboratory where it will be available for personal pickup.

2.12.13.3 Example 3

Item 5 Swab taken from inside of mailbox

RESULTS

Item 5 was examined using stereomicroscopy, solubility tests, microchemical tests, Fourier Transform Infrared Spectrophotometry (FTIR), Ion Chromatography (IC), and Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS).

An extract of the Item 5 swab was chemically consistent with that expected from a deflagrated black powder substitute such as Pyrodex®.

The evidence is being retained for personal pickup.

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FORENSIC SCIENCE

3 FIBERS**3.1 Analytical Approach**

- 3.1.1 List and describe each textile item. Include the label information as to fiber content, brand or manufacturer, manufacturer's numbers such as the RN# or WPL# and the size. Include a brief description of any design, logos or lettering present. Note the condition of the Item – intact, buttons missing, apparent rips or tears, etc. Supporting photographic documentation may be included.
- 3.1.2 The physical, chemical and optical properties of known and/or questioned fibers are noted and recorded. Examples of these properties are as follows: Physical – color (physical and instrumental using the microspectrophotometer), cross-section, diameter, delustrant, surface characteristics; Chemical - microsolubility tests, microchemical tests, FTIR; Optical - relative birefringence, sign of elongation, extinction and fluorescence. Relative refractive index is optional.
- 3.1.3 If a sufficient quantity of questioned fibers exists, then the known and questioned fibers will be compared and the properties noted in a side-by-side examination as follows: microchemical tests, microsolubility tests, cross-sectioning, polarized light microscopy (PLM), comparison microscopy, fluorescence microscopy, microspectrophotometry (MSP) and Fourier Transform Infrared Spectrophotometry (FTIR). The order of these exams and any additional testing is left to the discretion of the examiner and based upon the evidence at hand.
- 3.1.4 The exams used for natural fiber types would generally mirror those for synthetic fibers with the exception of instrumental analysis. Some natural fibers such as cotton, wool, and rabbit hair can be identified based solely upon their microscopic characteristics.
- 3.1.5 **Minimum Standards and Controls**
- Any mounting media with a stated expiration date will not automatically be discarded after the stated date. As long as the mounting media has not yellowed and continues to “flow” properly, as determined by the examiner, then it may continue to be used.
- 3.1.6 **References**
- 3.1.6.1 Robertson, J. and Grieve, M., Forensic Examination of Fibers, 2nd edition, Taylor and Francis, Inc., Philadelphia, PA, 1999.

3.2 Recovery of Hairs and/or Fibers

- 3.2.1 **Purpose**
- To examine evidence to locate, recover and preserve hairs/fibers for identification and/or comparison purposes.
- 3.2.2 **Summary**
- 3.2.2.1 Generally speaking, submitting the article(s) of evidence to the laboratory for the examiner to process is the best approach to the recovery of hairs and/or fibers. There are instances where this is not practical or possible, such as recovering hairs and/or fibers from wall-to-wall carpeting, a large piece of furniture, or a vehicle. In these instances, the recovery may be accomplished at the scene and the recovered hairs and/or fibers submitted for examination.
- 3.2.2.2 The order of preference for the recovery of hairs and/or fibers is manual removal with forceps followed by taping with Post-it type notes or another low tack tape. Gentle scraping may be necessary in certain instances. Vacuuming is rarely, if ever, performed because the debris recovered represents far more than recent hair and/or fiber transfers.

3.2.3 Minimum Standards and Control

- 3.2.3.1 The examiner shall change the examination paper between victim and suspect or scene exhibits. The examiner may change the paper between multiple victim, suspect or scene items, as necessary.
- 3.2.3.2 There should be only one exhibit opened at a time, unless two separate areas exist for this purpose.
- 3.2.3.3 The examiner shall change gloves and clean their tools between examining the evidence from the victim and the evidence from the suspect.
- 3.2.3.4 If possible, the victim's evidence and suspect's evidence should be examined in separate rooms. If this is not possible, then the separation of victim and suspect evidence in time and/or space will be necessary. Document in case file notes.
- 3.2.3.5 Use separate laboratory coats and evidence collection rooms, if available, for examining materials from victim and suspect to prevent possible cross-transfer contamination.
- 3.2.3.6 Avoid drafts around the examination area.

3.2.4 Analytical Procedure

- 3.2.4.1 Spread a clean piece of paper on the examination surface.
- 3.2.4.2 Examine each item of evidence visually or with the aid of an illuminated magnifier, UV light or stereomicroscope.
 - 3.2.4.2.1 If the item being examined contains hairs and/or fibers that are readily visible, collect these hairs and/or fibers with forceps. As hairs and/or fibers are collected, they should be placed in glassine packets or affixed to Post-it type notes.
 - 3.2.4.2.2 Take care with bulky items which require repositioning on the examination table, to avoid the loss of hairs and/or fibers in the repositioning process.
- 3.2.4.3 Post-it type notes or another low tack tape may be used to recover hairs and/or fibers. The adhesive surface is placed on the item being examined and then pulled away. Hairs and/or fibers will adhere to the adhesive on the tape.
 - 3.2.4.3.1 This method may be especially useful on large items or dark-colored items on which hairs and fibers of interest may be difficult to see.
 - 3.2.4.3.2 High tack tape can be used to ensure complete recovery of hairs but only for knit articles such as hats, caps, masks, and scarves. If fibers are probative to the case, a high tack tape must not be used.
 - 3.2.4.3.2.1 The only high tack tape approved for use is clear packing tape.
- 3.2.4.4 Scraping is generally discouraged as a method of collection for hairs and/or fibers. If scraping is necessary, the item to be examined is suspended above the examination surface and very gently scraped with a spatula. Scraping in a downward direction allows surface hairs and/or fibers to fall onto the examination paper for collection.

3.3 Fiber Identification

3.3.1 Purpose

The purpose of fiber identification is to identify animal and vegetable fibers and to identify manufactured fibers as to their generic class.

3.3.2 Safety Considerations

3.3.2.1 The use of xylene substitute, Permout, or Pro-Texx to mount the fibers requires caution. The process may be carried out in a well ventilated area or by using a "Nederman" point-of-use vent, if one is available.

3.3.2.2 Concentrated acids, solvents, and reagent chemicals (e.g., formaldehyde) should be used in a well-ventilated area with proper precautions being exercised to minimize skin contact.

3.3.3 Minimum Standards and Controls

3.3.3.1 Reference collections of animal, vegetable and manufactured fibers mounted on glass microscope slides as well as those not mounted are available. These reference collections can be useful to the examiner when identifying generic class of manufactured fibers as well as preparation of cross-sections for plant fiber identification.

3.3.3.2 A reference collection of FTIR spectra of known synthetic fiber generic classes and sub-classes is available. The resolution of the known fiber spectra must be equal to or better than that of the sample spectra.

3.3.3.3 Reagents shall be prepared in accordance with the QA/QC protocol (See Section 21.3) and reagent reliability checks will be recorded.

3.3.4 Analytical Procedures

3.3.4.1 Mount the specimen on a slide (using water, glycerin, xylene substitute, Permout, or Pro-Texx).

3.3.4.2 Using a compound microscope, examine the specimen. If the specimen can be identified at this point (e.g., cotton fibers or rabbit hair) it is not necessary to continue.

3.3.4.3 Cross-sections may be prepared as needed for vegetable fibers. This is typically accomplished using the Jolliff slide procedure as follows:

3.3.4.3.1 Jolliff slides are black plastic approximately 0.4 mm thick with holes 0.5 mm in diameter.

3.3.4.3.2 A needle threader or a looped strong, thin wire is inserted through a Jolliff slide hole.

3.3.4.3.3 Packing fibers are placed within the needle threader. Packing fibers are those with a simple cross-section and contrasting color. The packing fibers must fill the hole tightly enough so that they do not pull out when cut and they do not form a bulge in the plastic when drawn.

3.3.4.3.4 A few fibers to be sectioned are selected. A greater number of fibers to be sectioned require a corresponding decrease in the amount of packing fibers used.

- 3.3.4.3.5 The fiber(s) to be sectioned is placed in the bundle of packing fibers and pulled just enough so that the fiber(s) of interest is drawn through creating a U-shaped loop.
- 3.3.4.3.6 The bundle of fibers is cut flush on both sides of the Jolliff slide with a new, single-edged razor blade.
- 3.3.4.3.7 Compare the cross-sections with known reference cross-sections, if necessary, via the comparison microscope to assist in making identifications.
- 3.3.4.4 The dry twist test may assist in differentiating vegetable fibers as follows:
- 3.3.4.4.1 Soak fiber(s) in water for a few minutes until thoroughly wet.
- 3.3.4.4.2 Remove a single fiber, holding it at one end with forceps.
- 3.3.4.4.3 With the free end toward the observer, hold the fiber over a hot surface, such as a hot plate.
- 3.3.4.4.4 Observe whether the fiber rotates in a clockwise or counterclockwise direction (or if the direction of twist is inconclusive) as the fiber dries.
- 3.3.4.4.5 Repeat test with different fibers, if possible, to ensure reproducibility.
- 3.3.4.4.6 Refer to the table of Direction of Twist for Common Natural Fibers.

DIRECTION OF TWIST FOR COMMON NATURAL FIBERS [3.3.5.9]

Clockwise	Counterclockwise	Alternating Directions	Clockwise or Indecisive	Variable or None
Flax Ramie Kapok (Java) Nettle fiber	Hemp Jute Manila Sisal Henequen	Cotton	Coir	Kapok (Indian)

- 3.3.4.5 For manufactured and select other fibers, observe the optical properties of the fibers using the polarized light microscope. Refer to the table of Optical Properties of Common Fibers.

OPTICAL PROPERTIES OF COMMON FIBERS [3.3.5.9]

- Low Birefringence: Triacetate (+); n's ~ 1.469
Modacrylics (+ or -); n's ~ 1.53 & 1.54
Cellulose Acetate (+); n's ~ 1.474
Acrylics (-); n's ~ 1.51
- Moderate Birefringence: Viscose Rayon (+); n's ~ 1.55 & 1.52
Cotton (+); no extinction; n's ~ 1.58 & 1.53
Wool & other animals (+); n's ~ 1.56 & 1.55
Silk (+); n's ~ 1.59 & 1.54
Linen & other bast fibers (+); n's ~ 1.59 & 1.53
- High Birefringence: Nylon (+); n's ~ 1.57 & 1.52
Polyester (+); n's ~ 1.70 & 1.53
Nomex (+); n's ~ 1.75 & 1.67
Kevlar (+); n's ~ 2.35 & 1.64

- 3.3.4.6 For vegetable fibers, the Herzog Test may assist in identifying the fiber type. The Herzog Test is performed using the polarized light microscope with the fibers mounted on a slide in a suitable medium. Under crossed polars, align the fiber approximately north-south, or parallel to the vibration direction of the analyzer. The fiber ultimate should be the darkest in this orientation. Insert the full-wave plate and observe the color of the fiber. Note: This test should be repeated for color reproducibility and only completed on ultimates not bundles.

Expected results: [3.3.5.4]

Flax and Ramie = Yellow (sometimes orange or pink)
Hemp, Jute, Sisal, and Manila = Blue

- 3.3.4.7 For manufactured fibers, obtain an FTIR spectrum. This is most typically accomplished using the microcompression cell with diamond windows. Compare the obtained spectrum with reference spectra of synthetic fibers.
- 3.3.4.8 Generally, the solubility of a fiber in specific liquids can assist in generic class identification. Determine the solubility of the fibers in m-cresol, DMF, 75% sulfuric acid, concentrated nitric acid, concentrated hydrochloric acid, LeRosen, and others such as HFIP, as needed. Also, note any color reactions that take place. The chemicals and reagents used are selected at the discretion of the examiner and should be based upon the results of the other examinations. Refer to the table of Solubilities of Common Fibers.

Key to solubility reactions:

S = Soluble/Disintegrates (fades, splinters, or breaks apart and goes into solution)

P = Partially Soluble (not all portions of a fiber are soluble)

I = Insoluble (no reaction)

SOLUBILITIES OF COMMON FIBERS [3.3.5.7]

	ACT S/T	ACR	MOD D/V	NYL	NYT	OLE PE/PP	POL	RAY	SPA	CO/ FL	SI	WO
m-Cresol	S/S	I	I/I	S	I	I/I	I	I	I	I	I	I
DMF	S/S	P/I	S/S	I	S	I/I	I	I	I	I	I	I
Conc. HCl	S/P	I	I/I	S	I	I/I	I	I	I	I	S	I
Conc. HNO ₃	S/S	S	I/-	S	S	I/I	I	I	P/S	I	P/S	P/I
75% H ₂ SO ₄	S/S		I/I	S	I	I/I	I	S	P/S	S	P/S	I
LeRosen				S		I/I	S					
HFIP	S/S			S		I/I	S	I	S		I	

ACT S/T = acetate secondary/triacetate

ACR = acrylic

MOD D/V = modacrylic dynel/verel

NYL = nylon

NYT = nyltril

OLE PE/PP = olefin polyethylene/polypropylene

POL = polyester

RAY = rayon

SPA = spandex

CO/FL = cotton/flax

SI = silk

WO = wool

3.3.4.9 Identifying Characteristics of Some of the More Common Animal Fibers (Hair and Silk) Used in Textiles [3.3.5.9]

WOOL – faint to pronounced scales, medulla may or may not be present, usually no pigment or centric pigment distribution, diameter range 10-90 μm . Sources may include sheep, goat, cashmere, mohair, camel, alpaca, llama and vicuna.

RABBIT - uniserial to multiseriate medulla

SILK (BOMBYX MORI) - twin continuous filaments, cemented together, degummed filaments are fine, uniform, with no visible internal structure, triangular cross section with rounded corners

SILK (TUSSAH) - brownish, coarse, ribbon-like with striated and granular structure, wedge-shaped x-section

3.3.4.10 Identifying Characteristics of Some Common Plant Fibers [3.3.5.9 and 3.3.5.10]

COTTON - looks like twisted, flattened tubes (convoluted), very irregular in appearance, remains bright in all orientations between crossed polars

Mercerized Cotton: not as twisted, somewhat featureless, but still not regular

KAPOK - smooth, cylindrical, hollow, thin-walled, frequently bent over on itself, tapers to a point with other end having a bulbous base with annular or reticular markings; x-section is oval or circular with a wide lumen

FLAX - (linen) small, regular lumen, characteristic x-shaped cross markings; usually exists as ultimates; x-section is polygonal with thick cell walls surrounding a narrow/small lumen.

JUTE - large circular or elliptical lumen with constrictions; occasional nodes and markings, usually in bundles; x-section is polygonal with sharp defined angles and thick cell walls

HEMP - lumen not always evident (broad and flat when seen); many cross-markings; x-section is polygonal with thick cell walls

RAMIE - flat undeveloped lumen with radial cracks through thick cell walls; x-section is irregularly oval to polygonal, many characteristic cross-markings, many ridges and striations

SISAL - well defined fairly large lumen; spiral elements present; x-section is irregularly oval to polygonal; acicular crystal present; stigmata present

ABACA - (manila) lumen typically defined, round, and uniform in diameter; stigmata present; x-section is irregularly oval to polygonal with rounded corners and thin cell walls

COIR - very short fibers, surface covered with stigmata in longitudinal rows; x-section is thick cell walls surrounding an irregularly indented lumen outline

3.3.5 References

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- 3.3.5.8 Hicks, John, Microscopy of Hair: A Practical Guide and Manual, FBI, Issue 2, January 1977.
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- 3.3.5.11 Microtrace, Course notes from Forensic Fiber Identification course given at Texas Department of Public Safety, June 1998.
- 3.3.5.12 Palenik, S. and Fitzsimons, C., "Forensic Microscopy, Fiber Cross-Sections: Part II", *Microscope*, 38, 1990 pp. 313-320.
- 3.3.5.13 Scientific Working Group for Materials Analysis, "Forensic Fiber Examination Guidelines", *Forensic Science Communications*, Vol. 1, No. 1, April 1999.
- 3.3.5.14 The Textile Institute. Identification of Textile Materials, Seventh Edition, Manchester, 1985.

3.4 Fiber Comparison

3.4.1 Purpose

To determine if fibers from different sources could have had a common origin. Special considerations that should be noted when performing fiber comparisons:

- 3.4.1.1 The comparison of questioned fibers with fibers from a known source is performed in every step of the examination once the questioned fibers are recovered and the known target fibers are established.
- 3.4.1.2 The examiner may approach the fiber comparison by attempting to show that the samples are different. The failure to detect any meaningful differences, after exhausting the methodology available to the examiner, results in the conclusion that the K (known) and Q (questioned) fibers could have had a common origin.
- 3.4.1.3 When sample size is limited, destructive testing is generally performed after all non-destructive testing is complete.

3.4.2 Safety Considerations

- 3.4.2.1 Do not look directly into the fluorescence source.
- 3.4.2.2 Do not look directly into the FTIR laser source.

- 3.4.2.3 The MCT detector for the FTIR microscope accessory must be cooled with liquid nitrogen. Insulated gloves and safety glasses shall be worn when filling the transport Dewar as well as the instrument reservoir.
- 3.4.3 Minimum Standards and Controls
- 3.4.3.1 Extreme caution must be used when handling known and questioned fibers to avoid any possibility for cross-contamination.
- 3.4.3.2 Use the same mounting media for known and questioned fibers.
- 3.4.3.3 The known and questioned fibers shall be examined at the same time, in a side-by-side fashion. The fiber worksheet(s) shall be used to record all positive, probative comparisons.
- 3.4.3.4 The comparison microscope data of all positive, probative associations will be verified by a second qualified fiber examiner. The original fiber worksheet(s) will be initialed and dated by the second examiner in the space labeled "verification" which will denote agreement with the notes. The verification includes the K and Q comparison of those features in the highlighted areas on the worksheet(s).
- 3.4.3.5 Reagents shall be prepared in accordance with the QA/QC protocol (See Section 21.3) and reagent reliability checks will be recorded.
- 3.4.4 Analytical Procedures
- 3.4.4.1 Polarized Light Microscopy
- 3.4.4.1.1 Prepare temporary or permanent mounts of the known and questioned fibers.
- 3.4.4.1.1.1 Pro-Texx (RI = 1.495 ± 0.005), Permout (RI = 1.525), Norland Optical Adhesive 60[®] (RI = 1.56), xylene substitute (RI = Unk), or glycerin (RI = 1.47).
- 3.4.4.1.2 View the known and questioned fibers using a polarized comparison microscope. Using a fiber worksheet, record the physical and optical properties. These will include at a minimum: color, diameter, delustrant, extinction, relative birefringence, sign of elongation and optical cross-section.
- 3.4.4.1.2.1 Relative birefringence is determined by viewing the retardation colors using a full wave plate, a 4 order Berek compensator and/or a 20 order Berek compensator.
- 3.4.4.1.2.2 The relative refractive index may be noted. This is accomplished by observation of the Becke line movement with respect to an appropriate mounting medium. The mounting medium of choice shall be noted on the fiber worksheet.
- 3.4.4.1.3 Refer to the table of Optical Properties of Common Fibers. (See ¶ 3.3.4.5)
- 3.4.4.1.4 Comparisons should be performed side-by-side. Any major discrepancies between the K and Q samples are reason for elimination.
- 3.4.4.2 Fluorescence Microscopy
- Some fibers fluoresce when exposed to different wavelengths of light. Of those fibers, the observed color and intensity of emission under different excitation wavelengths are noted.

- 3.4.4.2.1 These observations are generally conducted following the observation of physical and optical properties.
- 3.4.4.2.2 Certain mounting media will fluoresce. Non-fluorescing media should be used to achieve optimum contrast with the background.
- 3.4.4.2.3 Fluorescence QC procedure:
- 3.4.4.2.3.1 Perform fluorescence QC check prior to using the fluorescence microscope for casework. Use the fluorescence QC check sample and record observations on the fluorescence worksheet. If the fluorescence QC check fails to produce acceptable colors or intensity, troubleshoot the fluorescence microscope as appropriate and repeat the QC check. If the QC check continues to fail, take the fluorescence microscope out of service until the problem is resolved.
- 3.4.4.2.3.2 Fluorescence cubes to be used are WU (wide UV – range 330 – 385nm), WBV (wide blue violet – range 400 – 440nm), WB (wide blue – range 450 – 480nm) and WG (wide green – range 510 – 550nm). Record observations on a fluorescence worksheet.
- 3.4.4.2.4 Differences may be seen between the fluorescence of known and questioned samples if one of the samples has been subjected to different conditions than the other sample.
- 3.4.4.2.5 Caution should be taken when eliminating samples based upon fluorescence data alone. This is because fluorescence can be caused by optical brighteners, detergents, bleaching agents, dyes, the chemical structures themselves or other additives. Detailed notes explaining the basis for the elimination are a must. While fluorescence is an optical property, eliminations based upon fluorescence will be reported as “due to differences in fluorescence”.
- 3.4.4.3 Comparison Microscope
- 3.4.4.3.1 Observe known and questioned fibers in temporary or permanent mounts with the comparison microscope.
- 3.4.4.3.2 Record whether the known and questioned fibers are consistent or not.
- 3.4.4.4 Microspectrophotometry (MSP)
- 3.4.4.4.1 Minimum Standards and Controls
- Ensure that the QC has been performed.
- 3.4.4.4.2 Analytical Procedures
- 3.4.4.4.2.1 Range of colors, color intensity and distribution as well as cross-sectional shape should be considered when determining the number of spectra to be collected for the comparison of the known and questioned fibers.
- 3.4.4.4.2.2 Comparison should be performed by overlaying the known and questioned spectra. Any major discrepancies between the two are reason for elimination.

3.4.4.4.2.3 The average of the known and questioned spectra may be printed and compared. However, individual spectra will always be included in the case file.

3.4.4.5 FTIR

This instrumental technique is generally not applicable for vegetable fibers or animal hair fibers.

3.4.4.5.1 Minimum Standards and Controls

3.4.4.5.1.1 Ensure that the QC has been performed.

3.4.4.5.2 Analytical Procedures

3.4.4.5.2.1 Obtain spectra of the known and questioned fibers either by rolling the fibers flat and placing them on a KBr window or by using the microcompression cell with diamond windows.

3.4.4.5.2.2 The spectra for the known and questioned are compared by overlaying the known and questioned spectra.

3.4.4.5.2.3 Any major discrepancies between the two are reason for elimination. It may be necessary to sample several known or questioned fibers or to take several spectra along the length of a given fiber to determine the reproducibility of the spectra.

3.4.4.6 Cross-sections

Frequently the cross-section of a fiber can be determined from the longitudinal view, also known as optical cross-sectioning. This may be the only technique available for cross-sections if the questioned fiber is too short.

3.4.4.6.1 Prepare cross-sections of the known and questioned fibers, treating both the known and questioned fibers in the same manner, using one of the following methods:

3.4.4.6.1.1 Polyethylene sheets - Single fibers are placed between two plastic sheets. The plastic "sandwich" is placed on a glass microscope slide, another slide or a cover slip is placed over the plastic "sandwich" and this is then transferred to a hot plate set on low. Some pressure may need to be applied as the plastic melts around the fiber. A new single-edged razor blade or scalpel blade may be used to slice thin cross-sections of the sandwiched fiber. The remaining portion of fiber is easily removed by cutting and lifting away the plastic sheets.

3.4.4.6.1.2 Super Glue® - Place the fibers to be cross-sectioned on a microscope slide. Add enough glue to cover the fibers. After fully drying, use a new single-edged razor blade or scalpel blade to slice thin cross-sections of the fiber.

3.4.4.6.1.3 Norland Optical Adhesive 60® - Place the fibers to be cross-sectioned on a microscope slide. Add enough adhesive to cover the fibers. Expose to long wave ultraviolet light (320-400nm) for at least 10 minutes, or until completely cured. A new single-

edged razor blade or scalpel blade may be used to slice thin cross-sections of the fiber.

3.4.4.6.1.4 Norland/plastic pipette – Grasp the tip of the pipette and draw it into a narrower cylinder. Insert the fibers to be cross-sectioned into the narrowed pipette tip. Using capillary action, draw Norland Optical Adhesive 60® into the pipette tip. Place tip with fiber and adhesive on microscope slide. Expose to long wave ultraviolet light (320-400nm) for at least 10 minutes, or until completely cured. A new single-edged razor blade or scalpel blade may be used to slice thin cross-sections of the pipette and fiber.

3.4.4.6.1.5 Jolliff slide method as previously described. (See ¶ 3.3.4.3)

3.4.4.6.2 Compare the prepared cross-sections using the comparison microscope and record observations on fiber worksheet. Any major discrepancies are reason for elimination.

3.4.4.7 Microsolubility/ Microchemical Tests

3.4.4.7.1 The chemicals and reagents used are at the discretion of the examiner and should be based upon results from other examinations and sample condition and quantity.

3.4.4.7.2 Known and questioned fibers are observed side-by-side for a comparison of any color reactions.

3.4.4.7.3 Generally, the solubility of a fiber in specific liquids can assist in generic class identification.

3.4.4.7.4 Microsolubility/ Microchemical test reagents that are used are: m-cresol, DMF, 75% sulfuric acid, concentrated nitric acid, concentrated hydrochloric acid, LeRosen, and others such as HFIP, as needed.

3.4.4.7.4.1 For those reagents where the QC check does not give an acceptable result, repeat the QC check with fresh reagent until an acceptable result is achieved.

3.4.4.7.5 Cut small portions of the known and questioned fibers and place them in wetted spot plates or on a microscope slide under a cover slip.

3.4.4.7.6 Add a drop or two of the microchemical test reagent. Observe reactions with the aid of the stereomicroscope and record observations using the fiber microchemical worksheet. Any major discrepancies are reason for elimination.

3.4.4.7.7 Refer to the table of Solubilities of Common Fibers. (See ¶ 3.3.4.8)

3.4.4.8 References

3.4.4.8.1 Robertson, J. and Grieve, M., Forensic Examination of Fibers, 2nd Edition, Taylor and Francis, Inc., Philadelphia, PA, 1999.

3.4.4.8.2 Scientific Working Group for Materials Analysis, “Forensic Fiber Examination Guidelines”, *Forensic Science Communications*, Vol. 1, No. 1, April 1999.

3.5 Fabric Construction

3.5.1 Analytical Approach

- 3.5.1.1 The examiner is typically requested to compare a questioned sample to a known fabric/carpet sample. Instead of a Q vs. K comparison, the examiner may be requested to conduct a determination if Q and K could have been constructed by the same manufacturer.
- 3.5.1.2 The questioned and known evidence should not be brought into direct contact until after a preliminary examination of both items has been completed separately.
- 3.5.1.3 The examiner first conducts a macroscopic examination of the fabric/carpet evidence. If appropriate, a fracture match examination can be conducted.
- 3.5.1.4 Preliminary examination should include but is not limited to noting size, shape, and condition (stains, patterns, buttons, cut/torn/damaged edges, etc.).
- 3.5.1.5 The examiner can continue analyzing the fabric/carpet evidence describing the construction and then comparing the fibers composing the fabric/carpet following the fiber comparison protocol.

3.5.2 Macroscopic/Microscopic Examination

3.5.2.1 Purpose

To examine submitted fabric/carpet evidence on a macroscopic and microscopic scale, noting physical properties regarding its construction, generally as the first step in the characterization and comparison of known and questioned fabric/carpet samples.

3.5.2.2 Analytical Procedures

- 3.5.2.2.1 Thoroughly clean the examination area prior to the evidence being retrieved.
- 3.5.2.2.2 The sample is examined, macroscopically and/or microscopically, to observe and record the physical construction properties to include, but not be limited to, the following, when applicable:
- Construction (woven, knit, nonwoven)
 - Yarns per inch in warp and weft direction
 - Yarn twist (s or z)
 - Staple or filament fibers in yarns
 - Color of fabric, yarns, fibers, or backing
 - Number of plies
 - Ply twist
 - Carpet pile (loops, cut, etc.)

3.5.2.3 If any meaningful differences are observed between the questioned and known fabric/carpet constructions, the examiner can discontinue the analysis.

3.5.2.4 When no meaningful differences are observed, the fibers within the fabric/carpet are further identified and compared following the fiber comparison protocol.

3.5.2.5 References

- 3.5.2.5.1 “Forensic Fiber Examination Guidelines: Fabrics and Cordage,” SWGMAT, *JASTEE*, Vol. 6(1), 2015, pp. 83-90.

- 3.5.2.5.2 Forensic Examination of Fibres, 2nd Edition, J. Robertson and M. Grieve, eds., Taylor and Francis, London, 1999.
- 3.5.2.5.3 Introduction to Hairs and Fibers (Training Materials), F.B.I., March 2007.
- 3.5.2.5.4 Standard Guide for Forensic Examination of Fabrics and Cordage: ASTM Method E 2225-10.
- 3.5.2.5.5 Textile Science, K. L. Hatch, West Publishing Company, New York, 1993.

3.6 Ropes and Cordage

3.6.1 Analytical Approach

- 3.6.1.1 The examiner is typically requested to compare a questioned sample to a known rope or cordage sample.
- 3.6.1.2 The questioned and known evidence should not be brought into direct contact until after a preliminary examination of both items has been completed separately.
- 3.6.1.3 The examiner first conducts a macroscopic examination of the cordage evidence. If appropriate, a fracture match examination can be conducted.
- 3.6.1.4 Preliminary examination should include, but is not limited to, noting size, shape, and condition (stains, knots, fraying/cut/torn/damaged ends, etc.).
- 3.6.1.5 All knots should be left intact.
- 3.6.1.6 The examiner can continue analyzing the cordage evidence describing the construction and then comparing the fibers composing the cordage following the fiber comparison protocol.

3.6.2 Macroscopic/Microscopic Examination

3.6.2.1 Purpose

To examine submitted cordage evidence on a macroscopic and microscopic scale, noting physical properties regarding its construction, generally as the first step in the characterization and comparison of known and questioned cordage samples.

3.6.2.2 Analytical Procedures

- 3.6.2.2.1 Thoroughly clean the examination area prior to the evidence being retrieved.
- 3.6.2.2.2 The sample is examined, macroscopically and/or microscopically, to observe and record the physical construction properties to include, but not be limited to, the following, when applicable:
- Color(s)
 - Diameter
 - Condition of the ends (e.g., cut, torn, crushed, burned)
 - Presence of a coating
 - Presence of a tracer
 - Core
 - Twisted, braided, non-twisted
 - Direction of twist
 - Number of strands

- Crowns or turns per inch
- Length of lay
- Staple or filament fibers
- Strand direction of twist
- Number of plies or braids
- Twist of each ply or braid
- Crowns or turns per inch of each ply or braid
- Number of filaments in each ply or braid

3.6.3 If any meaningful differences are observed between the questioned and known cordage constructions, the examiner can discontinue the analysis.

3.6.4 When no meaningful differences are observed, the fibers within the cordage are further identified and compared following the fiber comparison protocol.

3.6.5 References

3.6.5.1 "Forensic Fiber Examination Guidelines: Fabrics and Cordage," SWGMAT, *JASTEE*, Vol. 6(1), 2015, pp. 83-90.

3.6.5.2 Forensic Examination of Fibres, 2nd Edition, J. Robertson and M. Grieve, eds., Taylor and Francis, London, 1999.

3.6.5.3 Introduction to Hairs and Fibers (Training Materials), F.B.I., March 2007.

3.6.5.4 "Recognition, identification and comparison of rope and twine," Wiggins, K.G., *Science & Justice*, 35(1), 1995, pp. 53-58.

3.6.5.5 Standard Guide for Forensic Examination of Fabrics and Cordage: ASTM Method E 2225-10.

3.7 Documentation

3.7.1 The examiner's notes will include a description of each textile item (if applicable), the questioned fiber(s), including color, approximate amount and how the fiber was analyzed (e.g., microscopic examination, cross-section, dry twist test etc.). Approximate length will be noted when applicable.

3.7.2 All worksheets, FTIR spectra with appropriate reference spectra, and MSP data will be included, as appropriate.

3.8 Report Wording

3.8.1 Tests used in the analysis shall be identified in the body of the report. The report will generally read:

- Fibers composing Item ____ were examined using stereomicroscopy, comparison microscopy, fluorescence microscopy, polarized light microscopy (PLM), microchemical tests, microsolubility tests, Fourier Transform Infrared Spectrophotometry (FTIR), and Microspectrophotometry (MSP).
- Items ____ and ____ were examined visually and using stereomicroscopy. Fibers composing Items ____ and ____ were further examined using comparison microscopy, fluorescence microscopy, polarized light microscopy (PLM), microchemical tests, microsolubility tests, and Microspectrophotometry (MSP).

3.8.2 For identification purposes:

3.8.2.1 Questioned manufactured fibers that require only identification are generally reported as belonging to one of the following generic classes or subclasses:

- Acetate
- Acrylic
- Aramid
 - Kevlar
 - Nomex
- Chlorofiber
- Modacrylic
- Nylon
- Olefin
 - Polyethylene
 - Polypropylene
- Polyester
- Regenerated Cellulose (including, but not limited to, bamboo, rayon, Tencel®, and lyocell)
- Saran
- Spandex
- Triacetate
- Vinal
- Vinyon

3.8.2.2 Color will be reported and cross-sectional shape or end use may be reported as appropriate.

- Based upon the fibers examined, Item _____ contained red, trilobal nylon fibers that may be found in, but are not limited to, some carpets.
- The Item _____ (shirt, pants, etc.) fibers were composed of colorless cotton. Colorless cotton fibers do not possess a sufficient number of individual characteristics for meaningful comparison.

3.8.3 For comparison purposes:

3.8.3.1 If the known and questioned fibers can be eliminated based upon any of the testing the report will generally read:

- Based upon the fibers examined, the Item _____ fibers could not be associated with the Item _____ fibers due to differences in _____ (state color, physical properties, optical properties, chemical properties and/or fluorescence as the reason for the differences).
- Based upon the fibers examined, the _____ (color and/or type) fibers composing Item _____ could not be associated with the fibers composing Item _____ due to differences in _____.

3.8.3.2 If the known and questioned fibers cannot be eliminated based upon any of the testing the report will generally read:

- The red nylon (color and/or type) fiber in Item _____ was consistent in physical, chemical and optical properties with the fibers composing the Item _____ blanket. Based upon the fibers examined, it was concluded that the Item _____ red nylon (color and/or type) fiber could have originated from the Item _____ blanket (known) or another source composed of fibers with the same physical, chemical and optical properties.

- The Item _____ sweater was composed of two different types of blue acrylic (color and/or type) fibers. Two different types of blue acrylic fibers were recovered from the Item _____ pants which were consistent in physical, chemical and optical properties with those composing the Item _____ sweater. Based upon the fibers examined, it was concluded that these recovered Item _____ blue acrylic (color and/or type) fibers could have originated from the Item _____ sweater or another source composed of fibers with the same physical, chemical and optical properties.
- Blue and white polyester fibers and yarns in Item _____ were consistent in physical, chemical, and optical properties with the fibers and yarns composing the Item _____ shirt. Based upon the fibers examined, it was concluded that these Item _____ blue and white polyester fibers could have originated from Item _____ or another source composed of fibers with the same physical, chemical, and optical properties.

3.8.3.3 If foreign fibers were recovered and knowns are being requested for comparison purposes the report will generally read:

- Foreign (color and/or type) fibers (or “Foreign fibers of various colors and types”) were recovered from Item(s) _____ which were suitable for comparison purposes. If a possible source is located, resubmit Item(s) along with the possible source for comparison purposes.

3.8.3.4 If the examiner is requested to make a comparison based on construction the report will generally read:

- Items _____ were consistent in color and overall construction and were composed of fibers which were consistent in physical, chemical and optical properties. Based upon the fibers examined, it was concluded that these Items could have once been a part of the same set.
- The Item 1 and 2 washcloths were consistent in color and overall construction and were composed of cotton fibers which were consistent in physical, chemical and optical properties. Based upon the fibers examined, it was concluded that Items 1 and 2 could have originated from the same manufacturer.
- Items 1 and 2 were consistent in color and overall construction and were composed of polyester fibers which were consistent in physical, chemical, and optical properties. Based upon the fibers examined, it was concluded that Item 1 could have originated from the source represented by Item 2 or a different source manufactured in the same manner.
- Items 1, 2, and 3 were each twine samples composed of jute fibers which were consistent in physical, chemical and optical properties with the jute fibers composing Item 4. Based upon the fibers examined, it was concluded that these twine samples could have originated from the Item 4 roll of twine or another roll of twine manufactured in the same manner.

3.8.4 If a cross-transfer of fibers or materials has occurred, the resulting strength of conclusion is increased. Refer to ¶ 9, Paint, for wording.

3.8.5 Generally, textile materials are mass produced and it is not possible to state that a fiber originated from a particular garment to the exclusion of all other textile materials having the same color and type of fibers in their construction.

4 **FIRE DEBRIS****4.1 Analytical Approach**

- 4.1.1 Generate one or more Fire Debris Worksheets.
- 4.1.2 Briefly open each container, make a gross visual inspection and record a description of the contents. Do not intentionally smell the contents of the container while observing the contents but do make a notation of any unavoidable odor from the contents.
- 4.1.3 If a nonaqueous liquid is present, test to confirm its nonaqueous nature. A nonaqueous liquid will be immiscible with water and will be noted on the worksheet. A nonaqueous liquid or appropriate water miscible liquid (e.g., alcohols) may be injected directly into the gas chromatograph – mass spectrometer (GC-MS). See Direct Injection procedure, ¶ 4.2. The term “aqueous” applies to those liquids that are water and will be noted on the worksheet. An aqueous liquid will either be analyzed by the Passive Adsorption-Elution procedure (¶ 4.5) or extracted first by Ambient Headspace analysis (¶ 4.3) followed by the Solvent Extraction procedure (¶ 4.4).
- 4.1.4 It may be necessary to determine if an unknown liquid sustains a flame. This determination would only be necessary when a non-routine or otherwise unusual liquid is encountered. Attempt to ignite a small amount of the liquid on a watch glass in a hood and record the results on the Fire Debris Worksheet.
- 4.1.5 If the sample contains enough ignitable liquid to give an unavoidable strong odor, resists heating (e.g., insulation), or when a distinction is needed between Kerosene and Diesel Fuel, then solvent extraction should be employed. Unless the sample contains enough ignitable liquid to give an unavoidable strong odor or is not packaged in an airtight container, a headspace extraction should be performed before the solvent extraction.
- 4.1.6 If a latent prints examination is requested on a bottle or other container and no nonaqueous liquid is present, a solvent extraction of the interior may be performed using caution while wearing gloves or holding the item with tongs or forceps.
- 4.1.7 Any sample extract vials generated during analysis are returned in the can, or other container, corresponding to their particular item. Any deviation is noted on the Fire Debris Worksheet to include in which container vials that contain combined extracts are being returned.
- 4.1.8 The GC-MS Total Ion Chromatogram (TIC) for case samples is compared to the GC-MS TIC for known reference samples according to retention times and overall patterns. When extracted ion chromatography (EIC), also known as mass spectral ion profiling (MSIP), is applied to case samples and to known reference samples, the patterns that are generated are compared.
- 4.1.9 If data generated from a Passive Adsorption-Elution extraction contains peaks/pattern that may be consistent with a heavy petroleum distillate, then a follow-up Solvent Extraction may be performed.
- 4.1.10 Safety Considerations
- Intentional inhalation of the odor of the contents of items for fire debris analysis is prohibited due to the potential health concerns related to this activity.
- 4.1.11 Minimum Standards and Controls
- 4.1.11.1 Carbon disulfide (CS₂) - Each new bottle or lot of carbon disulfide must be tested prior to its use in casework. Approximately 5 ml of the new solvent is evaporated to a few drops. 2 µl of this evaporated solvent is injected into the GC-MS. If the resulting data has no significant peaks which interfere with the analysis, then the solvent is available for use in casework. The data is labeled with: manufacturer, lot number, start volume, end volume and is placed in a file.

- 4.1.11.2 Pentane (C₅) - Each new bottle or lot of pentane must be tested prior to its use in casework. Approximately 500 ml of the new solvent is evaporated to a few drops. 1-2 µl of this evaporated solvent is injected into the GC-MS. If the resulting data has no significant peaks which interfere with the analysis, then the solvent is available for use in casework. The data is labeled with: manufacturer, lot number, start volume, end volume and is placed in a file.
- 4.1.11.3 The Resolution Test Mixture (RTM) shall be injected daily when the instrument is used for fire debris casework. This mixture shall be a certified reference material purchased from an approved vendor. The fire debris check sample (FDCS) may be injected into the GC-MS prior to analyzing case samples. At least one copy of the daily RTM must be included in each case file.
- 4.1.11.4 The oxygenate check sample (OCS) must be injected into the GC-MS prior to analyzing case samples. At least one copy of the daily OCS must be included in each case file. The method for the oxygenate check sample may be truncated to stop after the last compound has eluted.
- 4.1.11.5 When the polar column GC is used, the polar check sample (PCS) must be injected into the GC prior to analyzing case samples. At least one copy of the PCS must be included in each case file.
- 4.1.11.6 There must be at least one blank between each case sample injected. If the autosampler sequence runs longer than the work day or overnight, then either the FDCS or another petroleum product reference should be included at the end of the sequence.

4.2 Direct Injections

4.2.1 Purpose

When a whole liquid is obtained, a direct injection may be performed. The purpose of a direct injection is to introduce a whole liquid into the GC-MS.

4.2.2 Minimum Standards and Controls

4.2.2.1 A small amount, determined by the examiner, should be injected to ensure that the GC-MS is not overloaded.

4.2.2.2 If a solvent is used to dilute the sample, then a solvent blank must be included in the case file.

4.2.3 Analytical Procedures

4.2.3.1 Whole liquid samples can be injected directly into the gas chromatograph-mass spectrometer (GC-MS).

4.2.3.2 Alternatively, whole nonaqueous liquid samples may be diluted with either pentane or carbon disulfide and injected directly into the GC-MS.

4.2.4 References

4.2.4.1 Midkiff, Charles R. "Arson and Explosive Investigation," Forensic Science Handbook, Volume 1, 2nd Edition, Pearson Education, Inc., 2002, pages 479-524.

4.2.4.2 Stauffer, E., Dolan, J., and Newman, R., Fire Debris Analysis, Burlington, MA, Elsevier, Inc., 2008, pp. 296-354.

4.3 Ambient Headspace

4.3.1 Purpose

If a sample contains highly volatile components, these components will be in the vapor state at room temperature. The purpose of an ambient headspace extraction is to sample volatile components present in the air space above the debris. A portion of this vapor sample is injected directly into the gas chromatograph-mass spectrometer (GC-MS).

Special considerations that should be noted with this extraction technique:

- 4.3.1.1 May be affected by the presence of water in the sample, particularly if the compound of interest is miscible with water.
- 4.3.1.2 Produces a disproportionate representation of low boilers (highly volatile) in the headspace i.e., swamping of headspace with more volatile components producing an incomplete pattern OR ignitable liquid mixtures may be masked by the presence of only the most volatile components.
- 4.3.1.3 Must follow-up with a more comprehensive recovery method such as solvent extraction or adsorption-elution if more complex hydrocarbon mixtures are to be identified.

4.3.2 Minimum Standards and Controls

- 4.3.2.1 Use a new disposable syringe for each sample and standard injected.
- 4.3.2.2 A syringe/room air blank must be run prior to each sample. These blank runs must be satisfactory, having no significant peaks in the region of interest, before injecting case samples.

4.3.3 Analytical Procedures

- 4.3.3.1 Using a disposable syringe that has been flushed with room air, draw off about 1 ml of vapor from the interior of the container.
- 4.3.3.2 Inject the vapor sample directly into the GC-MS. Generally, the headspace method is used.
 - 4.3.3.2.1 If no significant or identifiable peaks of interest are present continue with another recovery method. Peaks that are considered significant should be pursued, however, if they are not pursued, an explanation shall be recorded in the case notes.
 - 4.3.3.2.1.1 The presence of an oxygenated solvent would be considered significant if it is present at an abundance of at least one order of magnitude above the overall matrix peaks in the chromatograph. If there are no matrix peaks in the chromatograph, the oxygenated solvent would be significant if it were present at an abundance of at least one order of magnitude above the background.
 - 4.3.3.2.1.2 The examiner may pursue an oxygenated solvent for identification should he/she feel that the presence of the solvent at low levels would be significant to the case. (i.e., Isopropanol is observed at low levels in a case where the investigator states that rubbing alcohol is suspected to have been used as an ignitable liquid.)

- 4.3.3.2.2 If a peak(s) of interest is present, run a standard(s) using the library search as a guideline.
- 4.3.3.2.2.1 Inject the vapor sample directly into the polar column GC. A semi-polar column may be used, if necessary.
- 4.3.3.2.2.2 Inject the appropriate standard(s).
- 4.3.3.2.2.3 Continue with another recovery method.

4.3.4 References

- 4.3.4.1 ASTM E 1388-12, Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples.
- 4.3.4.2 Caddy B., Smith F. P., Macy J.: Methods of Fire Debris Preparation for Detection of Accelerants: *Forensic Science Review* 3: 57; 1991.
- 4.3.4.3 Midkiff, Charles R. "Arson and Explosive Investigation," *Forensic Science Handbook*, Volume 1, 2nd Edition, Pearson Education, Inc., 2002, pages 479-524.
- 4.3.4.4 Stauffer, E., Dolan, J., and Newman, R., *Fire Debris Analysis*, Burlington, MA, Elsevier, Inc., 2008, pp. 395-399.
- 4.3.4.5 ASTM E 1618-14, Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.
- 4.3.4.6 Phelps, J. L., Chasteen, C. E., Render, M. M., "Extraction and Analysis of Low Molecular Weight Alcohols and Acetone From Fire Debris Using Passive Headspace Concentration," *Journal of Forensic Sciences*, Vol. 39, No. 1, Jan. 1994, pp. 194-205.

4.4 Solvent Extraction

4.4.1 Purpose

The purpose of a solvent extraction is to remove petroleum products which may be present in the debris. This extract may be concentrated by evaporation, if needed, and a portion injected into the gas chromatograph-mass spectrometer (GC-MS).

Special considerations that should be noted with this extraction technique:

- 4.4.1.1 Many components from the debris are also soluble in pentane and these may interfere with chromatogram interpretation. Generally, solvent extraction is not preferred for the extraction of clothing, leather goods and melted plastics due to these interferences.

4.4.2 Safety Considerations

- 4.4.2.1 Pentane (C₅) should be kept from heat, heat sources and sources of ignition.
- 4.4.2.2 Perform extractions in a fume hood.

4.4.3 Minimum Standards and Controls

- 4.4.3.1 Previously cleaned glassware is rinsed with pentane prior to use.
- 4.4.3.2 With this procedure an extraction blank must be obtained at the same time as the debris extraction. Approximately the same volume of pentane decanted off for the extraction should

be used for the extraction blank and this extraction blank should be reduced to at least the same approximate volume as the most reduced sample. For example, if 80 ml of pentane is used to extract the debris and only approximately 50 ml is decanted off and the extract is evaporated to 2 ml, then use approximately 50 ml of pentane and evaporate to 2 ml for the extraction blank. Compare the extraction blank to the debris extract(s).

- 4.4.3.3 If the sample extract is to be concentrated by evaporation, the extraction blank will be evaporated a similar amount. A copy of the extraction blank will be placed in each case file extracted at that time.

4.4.4 Analytical Procedures

- 4.4.4.1 Add pentane to the selected debris. The amount of pentane needed will vary with the amount and kind of debris. Use enough solvent to permit thorough "wetting" of the debris.
- 4.4.4.2 The debris may be transferred to a beaker, evaporating dish or other suitable container or may be wetted with pentane while inside the original paint can container.
- 4.4.4.3 As appropriate, the entire sample or a representative portion may be selected for extraction. If only a portion is extracted, a description of the portion selected is recorded in the case notes.
- 4.4.4.4 If the extraction is performed in a beaker and the debris is flexible/absorbent, a smaller beaker may be used as a plunger to promote removal of the pentane from the debris.
- 4.4.4.5 If the debris is nonporous, the extraction may be performed by rinsing the surfaces.
- 4.4.4.6 Decant the pentane, filter and concentrate, as necessary.
- 4.4.4.6.1 Decant the pentane into an evaporating dish, beaker or other suitable container.
- 4.4.4.6.1.1 If only pentane is present, Whatman #1 or #1PS filter paper or a Pasteur pipette with a small plug of glass wool may be used.
- 4.4.4.6.1.2 If two phases are present (i.e., pentane and water), the water may be removed by using Whatman #1PS filter paper or a separatory funnel.
- 4.4.4.6.1.3 Alternatively, either the solvent layer or the water layer may be removed with a Pasteur pipette.
- 4.4.4.6.1.4 If filter paper or glass wool is used, filter the extraction blank as well.
- 4.4.4.6.2 Alternatively, decant the pentane into an Erlenmeyer flask. Place the flask in a dish of warm water or an ultrasonic cleaner and apply a vacuum to a disposable pipette inserted into a rubber stopper in the neck of the flask.
- 4.4.4.6.3 The pentane may be concentrated using a disposable pipette attached to a vacuum line to pull off the vapors above the extract, by using house air with, or without, a microevaporator, or by allowing the uncapped vials to sit in the hood.
- 4.4.4.7 The amount of concentrating (evaporating) will be based upon the sample. A strong sample (one which possesses an unavoidable strong petroleum odor) may need little to no evaporation. A sample that is weak (one which does not possess an unavoidable petroleum odor) should be evaporated down to about 1.0 – 0.5 ml.
- 4.4.4.8 Transfer extract to a labeled vial.

4.4.4.9 Record in the notes the approximate volume of pentane “in”, the approximate volume of pentane “out” and the approximate volume to which the pentane is reduced for both the case sample(s) and the extraction blank, as appropriate.

4.4.4.10 Inject an appropriate volume of pentane into the GC-MS.

4.4.5 References

4.4.5.1 ASTM E 1386-10, Standard Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris Samples by Solvent Extraction.

4.4.5.2 Caddy B., Smith F. P., Macy J.: “Methods of Fire Debris Preparation for Detection of Accelerants”: Forensic Science Review 3: 57; 1991.

4.4.5.3 Midkiff, Charles R. "Arson and Explosive Investigation," Forensic Science Handbook, Volume 1, 2nd Edition, Pearson Education, Inc., 2002, pages 479-524.

4.4.5.4 Stafford, David T. "Fire Investigation. Part II: Laboratory Investigation", Crime Laboratory Digest Vol. 14, No. 1 January 1987.

4.4.5.5 Stauffer, E., Dolan, J., and Newman, R., Fire Debris Analysis, Burlington, MA, Elsevier, Inc., 2008, pp. 395-399.

4.5 Passive Adsorption-Elution

4.5.1 Purpose

The purpose of the passive adsorption-elution extraction is to remove volatile compounds, petroleum products and other ignitable liquids which may be present in the debris and put them into a liquid which can be analyzed via gas chromatography-mass spectrometry (GC-MS). Passive adsorption-elution is a headspace concentration method, also referred to as the **charcoal strip method**, which relies on the volatility of ignitable liquids. Volatilized ignitable liquids are removed by heat without a vacuum and are trapped on a charcoal strip. Ignitable liquids are eluted from the charcoal with a solvent (carbon disulfide) and analyzed.

Special considerations that should be noted with this extraction technique:

4.5.1.1 Samples containing a large amount of petroleum products can exhibit selective adsorption of the components.

4.5.1.2 If a sample resists heating, such as insulation, this extraction method is not preferred.

4.5.1.3 This extraction method may not fully recover petroleum products of low volatility. (i.e., Kerosene and Diesel Fuel cannot be distinguished by this method.) If a heavy petroleum distillate is indicated by the data, a follow-up pentane extraction may be conducted.

4.5.2 Safety Considerations

4.5.2.1 Carbon disulfide (CS₂) is a hazardous chemical with respect to both health and safety and must be handled with extreme care. Avoid physical contact with CS₂. Avoid inhalation of CS₂.

4.5.2.2 CS₂ should be kept from heat, heat sources and sources of ignition.

4.5.2.3 Perform elutions in a fume hood.

- 4.5.2.4 Care should be exercised when removing cans from ovens. Cans must be allowed to cool to approximately room temperature prior to opening for removal of the charcoal strip.
- 4.5.3 Minimum Standards and Controls
- 4.5.3.1 For each day that samples are prepared using this method, an extraction blank will be prepared and extracted in each oven used. To prepare an extraction blank, one pre-cut charcoal strip will be extracted using the same procedure as for the evidentiary samples. A copy of the extraction blank will be placed in each case file extracted that day.
- 4.5.3.2 If the sample extract is to be concentrated by evaporation, the extraction blank will be evaporated a like amount.
- 4.5.3.3 A new charcoal strip is used for every case item.
- 4.5.3.4 Ovens will be regularly checked to ensure they are functioning properly.
- 4.5.4 Analytical Procedures
- 4.5.4.1 The sample must be in a metal can or polyethylene terephthalate (PET) can.
- 4.5.4.2 Suspend a pre-cut charcoal strip inside the evidence container via a paper clip. Close the container.
- 4.5.4.3 Lids should be taped down during extraction.
- 4.5.4.3.1 If a lid does pop up or off, document this in the case file notes and determine if the extraction blank has been affected by this event.
- 4.5.4.4 Place the can in an oven set at 65 °C for approximately 16 hours.
- 4.5.4.4.1 Record the oven used for each case sample on the Fire Debris Worksheet.
- 4.5.4.5 Remove the cans from the oven and allow them to cool, if necessary.
- 4.5.4.6 Open the lid and remove the suspended strip. Place the strip into a fully labeled vial.
- 4.5.4.7 Add no more than 500 µL of CS₂ to the vial.
- 4.5.4.8 Inject a portion of the CS₂ extract into the GC-MS.
- 4.5.4.9 If the autosampler is to be used, agitate the vial, and transfer the extraction solution to a labeled autosampler vial fitted with an insert via disposable pipette.
- 4.5.4.9.1 Autosampler vials should be recapped if further analysis is necessary.
- 4.5.5 References
- 4.5.5.1 ASTM E 1412-12, Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris by Passive Headspace Concentration with Activated Charcoal.
- 4.5.5.2 Dietz, W.R., "Improved Charcoal Packaging for Accelerant Recovery by Passive Diffusion," *Journal of Forensic Sciences*, Vol. 36, No. 1, Jan. 1991, Pp 111-121.
- 4.5.5.3 Material Safety Data Sheet for Carbon Disulfide.

- 4.5.5.4 Newman, R.T., Lothridge, K. And Dietz, W., "The Use of Activated Charcoal Strips for Fire Debris Extractions by Passive Diffusion Part 1: The Effects of Time, Temperature, Strip Size, and Sample Concentration," *Journal of Forensic Sciences*, Vol. 41, No. 3, May 1996, Pp. 167-176.
- 4.5.5.5 Stauffer, E., Dolan, J., and Newman, R., *Fire Debris Analysis*, Burlington, MA, Elsevier, Inc., 2008, pp. 399-415.

4.6 Gas Chromatography-Mass Spectrometry (GC-MS)

4.6.1 Purpose

Gas Chromatography-Mass Spectrometry (GC-MS) provides a Total Ion Chromatogram (TIC) pattern for the sample as well as the ability to extract patterns for groups of specific ions. Extracted ion chromatography (EIC), or mass spectral ion profiling (MSIP), is especially useful when samples show a high degree of debris interference.

4.6.2 Analytical Procedures

- 4.6.2.1 Each day that the GC-MS is used for case samples, the Resolution Test Mixture (RTM) and the Oxygenate Check Sample (OCS) (1:2:1 acetone, ethanol and methylethylketone in CS₂) must be injected prior to case samples. This will serve as a check of the resolution and proper functioning of the instrument. The Fire Debris Check Sample (FDCS) may be injected into the GC-MS prior to analyzing case samples at the discretion of the examiner.

4.6.2.1.1 Acceptance criteria:

- 4.6.2.1.1.1 RTM: The retention times for all compounds must be within 0.033 minutes of the retention times from the previous RTM QC check.

OCS: The abundance from the baseline to the valley between the acetone and ethanol peaks must be no greater than approximately 10% of the abundance of the acetone peak.

- 4.6.2.1.1.2 If any acceptance criterion is not met, instrument maintenance shall be performed and documented.

- 4.6.2.1.1.3 If documented maintenance is conducted on the instrument which results in a retention time shift, an RTM must be run prior to any case samples.

- 4.6.2.1.2 Check samples shall be maintained in the laboratory for a minimum of six years.

4.6.2.2 Mass Spectral Quality Control

- 4.6.2.2.1 Full scan mass spectral classification of an ignitable liquid: No rigid TIC or Ion Profile match criteria are defined to classify a product. Flexibility is given to the experienced interpreter because rigid criteria can lead to misidentification as well as under-identification. Classification will be based on a number of factors, such as, relative retention times, compounds present, patterns present in the TIC as well as the patterns and relative profile abundances present in the Ion Profiles, contribution from the matrix and literature references.
- 4.6.2.2.2 Whenever possible, the classification of an ignitable liquid product should be based on a comparison of TIC and Ion Profile patterns from the sample and from

an actual reference material. If a reference material is not available, literature references or internal historical data may be used.

- 4.6.2.2.3 The data files for the reference materials used for direct comparison to the samples should be collected within approximately two months of the sample data files. These data files may only be used if no instrument maintenance affecting the RTM acceptance criteria retention times was completed within the approximate two month time period.
- 4.6.2.3 Systematic naming shall be used for standard fire debris methods with the autosampler: AUTF + injection volume + split. Systematic naming shall be used for standard fire debris methods using manual injection: MANF + split.
- 4.6.2.4 The injection volume and split ratio will vary for the AUTF and MANF methods, but the temperature and mass spec acquisition parameters will be the same.
- 4.6.2.4.1 These methods cannot be changed without prior approval of the Chemistry Program Manager.
- 4.6.2.5 Systematic naming and content shall be used for standard fire debris macros.
- 4.6.2.5.1 These macros cannot be changed without prior approval of the Chemistry Program Manager.
- 4.6.2.6 A portion of the extract or whole liquid is injected into the GC-MS.
- 4.6.2.7 A Library Summary Report may be generated to give an indication of the types or classes of compounds present when many peaks are present in the TIC.
- 4.6.2.8 The data handling system is used to extract selected ion chromatograms for each compound type. Two or more characteristic ions are summed to enhance signal-to-noise and decrease interference by extraneous compounds that contain only one of the ions. The following ions are used for the individual classes of hydrocarbons:
- Alkanes: Ions 57, 71, 85, and 99
 - Aromatics: Ions 91, 105, 119, 134
 - Indanes: 117, 131
 - Olefins/Cycloparaffins: 55, 69, 83
 - Polynuclear aromatics (PNAs): 128, 142, 156
- 4.6.2.9 Total ion chromatograms and extracted ion chromatograms of the unknown sample are visually compared against the corresponding extracted ion chromatograms from reference samples.
- 4.6.3 References
- 4.6.3.1 ASTM E 1618-14, Standard Guide for Identification of Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry.
- 4.6.3.2 Newman, R., Gilbert, M., Lothridge, K. "GC-MS Guide to Ignitable Liquids," CRC Press, 1998, pages 1-5.
- 4.6.3.3 Stauffer, E., Dolan, J., Newman, R. "Fire Debris Analysis," Elsevier, Inc., 2008, pages 235-293.

4.7 Identification Criteria

- 4.7.1 Possible reported results include: No Identification or Identification.
- 4.7.1.1 Used less frequently, but still appropriate in some circumstances, would include: Consistent.
- 4.7.2 No Identification - The sample does not contain sufficient peaks/pattern that correlate to reference patterns on file. The report would generally read: No ignitable liquids were identified in the Item ___ extract.
- 4.7.2.1 If the sample contains some peaks/patterns (as indications) of a reference pattern on file, but due to being weak, having interferences, or other factors, no identification is made, a descriptive sentence may be added to the report. This would generally read: However, the data contains some peaks/patterns of a ___reference sample but, due to being weak, having interferences or other factors, an identification could not be made.
- 4.7.2.1.1 At least one “reason” must be listed in the descriptive sentence.
- 4.7.3 Identification - The sample contains sufficient peaks/pattern correlating to a reference pattern on file. There is no set number assigned to the number of peaks that must correlate. It is the overall patterns that are compared to the reference patterns. The report would generally read: The Item __ extract contained a _____.
- 4.7.4 Consistent – The sample contains peaks/patterns that correspond well to portions of a reference pattern or a combination of reference patterns on file but there is no one reference pattern that correlates in its entirety with the data. The report would generally read: The Item __ extract was consistent with containing a _____.

4.8 Documentation

- 4.8.1 As a minimum, each case file will include:
- A Fire Debris Worksheet(s),
 - One or more full page, landscape format, integrated TIC’s covering the entire sample run time for each Item of evidence analyzed,
 - One or more full page, landscape format, integrated TIC’s covering the region of interest for each Item of evidence analyzed with pattern at least 75% on scale,
 - Blanks before each Item of evidence analyzed. Either full page, landscape format covering the region of interest at a minimal abundance or, if identifying individual peaks, extracted ions for the individual compounds over the region of interest,
 - One or more sets of EIC’s for each Item of evidence analyzed, as appropriate,
 - Data for each reference necessary to support the conclusions,
 - An extraction blank and, as appropriate, a concentrated extraction blank,
 - Resolution Test Mixture and Oxygenate Check Sample integrated TICs,
 - A Library Search Report for all samples being reported as negative, as appropriate. For example, it would not be necessary to include a Library Search Report for a sample showing no peaks.
- 4.8.2 Each case sample shall be checked at the lowest abundance setting as practicable to ensure that no other patterns are present that should be addressed.
- 4.8.3 The minimum requirements for labeling data are as follows:
- Case Number
 - Item Number
 - Handwritten Examiner’s Initials
 - Injection volume (except Auto method)

- Injection solvent
 - Note if the original sample has been concentrated
 - If the reference has been diluted, note solvent used
- 4.8.4 Blanks shall be printed at the same abundance value as or less than the case sample chromatograms.
- 4.8.5 When identifying single components such as alcohol, acetone, pinene, etc., retention time data for samples and standards are needed from at least the semi-polar or polar GC (or GC-MS) column along with retention time and spectral data from the nonpolar GC-MS column.
- 4.8.6 When identifying normal alkanes products, retention times and mass spectra for the major alkane peaks are necessary.
- 4.8.6.1 At a minimum, retention times and mass spectra should be compared to primary standards. In addition, a minimum of one (1) product from the reference collection should be included as an example of a commercially available normal alkanes product.
- 4.8.6.1.1 If an appropriate commercially available normal alkanes product is available and examined for inclusion in the case file for pattern comparison, only the nonpolar GC-MS retention time data and mass spectra are necessary.
- 4.8.6.1.2 If no appropriate commercially available normal alkanes product is available or if only one or two normal alkanes are present for identification (no pattern exists), retention time data from a second GC system is necessary.
- 4.8.7 For EIC: at a minimum, include the following:
- A single-page, portrait format, printout of a series of five summed extracted ion profiles (EIPs) covering the entire sample/standard run time, including
 - Alkanes (57 + 71 + 85 + 99)
 - Aromatics (91 + 105 + 119 + 134)
 - Indanes (117 + 131)
 - Olefins/Cycloparaffins (55 + 69 + 83)
 - Polynuclear aromatics (PNAs) (128 + 142 + 156)
 - A single page portrait format printout of the series of five summed extracted ion profiles covering the sample/standard run time of interest.
- 4.8.8 If GC-FID chromatograms have also been generated, include: one or more chromatograms for each Item of evidence analyzed and each reference necessary to support the conclusions, blanks before each Item of evidence analyzed, and a check sample. Label chromatograms as per ¶4.8.3.
- 4.8.8.1 To the maximum extent possible, case samples and references need to be at least 75% on scale or greater in the printed chromatogram.
- 4.8.8.2 Blanks shall be printed at the same mV value or less than the case sample chromatograms

4.9 Report Wording

To the maximum extent possible, report RESULTS wording will be selected from the following:

- 4.9.1 If a container is submitted and not described with any detail in the item description, then provide this information in the Results.
- 4.9.1.1 For example: Item 1 White plastic bottle. RESULTS: Item 1 was commercially labeled, "Safeway Charcoal Lighter...32 FL OZ".

4.9.1.2 In some instances it may be more appropriate to report that Item 1 was commercially labeled in part... or that the Item 1 bottle was melted and charred and the only legible labeling read...

4.9.1.3 When the liquid from the container or the extract of the container is determined to be consistent with what would be its expected use: ...consistent with the Item _____ product as labeled; consistent with the contents label on Item _____; consistent with the labeling on the Item _____ container.

For example: Item 1 White plastic bottle RESULTS: Item 1 was commercially labeled, "Safeway Charcoal Lighter...32 FL OZ". The Item 1 liquid was identified as a medium petroleum distillate which is consistent with the labeling on the Item 1 container.

Or: Item 5 Bottle of Lamplight Farms Cherry Lamp Oil RESULTS: The Item 5 extract contained a medium naphthenic-paraffinic product consistent with the product as labeled.

4.9.2 Tests used in the analysis shall be identified in the body of the report. The report will generally read:

- Item _____ was extracted using a passive adsorption-elution technique. The Item _____ extract was examined using Gas Chromatography-Mass Spectrometry (GC-MS).
- Items _____ and _____ were extracted using a passive adsorption-elution technique. The Item _____ and _____ extracts were examined using Gas Chromatography-Mass Spectrometry (GC-MS).
- Item _____ was extracted using ambient headspace and passive adsorption-elution techniques. The Item _____ extracts were examined using Gas Chromatography (GC) and Gas Chromatography-Mass Spectrometry (GC-MS).

4.9.3 No ignitable liquids were identified in the Item _____ extract.

4.9.4 No ignitable liquids were identified in the Item _____ or _____ extracts.

4.9.5 No ignitable liquids were identified in the extract of the Item _____ aqueous liquid.

4.9.6 The Item _____ extract contained _____.

4.9.7 The Item _____ extract contained _____ which can be found in, but is not limited to, some _____ (give examples of general product types).

4.9.8 The Item _____ and _____ extracts each contained _____.

4.9.9 The Item _____ extract contained a mixture of _____ and _____.

4.9.10 The Item _____ and _____ extracts each contained a mixture of _____ and _____.

4.9.11 The combined Item _____ and _____ extracts contained a mixture of _____ and _____.

4.9.12 The combined Item _____ and _____ extracts contained a mixture of _____ and _____. The _____ can be found in, but is not limited to, some _____ (give examples of general product types).

4.9.13 The Item _____ liquid was identified as _____.

4.9.14 The Item _____ and _____ liquids were each identified as _____.

4.9.15 No ignitable liquids were identified in the Item ___ extract. However, the data contained some peaks/patterns of a ___ reference sample but, due to being weak, having interferences, or other factors, an identification could not be made.

4.9.16 Due to the nature of heavy oil/lubricant petroleum products, there is some flexibility in how they may be reported.

The Item _____ extract was consistent with containing a heavy oil/lubricant petroleum product. Consistent with means that the data contained peaks/patterns that corresponded well to portions of a reference pattern or a combination of reference patterns on file but there was no one reference pattern that correlated in its entirety with the data.

4.9.16.1 If physical properties of a heavy oil/lubricant petroleum product are also present:

The Item _____ extract contained a heavy oil/lubricant petroleum product.

The Item _____ extract contained gasoline and was physically and chemically consistent with containing a heavy oil/lubricant petroleum product. Chemically consistent with means that the data contained peaks/patterns that corresponded well to portions of a reference pattern or a combination of reference patterns on file but there was no one reference pattern that correlated in its entirety with the data.

4.9.17 If an aqueous liquid is encountered, report as follows:

4.9.17.1 The extract of the Item _____ aqueous liquid contained _____.

4.9.18 If applicable, an aqueous and a nonaqueous liquid may be described as “immiscible”.

Item _____ Two immiscible liquids

RESULTS The Item _____ nonaqueous liquid was identified as _____.

The Item _____ aqueous liquid consisted essentially of _____. or,
The extract of the Item _____ aqueous liquid contained _____.

4.9.19 When data suggests compounds present in both wood and turpentine and wood is present or may be present in an item, the examiner may identify the α -pinene and report as follows:

The Item _____ extract contained α -pinene. It should be noted that α -pinene occurs naturally in some woods and is also found in turpentine and some cleaning products.

4.9.19.1 If more than one terpene is identified, either list the individual compounds identified or use the word terpenes [e.g., The Item _____ extract contained terpenes. It should be noted that terpenes (or list the individual compounds) occur naturally in some woods and are also found in turpentine and some cleaning products.]

4.9.20 When data suggests compounds present in turpentine and the item is a whole liquid, then report as follows:

The Item _____ liquid was physically and chemically consistent with turpentine (a medium miscellaneous product).

4.9.21 When data suggests compounds present in turpentine and no wood is present and the item is an extract, then report as follows:

The Item _____ extract contained _____ (terpenes or list individual compounds). It should

be noted that terpenes (or list the individual compounds) occur naturally in some woods and are also found in turpentine and some cleaning products.

4.9.22 When reporting normal alkanes products when a commercially available product is available:

The Item _____ extract contained a mixture of decane, undecane and dodecane (light, medium or heavy normal alkanes product), which can be found in, but is not limited to, some lamp oils.

4.9.22.1 If physical properties of wax are also present:

Item _____ contained a paraffin wax.

4.9.23 When reporting normal alkanes when no commercially available product is available or if only one or two normal alkanes are identified, the individual alkanes should be listed on the report.

The Item _____ extract contained a mixture of undecane and dodecane (normal alkanes).

4.9.24 If the RFLE requests analysis for volatiles (e.g., ethanol, acetone, toluene) only, select as appropriate from the following:

The Item _____ liquid contained _____ (e.g., methanol, ethanol, isopropanol, acetone) (an oxygenated solvent).

The Item _____ liquid consisted essentially of _____ (e.g., methanol, ethanol, isopropanol, acetone) (an oxygenated solvent).

No oxygenated solvents or other ignitable liquids were identified in the Item _____ extract.

No alcohols or other ignitable liquids were identified in the Item _____ extract.

No ethanol was identified in the Item _____ extract.

No ethanol or other ignitable liquids were identified in the Item _____ extract.

No ignitable liquids were identified in the Item _____ extract.

The Item _____ extract contained _____ (e.g., ethanol, acetone, toluene) (an oxygenated solvent).

4.9.24.1 If appropriate, common sources of the compound may be included:

Item 1 consisted essentially of isopropanol (an oxygenated solvent), commonly referred to as rubbing alcohol.

4.9.25 Generally speaking, the ignitable liquid classification categories reported are based on ASTM E1618. With the exception of gasoline, these classes are further classified as light, medium or heavy based on n-alkane (boiling) range. A light product is generally n-C4-n-C9, a medium product is generally n-C8-n-C13, and a heavy product is generally n-C9-n-C20+. A product may also be classified as "light to medium" or "medium to heavy" as appropriate. The ignitable liquid classification categories are as follows:

- Gasoline,
- Petroleum Distillates (Light, Medium or Heavy),
- Isoparaffinic Products (Light, Medium or Heavy),
- Aromatic Products (Light, Medium or Heavy),
- Naphthenic-Paraffinic Products (Light, Medium or Heavy),
- Normal-Alkanes products (Light, Medium or Heavy),

- Oxygenated Solvents, and
- Miscellaneous products (Light, Medium or Heavy).

4.9.25.1 The commercial products used as examples for Heavy Petroleum Distillates include Kerosene or Diesel Fuel as appropriate.

Heavy Oil/Lubricant petroleum products may also be reported. This classification is generally not considered ignitable and is not covered under ASTM 1618.

A minimum of two (if possible) and maximum of three (if necessary) examples of commercial products would typically be listed for most of the above ignitable liquid classifications. The examples used most routinely for medium petroleum distillates are some mineral spirits, paint thinners and charcoal starter fluids.

4.9.25.2 Certain types of debris (such as leather, newsprint, and carbonless copy paper) are known to give petroleum product patterns on GC-MS. When this type of debris is analyzed and a petroleum product is identified, the following statement should be used:

It should be noted that some _____ (e.g., leather goods) may contain _____ (e.g., heavy petroleum distillates).

4.9.25.3 If the example used is an appropriate “heavy petroleum distillate”, add the sentence that Kerosene or Diesel Fuel are examples of heavy petroleum distillates.

It should be noted that some leather goods may contain a heavy petroleum distillate. Kerosene and Diesel Fuel are examples of heavy petroleum distillates.

4.9.26 Generally speaking, when a comparison between items has been requested and the result is implied, then no specific mention of the comparison is made in the CoA.

4.9.26.1 The results for both items are the same classification, e.g., Items 1 and 2 contain gasoline; therefore, it is implied that they could have had a common origin.

4.9.26.2 The results for both items are a different classification, e.g., Item 1 contains gasoline and Item 2 contains a medium isoparaffinic product; therefore, it is implied that they could not have had a common origin.

4.9.27 Upon occasion it may be necessary to include a statement regarding the comparison of samples. For example:

4.9.27.1 The Item ____ liquid was identified as acetone (an oxygenated solvent) and is consistent with the labeling on the can. Therefore, Item ____ was eliminated as a possible source of the Item ____ light petroleum distillate due to differences in chemical composition.

4.9.27.2 Item ____ could not be associated with the Item ____ extract due to differences in chemical composition.

4.9.28 When a homemade napalm material is received, in addition to the petroleum product, the identity of the added material should be pursued.

4.9.28.1 The Item ____ extract contained gasoline. Additionally, Item ____ was physically and chemically consistent with containing _____. (e.g., polystyrene, soap)

4.9.28.2 The following statement may also be added: It should be noted that some mixtures of gasoline and _____ (e.g., polystyrene, soap) may be referred to as “napalm”.

4.9.29 Full Report Wording Examples – Fire Debris

4.9.29.1 Example 1

Item 1 Fire debris from couch
 Item 2 Fire debris from living room floor

RESULTS

Items 1 and 2 were extracted using a passive adsorption-elution technique. The Item 1 and 2 extracts were examined using Gas Chromatography-Mass Spectrometry (GC-MS).

The Item 1 extract contained gasoline.

No ignitable liquids were identified in the Item 2 extract.

The evidence is being retained for personal pickup.

4.9.29.2 Example 2

Item 12 Carpet and padding from living room
 Item 13 Carpet and padding from dining room
 Item 14 Carpet and padding from bedroom
 Item 15 Liquid from red plastic container on porch

RESULTS

Items 12 through 14 were extracted using a passive adsorption-elution technique. The Item 12 through 14 extracts were examined using Gas Chromatography-Mass Spectrometry (GC-MS).

Item 15 was examined using Gas Chromatography-Mass Spectrometry (GC-MS).

The Item 15 liquid was identified as gasoline.

No ignitable liquids were identified in the Item 12 or 13 extracts. However, the data contained some peaks/patterns of a gasoline reference sample but, due to being weak, having interferences, or other factors, an identification could not be made.

The Item 14 extract was consistent with containing a heavy oil/lubricant product. Consistent with means that the data contained peaks/patterns that corresponded well to portions of a reference pattern or a combination of reference patterns on file but there was no one reference pattern that correlated in its entirety with the data.

The evidence is being retained for personal pickup.

4.9.29.3 Example 3

Item 6 Liquid from plastic bottle
 Item 7 Shirt from victim

RESULTS

Item 6 was examined using Gas Chromatography-Mass Spectrometry (GC-MS).

Item 7 was extracted using a passive adsorption-elution technique. The Item 7 extract was examined using Gas Chromatography (GC) and GC-MS.

The Item 6 liquid was identified as a medium petroleum distillate which can be found in, but is not limited to, some mineral spirits, paint thinners and charcoal starter fluids.

The Item 7 extract contained isopropanol (an oxygenated solvent), commonly referred to as rubbing alcohol.

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5 FRACTURE MATCH**5.1 Purpose**

To physically fit evidentiary materials back together along fractured edges. When accomplished, this examination provides conclusive evidence that the materials were at one time a single unit.

5.2 Analytical Approach

Items being submitted for fracture match, or physical fit, analysis include a variety of materials. Typically, when a material having reasonable rigidity breaks, irregularities appear at the break which may create pieces analogous to those of a “jigsaw puzzle”. Other types of materials may be subject to separation by cutting, tearing, shattering and so forth. When matching pieces are found that can be physically fitted back together again, the examiner will say with absolute certainty that the matching pieces were at one time a single unit.

5.2.1 Fiber examiners will conduct fracture match examinations of textile materials or other materials that contain fabric/fibers (e.g., duct tape).

5.3 Minimum Standards and Controls

All reported identifications (K to Q) will be verified by a second qualified examiner who will conduct the verification from the original evidence. If the initial examiner has reconstructed the material for the fracture match, it may be left assembled for the verifying examiner. The examiner conducting the verification must take the pieces apart to verify the fracture match(s). The examiner’s case file notes shall be initialed and dated by the second examiner who will include a notation to indicate that the fracture match conclusion was verified.

5.4 Analytical Procedures

- 5.4.1 Spread a clean piece of paper on the examination surface.
- 5.4.2 Examine each item of evidence visually to determine the correct orientation of the pieces. Begin with matching a surface (e.g., the outer color coat of paint chips, the fluorescing surface of float glass pieces, the smooth or finished surface of plastic fragments, etc.). After the pieces are marked for identification and “sided”, contours, edges, colors, surface markings and so forth, can be used to help align pieces correctly. It is often helpful to fit together pieces of a given item to themselves (“knowns”) and then fit them to additional items (“questioned”) in the case.
- 5.4.3 Observe all orientations of the fit, using stereomicroscopy when possible and appropriate. Scratches, stains or defects that traverse the broken, cut or torn edge may serve to reinforce the physical fit conclusion.
- 5.4.4 Observe the matching sides “on end” when possible and appropriate. Matching hackle marks and defects may be seen and serve to reinforce the physical fit conclusion.
- 5.4.5 A cast of one or more of the separated edges may be prepared for comparison purposes. The cast (s) shall be designated as a sub-item(s) of the evidence from which it was generated, listed on the RFLE, in LIMS, and on the CoA. Casts shall be individually sealed and returned in the same container from which the sub-item was generated.
- 5.4.5.1 Casting is a procedure used to create a reverse image of a fractured surface, which can be used for comparison purposes.
- Mix casting compound as per manufacturer’s instructions
 - Apply casting material over the surface to be cast
 - When casting material is set, gently loosen the cast from the item
 - Consideration should be given regarding placement of identifying/orientation marks onto the item being cast

- 5.4.6 Matching fabric pieces involves examination of general size and shape, construction, fiber type, colors, and long versus short threads.
- 5.4.7 When matching flexible materials (e.g., fabric, tape and some plastics), care must be taken to account for edge rolling, stretching, and twisting. Use of double stick tape on a rigid plate may be useful to stabilize the edge during comparison and to reduce the effects of distortion of stretching or twisting.

5.5 References

- 5.5.1 Saferstein, Richard, Criminalistics: An Introduction to Forensic Science, Prentice-Hall, Inc., 1995, pp. 65-67.
- 5.5.2 "Mikrosil Casting Material Information". AFTE Journal. Vol. 15, No. 2, p. 80.
- 5.5.3 "A Simple, Inexpensive Method for Making Castings from Silicone Molds". AFTE Journal. Vol. 47, No. 4, p. 234-236.
- 5.5.4 Barber, D.C. and Cassidy, F.H. "A New Dimension with 'Mikrosil' Casting Material". AFTE Journal. Vol. 19, No. 3, p. 328.
- 5.5.5 Ojena, S. M. , "A New Silicone Rubber Casting Material Designed for Forensic Science Application", Journal of Forensic Sciences, JFSCA, Vol. 29, No. 1, Jan. 1984, pp. 317-321.

5.6 Documentation

Along with case file notes, documentation for fracture match cases will include one or more of the following:

- Photographs of the fracture match
- Detailed sketches of the fracture match

- 5.6.1 Each of the above is to be documented within the case notes and should have legible markings for identification which are also described in the notes if not obvious from the photograph. These notations may be made directly on photographs or sketches in the notes.
- 5.6.2 The method of documentation should be appropriate for each individual case and will be left to the discretion of the examiner.

5.7 Report Wording

- 5.7.1 The techniques utilized in the analysis will be listed on the report.

Items _____ and _____ were examined visually and using stereomicroscopy.

A cast of Item 1 was created, designated as Item 1A, and compared to Item 2. Items 1, 1A and 2 were examined visually and using stereomicroscopy.

- 5.7.2 The reporting of "fracture match" results falls into 3 categories: positive, inconclusive with additional testing to be conducted (in most situations) and disassociation. It is recognized that some minor variations may occur to these statements.

5.7.2.1 Positive:

Items ___ and ___ were physically fitted together and were at one time a portion of a single unit.

It was concluded that Item 1 and 2 were at one time a portion of a single unit.

One (1) of the eleven (11) fragments in Item ____ was physically fitted to the Item ____ headlamp assembly. It was concluded that this Item ____ fragment and the Item ____ headlamp assembly were once a portion of a single unit.

5.7.2.2 Inconclusive with additional testing to be conducted:

Items __ and __ could not be physically fitted together. The results of additional examinations will be reported separately.

5.7.2.2.1 In a few situations further testing will not be possible and, therefore, this statement would not be applicable.

5.7.2.2.2 If the additional testing is conducted by the same Trace Evidence examiner, use the first sentence and include what testing was conducted and the conclusion that was reached.

For example:

Items __ and __ could not be physically fitted together. Items __ and __ were consistent in physical and chemical properties and could have had a common origin. (Or shape, construction, design characteristics, and so forth, as applicable.)

5.7.2.3 Disassociation: Items __ and __ could not be associated due to differences in _____ (color, width, construction, thickness, etc.)

5.7.2.3.1 Most important is to give a reason for the disassociation which could be of investigative importance to the investigating officer.

6 GENERAL CHEMICAL**6.1 Analytical Approach**

- 6.1.1 Items submitted for general chemical examinations are the miscellaneous samples not included in another examination (e.g., greases, glues, cosmetics, unknown substances). They are often referred to as any chemistry-related examination requested of the laboratory that is not a request for the identification of a controlled substance.
- 6.1.2 While some evidence types are encountered on a fairly routine basis, others may require method development at the time the case is being worked. No procedures manual could encompass methods for every general chemical evidence type analyzed. This section will provide analysis schemes for some evidence types that have been analyzed with some regularity.
- 6.1.3 Good scientific principles and a logical analysis scheme, along with appropriate consultations with the supervisor, are applied to those evidence types that have not been encountered before based upon the type and amount of evidence submitted and the case information received. Generally speaking, a visual macroscopic/ microscopic examination will be performed (stereo, compound, comparison and polarizing microscopes) for color, texture, viscosity, morphology, optical properties and homogeneity. Record the general amount of evidence received (e.g., the weight or volume).
- 6.1.4 Analytical approaches will vary depending upon the amount and type of sample received. With a limited amount of sample, nondestructive testing is performed first. Solubility/miscibility tests with organic/inorganic and polar/nonpolar solvents (e.g., water, carbon disulfide, pentane, acetone, chloroform) may be done. The pH of water soluble/miscible samples may be taken using a pH meter. Indicator strips (pH paper) may be used to screen for acids and bases prior to using a pH meter. Microchemical tests may be performed as indicators of a class of compounds present (e.g., for sulfates, chlorides, nitrates, sugars).
- 6.1.4.1 Reagents must pass the QC check prior to use in casework.
- 6.1.5 Necessary instrumentation is used to identify or to compare samples.
- 6.1.6 It may not be possible to fully identify an unknown. However, general classification is usually possible. When comparison samples are submitted or obtained, it may be possible to report that the unknown shares the same physical and/or chemical properties as the comparison sample. Reported conclusions shall be based upon the samples selected and examined.

6.2 Minimum Standards and Controls, Documentation, Report Wording

- 6.2.1 These are dependent upon the type of evidence being submitted and the examinations being performed.
- 6.2.2 Generally, blanks, reference standards and controls are run along with the evidence items, and all of these results are recorded in the case file notes. All documentation is included to support the conclusion made. Instrument output requirements are detailed throughout other sections of this manual. Report wording is as consistent as possible with previously reported exams of a given type. When reporting conclusions based upon the examination of “new” evidence types, the supervisor and/or technical reviewer will help ensure that the report wording is accurate, clear and concise.

6.3 Acids and Bases

6.3.1 Purpose

To identify common acids and bases that may be encountered in casework.

6.3.2 Safety Considerations

- 6.3.2.1 Acids and bases, e.g., HCl, HNO₃, H₂SO₄, H₃PO₄, NH₄OH, NaOH, KOH, may be encountered as evidence. These are very corrosive. Eye and skin protection must be used.
- 6.3.2.2 Acids may be very reactive with chlorates, acetone, flammable liquids and water. Extreme care must be taken when mixing these compounds.

6.3.3 Minimum Standards and Controls

- 6.3.3.1 Treat the questioned samples and any control/standard samples in the same manner.
- 6.3.3.2 When performing extractions analyze a blank of the extraction liquid and an undisturbed portion of the substrate, if possible.
- 6.3.3.3 Controls and blanks will be run with each case when performing testing with microchemical spot test reagents. These results will be recorded in the case file notes.

6.3.4 Analytical Procedures

- 6.3.4.1 If the sample is a solid or is on a solid substrate, dissolve in DI water. If the sample is a liquid, check if it is miscible with water by adding a small amount of the liquid to DI water.
- 6.3.4.2 A preliminary pH may be determined with pH paper. If an acid or base will be identified, the pH shall be determined using a pH meter.
- 6.3.4.3 If the pH is acidic proceed as follows:
- 6.3.4.3.1 Hydrochloric acid: Test unknown with a few drops of silver nitrate reagent. A white precipitate indicates the presence of a chloride. An optional follow-up test can be performed on the white precipitate by adding drops of ammonium hydroxide and noting whether it is soluble or insoluble. A white precipitate that is soluble in ammonium hydroxide further indicates the presence of chloride.
- 6.3.4.3.2 Sulfuric acid: Test unknown with a few drops of barium chloride reagent. A white precipitate indicates the presence of a sulfate. An optional follow-up test can be performed on the white precipitate by adding drops of concentrated acetic acid and noting whether it is soluble or insoluble. A white precipitate that is insoluble in concentrated acetic acid further indicates the presence of sulfate.
- 6.3.4.3.3 Nitric acid: Test unknown with a few drops of diphenylamine reagent. Immediate development of a deep blue color indicates the presence of nitrates.
- 6.3.4.3.4 Phosphoric acid: Test unknown by placing 6 ml of acetone in a small test tube. Place 3 drops of unknown liquid into the test tube and mix. Add 3 drops of ammonium hydroxide and mix. A precipitate indicates the presence of ammonium phosphate dibasic, (NH₄)₂ HPO₄, if phosphoric acid is present.
- 6.3.4.3.5 Specific anion test strips (such as EM Quant[®]) may be used in place of, or in addition to, any of the above microchemical tests. While these test strips may have an expiration date, they will continue to be used as long as they test positive with an appropriate known acid.
- 6.3.4.4 If the pH is basic proceed as follows:
- 6.3.4.4.1 Ammonium hydroxide: Test the unknown with Nessler's reagent. The formation of an orange to brown precipitate indicates the presence of ammonium ions.

Ammonium test strips (such as EM Quant[®]) may be used in place of, or in addition to, the Nessler's reagent test.

6.3.4.4.2 Sodium hydroxide: Basic pH (≥ 11).

6.3.4.4.2.1 When the sample is in pellet/solid form, sodium hydroxide is identified using XRD. The sample may be further analyzed using SEM-EDS, if appropriate.

6.3.4.4.3 Potassium hydroxide: Basic pH (≥ 11).

6.3.4.4.3.1 When the sample is in pellet/solid form, potassium hydroxide is identified using XRD. The sample may be further analyzed using SEM-EDS, if appropriate.

6.3.4.4.4 A report may be generated at this time if the results of the pH test and the spot tests are sufficient for the case report.

6.3.4.5 If further characterization of the acid/base is necessary, then the extract and/or the precipitate from the microchemical testing may be run using XRD, SEM-EDS, IC, or FTIR, as appropriate.

6.3.4.6 Ammonium Nitrate: Ammonium nitrate is not a strong acid or base, but it can be encountered with acids and bases, and its analysis is treated similarly. Unknowns can be presumptively tested with Diphenylamine (DPA) and/or Nessler's reagent. Ammonium test strips (such as EM Quant[®]) may be used in place of, or in addition to, the Nessler's reagent test. With DPA, immediate development of a deep blue color indicates the presence of nitrates. With Nessler's reagent, the formation of an orange to brown precipitate indicates the presence of ammonium ions. Ammonium nitrate is identified using XRD.

6.3.4.6.1 When ammonium nitrate is in pellet/solid form, XRD shall be utilized.

6.3.5 Report Wording

6.3.5.1 If the pH is neither acidic nor basic report:

No acids or bases were identified in Item ___ or the Item ___ extract. (As appropriate)

6.3.5.2 If the results of the pH test and the spot tests are sufficient for the case report:

The Item ___ extract was indicative of _____ (name of acid or base).

Item ___ was an acidic solution that was indicative of containing _____ (name of acid or base).

6.3.5.3 If identifying the acid or base:

Item 1 was an acidic/basic solution that contained _____ (name of acid or base).

Item 1 was identified as (sodium hydroxide, ammonium nitrate, etc.).

Item 1 was an acidic solution that contained dilute sulfuric acid, a listed substance in § 18.2-248 (J). Sulfuric acid is commonly found as battery acid and drain cleaners.

6.3.5.3.1 Alternatively, the following may be used as appropriate:

Item 1 was an acidic/basic solution consistent with _____ (name of acid or base).

6.3.5.3.2 If appropriate, common sources of the acid/base may be included:

6.3.5.3.2.1 Hydrochloric acid, commonly found as muriatic acid.

6.3.5.3.2.2 Sulfuric acid, commonly found as battery acid and drain cleaners.

6.3.5.4 Techniques used in the analysis shall be identified in the body of the report. See ¶ 1.9.2 for example wording.

6.3.6 References

6.3.6.1 Anger, V., and Feigl, F., Spot Tests in Inorganic Analysis, 6th ed., Elsevier Publishing Company: Amsterdam, The Netherlands, 1972.

6.3.6.2 Illinois State Police, Trace Chemistry Procedures Manual, Common Acids Identification, February 1, 1997.

6.3.6.3 Jungreis, Ervin.; "Spot Test Analysis, Clinical, Environmental, Forensic, and Geochemical Applications", Volume 75, 1985.

6.4 Bank Dyes

6.4.1 Purpose

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

6.4.2 Safety Considerations

6.4.2.1 Care should be taken as MAAQ is a prolific dye that does not wash off with water.

6.4.2.2 Lachrymators, such as CS, are compounds that irritate the eyes and mucous membranes and should be handled accordingly.

6.4.3 Minimum Standards and Controls

6.4.3.1 Treat the questioned samples and any control/standard samples in the same manner.

6.4.3.2 When performing extractions also analyze a blank of the extraction liquid and an unstained portion of the substrate, if possible. If an unstained portion of the substrate is not possible, analyze the extraction solvent as the negative control.

6.4.4 Analytical Procedures

6.4.4.1 Visually examine the evidence for any red, red-orange or pink stains.

6.4.4.2 Examine the evidence using short and long wave UV light, as needed.

6.4.4.3 Cut out small areas containing the stains of interest and extract with a minimum volume of chloroform or methanol.

- 6.4.4.3.1 If the areas of interest cannot be cut out, rub cotton swabs wetted with solvent (methanol or chloroform) over the stained areas.
- 6.4.4.3.2 If the stained area is dark and powdery, scrape the material into a sample vial and add solvent.
- 6.4.4.4 If the evidence is dark in color and no stains are visible, rub cotton swabs wetted with solvent over a small area of the item at a time. Observe any red, red-orange or pink color transferred to the swab and cut out these areas for extraction. If no color is transferred to the swab, continue swabbing additional areas as appropriate.
- 6.4.4.4.1 If no color is transferred to any of the swabs, no further testing is necessary.
- 6.4.4.5 If stained areas do not extract into methanol or chloroform, no further testing is necessary.
- 6.4.4.6 Use the same approximate size of unstained material, if available, for the substrate control or analyze the extraction solvent or unstained swabs wetted with extraction solvent.
- 6.4.4.7 If necessary, concentrate the extract to as dark a red color as possible. Concentrate the blank a like amount.
- 6.4.4.7.1 Do not take the extract to dryness because of the volatility of CS.
- 6.4.4.8 If the extract is relatively “clean”, the red dye may be analyzed using FTIR.
- 6.4.4.9 Analyze the extract using GC-MS.
- 6.4.4.10 If no peaks are present with the proper retention times and spectral data of CS or MAAQ, no further analysis is required.
- 6.4.4.11 If either or both compounds were present on GC-MS, analyze the extract on a semi-polar or polar GC or GC-MS column. (For carbowax, use the following conditions: Injector and Detector: 250° C, Oven: 80° C for 2min., Ramp: 15° C /min. to 250° C, Hold: 20 min. CS Rt ~ 11 minutes, MAAQ Rt ~ 27 min.)
- 6.4.4.12 Both the dye and the lachrymator (when present) should be identified when possible.
- 6.4.5 Report Wording
- 6.4.5.1 If a known bank dye pack is submitted and the dye is soluble in chloroform or methanol and none of the stains on the evidence extract into the solvent:
- The red stains on Item ____ could not be associated with the red dye in Item ____ due to differences in physical properties.
- 6.4.5.2 If a known bank dye pack is submitted, the dye is soluble in chloroform or methanol, and the stains on the evidence DO extract into the solvent:
- The Item _ and _ extracts each contained 1-methylaminoanthraquinone, a red dye [, and o-chlorobenzylidenemalononitrile, a lachrymator].
- 6.4.5.3 If NO known bank dye pack is submitted and all of the stains on the evidence do NOT extract into chloroform or methanol:
- The red stains on Item ____ could not be associated with the red dye commonly used in exploding bank dye packs due to differences in physical properties.

- 6.4.5.4 If stains extract but are not identified as CS and/or MAAQ:
- 6.4.5.4.1 No 1-methylaminoanthraquinone, a red dye commonly found in exploding bank dye packs, was identified in the Item ___ extract.
 - 6.4.5.4.2 No 1-methylaminoanthraquinone or o-chlorobenzylidenemalononitrile, common components of exploding bank dye packs, was identified in the Item ___ extract.
 - 6.4.5.4.3 If appropriate: The extract of Item ____ could not be associated with _____ due to differences in chemical properties.

- 6.4.5.5 If CS and/or MAAQ are identified:

The extract of Item ____ contained 1-methylaminoanthraquinone, a red dye, and o-chlorobenzylidenemalononitrile, a lachrymator. It should be noted that these materials are commonly found in exploding bank dye packs.

- 6.4.5.6 Techniques used in the analysis shall be identified in the body of the report. See ¶ 1.9.2 for example wording.

6.4.6 References

- 6.4.6.1 Martz, R.M., Reutter, D.J., and Lasswell, III, L.D., “A Comparison of Ionization Techniques for Gas Chromatography/Mass Spectroscopy Analysis of Dye and Lachrymator Residues from Exploding Bank Security Devices”, *Journal of Forensic Sciences*, Vol. 28, No. 1, January 1983, pp. 200-207.
- 6.4.6.2 Personal Communication, ICI Americas, Inc., August 1981.

6.5 Salt

6.5.1 Purpose

To identify sodium chloride (NaCl) and other salts that may be encountered in casework. The most routine cases requesting this examination are deer “baiting” cases, but the analysis scheme is readily applied to other situations as well. Samples submitted for this examination may consist of questioned crystalline material or soil samples suspected to have been adulterated with a questioned material.

6.5.2 Minimum Standards and Controls

- 6.5.2.1 Treat the questioned samples and any submitted control/standard samples in the same manner.
- 6.5.2.2 Controls and blanks will be run with each case when performing testing with microchemical spot test reagents. These results will be recorded in the case file notes.
- 6.5.2.3 Extraction procedures will not be conducted on questioned soil samples without an appropriate control soil sample.

6.5.3 Analytical Procedures

- 6.5.3.1 Examine the sample(s) with the aid of the stereomicroscope to determine if there are any obvious cubic crystals present. If cubic crystals are observed, remove them and perform identification by XRD.
 - 6.5.3.1.1 Some deer baiting products contain carbonates or bicarbonates as major components. Samples of this type are generally crystalline powders. Questioned soil will not be extracted for carbonates or bicarbonates. If applicable, a pH

measurement may be performed on the sample as a screening tool. If crystalline material is observed, remove and examine using XRD.

- 6.5.3.2 If cubic crystals are not observed, break up the sample material sufficiently to permit thorough wetting during the extraction process.
- 6.5.3.3 Weigh the samples. Use about one-half of the smallest sample's weight or approximately 50 grams, whichever is less, as the standard weight for all extraction samples.
- 6.5.3.4 Place each sample in a labeled beaker and add enough DI water to each beaker to cover the sample. Stir and allow samples to stand for 10 minutes.
- 6.5.3.5 Centrifuge and/or filter the samples and transfer each extract to a clean, labeled beaker.
- 6.5.3.6 Chloride ion screening: Perform silver nitrate spot test on the extract and record the results. If no precipitate forms, the results are negative and no further testing for NaCl is necessary.
- 6.5.3.6.1 If the results are indicative of a chloride, remove a portion of the extract for IC analysis. Evaporate the remaining portion to dryness and analyze the residue by XRD and, if necessary, SEM-EDS.
- 6.5.3.7 Salt will only be reported as "identified" in extracted samples when found in the questioned sample and when NOT found in the accompanying control sample.
- 6.5.4 Report Wording
- 6.5.4.1 If testing is negative for sodium chloride:
- No sodium chloride was identified in the Item __ extract.
- 6.5.4.2 If sodium chloride crystals are present:
- The white crystalline material in Item __ was identified as sodium chloride, commonly known as table salt.
- 6.5.4.3 If sodium chloride is identified from a water extract:
- The extract of Item __ contained sodium chloride, commonly found in table salt.
- 6.5.4.4 If the RFLE does not contain a specific request for sodium chloride it may be appropriate to report:
- No salts were identified in the Item __ extract.
- 6.5.4.5 Follow the above listed wording guidelines to report results of testing for carbonates as appropriate.
- 6.5.4.6 Techniques used in the analysis shall be identified in the body of the report. See ¶ 1.9.2 for example wording.

6.6 Sugars

6.6.1 Purpose

To identify common sugars (such as sucrose, dextrose, fructose or lactose) that may be encountered in casework. Sugar may be encountered in the vandalism of motor vehicles by its addition to the fuel supply or the crankcase oil. Because the solubility of sugar in gasoline is so low (1.5 mg/L), a sampling of any

solid residues in the fuel tank as well as the fuel or oil filters, as appropriate, should be included. Sugars may also be encountered as a cutting agent in controlled substance cases or as part of a sugar/chlorate mixture in improvised explosive devices.

6.6.2 Minimum Standards and Controls

- 6.6.2.1 Treat the questioned samples and any control/standard samples in the same manner.
- 6.6.2.2 When performing extractions also analyze a blank of the extraction liquid and an undisturbed portion of the substrate, if possible.
- 6.6.2.3 Controls and blanks will be run with each case when performing testing with microchemical spot test reagents. These results will be recorded in the case file notes.

6.6.3 Analytical Procedures

- 6.6.3.1 Stereoscopically examine the sample(s) to determine if there are any obvious crystals present and if the sample is homogeneous. If crystals are observed, remove them and perform solubility tests (aqueous/organic, such as water and chloroform, sugars are soluble in water).
- 6.6.3.2 If solid material is present, it may be necessary to wash away residue left on the solid material (for instance, gasoline in vandalism cases). Sugar is not soluble in pentane and this, or another suitable solvent, may be used.
- 6.6.3.3 If there is no solid material present, perform a water extraction of the sample containing the suspected sugar. (If the case involves an improvised explosive device, follow the procedures for explosives analysis.) Evaporate to dryness.
- 6.6.3.4 Perform either the Fehling's, triphenyltetrazolium chloride, naphthol or anthrone microchemical spot test. (With Fehling's or triphenyltetrazolium chloride, remember to acidify sucrose when performing the test. Run both sucrose and a known reducing sugar as positive controls.) A negative test is indicative of no sugars being detected.

6.6.3.4.1 Anthrone Microchemical Spot Test

Place several crystals of anthrone in a white spot plate. A powder sample, or an aqueous solution of the unknown, is added and mixed well. Three drops of sulfuric acid are added drop wise; a blue to blue-green color formation indicates the presence of sugar.

Notes: The color may take up to 5 minutes to form. Anthrone is normally used to detect carbohydrates. The test is most sensitive when a dry sample is ground with the anthrone prior to acid addition. Dilute water extracts are best evaporated to dryness prior to testing.

- 6.6.3.5 Use the FTIR or the XRD for identification.
- 6.6.3.6 Alternatively, use the trimethylsilylimidazole (TMSI) in pyridine derivatization procedure from Pierce Chemical Company on the GC (polar or semi-polar column) and the GC-MS.

6.6.4 Report Wording

- 6.6.4.1 If the questioned sample exhibits a negative microchemical spot test:

No sugars were identified in the Item ___ extract.

6.6.4.2 If the solid material or the extracted residues are identified as a sugar:

6.6.4.2.1 The Item ___ white crystalline solid was identified as _____ (name of sugar).

6.6.4.2.2 The Item ___ extract contained _____ (name of sugar).

6.6.4.3 If appropriate, common sources of the sugar may be named:

The Item ___ white crystalline solid was identified as sucrose, commonly referred to as table sugar.

6.6.4.4 Techniques used in the analysis shall be identified in the body of the report. See ¶ 1.9.2 for example wording.

6.6.5 References

6.6.5.1 Feigl, F. "Spot Tests In Organic Analysis" 7th edition, Elsevier Publishing, Amsterdam, 1966.

6.6.5.2 Jungreis, Ervin.; "Spot Test Analysis, Clinical, Environmental, Forensic, and Geochemical Applications", Volume 75, John Wiley & Sons, 1985.

6.6.5.3 1986-1987 Handbook and General Catalog, Pierce Chemical Company, Rockford, IL.

6.6.5.4 Spots Tests, Systematic Analysis of Low Explosives, Bureau of Alcohol, Tobacco and Firearms, 6/88.

6.7 Tapes and Adhesives

6.7.1 Analytical Approach

6.7.1.1 The examiner is typically requested to compare a questioned tape/adhesive sample to a known tape/adhesive source. The adhesive may be received as a part of a pressure sensitive tape or as an independent sample of adhesive. Instead of Q versus K comparison, the examiner may be requested to provide information regarding the manufacturer or end use of a tape/adhesive. If a tape sample contains fiber reinforcement, the case will be assigned to a fiber examiner.

6.7.1.2 The examiner first conducts a macroscopic examination of the tape/adhesive evidence keeping in mind the potential for the presence of other evidence (e.g., fibers, hairs, fingerprints). If appropriate, a fracture match comparison can be conducted.

6.7.1.3 Tapes/adhesives can be characterized by a number of physical and chemical properties.

6.7.1.4 The size and condition of the evidence may affect the ability to compare all properties. If necessary, the examiner may attempt to detach tape that is folded onto itself by using heat from a heat gun/hair dryer, liquid nitrogen, a "Dust-Off"-type product or solvents such as hexane, methanol, or another appropriate solvent.

6.7.1.5 During a macroscopic and microscopic examination, the examiner notes any physical properties which assist in characterizing the tape/adhesive. The adhesive and/or each component of the tape will be further compared with instrumental analysis including fluorescence microscopy and FTIR.

6.7.2 Macroscopic/Microscopic Examination of Tapes

6.7.2.1 Purpose

To examine submitted tape evidence on a macroscopic and microscopic scale, noting physical properties, generally as the first step in the characterization and comparison of known and questioned tape samples.

6.7.2.2 Minimum Standards and Controls

6.7.2.2.1 Clean tools and work areas are a must.

6.7.2.3 Analytical Procedures

6.7.2.3.1 Thoroughly clean the examination area prior to the evidence being retrieved.

6.7.2.3.2 The sample is examined visually and with a stereomicroscope and the physical properties of each tape sample are noted and recorded. At a minimum note the following physical properties, if applicable:

- width of tape in inches and millimeters
- color of backing
- color of adhesive
- texture of the backing
- thickness of the backing and adhesive in inches. (Overall thickness and thickness of backing alone.)
- any machine or calendering marks on the backing (Viewed with the polarized light attachment on the stereo microscope)

6.7.2.3.3 If fiber reinforcement is present, note at a minimum the following physical properties, if applicable:

- scrim pattern
- number of machine (warp) yarns per inch
- number of fill (weft) yarns per inch

6.7.2.3.3.1 The physical, chemical, and optical properties of the fibers will be compared and documented in accordance with the procedures for fiber examinations in Section 3.

6.7.2.3.4 Transparent tape backings should be visualized between crossed polarizing sheets (polarizing films) when comparing questioned vs. known. Record observations.

6.7.2.3.4.1 If the known and questioned cannot be eliminated, view the backing between crossed polars using the polarized comparison light microscope. Record observations.

6.7.2.3.4.2 The adhesive may be isotropic and, therefore, may not need to be removed. Brown packing tapes with clear film backings and colored adhesive must have the adhesive removed.

6.7.2.3.5 Tape may be manipulated with forceps and may be dissected using a fine scalpel blade to allow the examiner to thoroughly characterize the physical properties.

- 6.7.2.3.6 An appropriate solvent may be used to separate the adhesive from the backing or fiber reinforcement (e.g., hexane for duct tape, methanol for black electrical tape).
- 6.7.2.3.7 Following removal of the adhesive, duct tapes should be cross-sectioned to determine layer structure, the known and questioned compared and observations recorded.
- 6.7.2.3.8 If any meaningful differences are observed between the questioned and known tapes, the examiner can discontinue the analysis.

6.7.3 Macroscopic/Microscopic Examination of Adhesives

6.7.3.1 Purpose

To examine submitted adhesive evidence when no associated tape backing is present on a macroscopic and microscopic scale, noting physical properties, generally as the first step in the characterization and comparison of known and questioned adhesive samples.

6.7.3.2 Minimum Standards and Controls

- 6.7.3.2.1 Clean tools and work areas are a must.

6.7.3.3 Analytical Procedures

- 6.7.3.3.1 Thoroughly clean the examination area prior to the evidence being retrieved.
- 6.7.3.3.2 The sample is examined visually and with a stereomicroscope and the physical properties of each adhesive sample are noted.
- 6.7.3.3.3 Determine the solubility of the adhesive material in at least hexane, toluene and methanol.
- 6.7.3.3.4 The sample is examined with a compound microscope and observations recorded.
- 6.7.3.3.5 If any meaningful differences are observed between the questioned and known adhesives, the examiner can discontinue the analysis.

6.7.4 Fluorescence

6.7.4.1 Safety Considerations

Do not look directly into the fluorescence source.

6.7.4.2 Analytical Procedure

- 6.7.4.2.1 Some tape backings and adhesives fluoresce when exposed to different wavelengths of light. Of those tapes and adhesives which fluoresce, the observed color and approximate intensity of emission under different excitation wavelengths are noted. Fluorescence can be caused by dyes, pigments, chemical structures, other additives or impurities.

6.7.4.2.1.1 Fluorescence QC procedure:

- 6.7.4.2.1.1.1 Perform fluorescence QC check prior to using the fluorescence microscope for casework. Use

the fluorescence QC check sample and record observations on the fluorescence worksheet. If the fluorescence QC check fails to produce acceptable colors or intensity, troubleshoot the fluorescence microscope as appropriate and repeat the QC check. If the QC check continues to fail, take the fluorescence microscope out of service until the problem is resolved.

6.7.4.2.1.1.2 Fluorescence cubes to be used are WU (wide UV – range 330 – 385nm), WBV (wide blue violet – range 400 – 440nm), WB (wide blue – range 450 – 480nm) and WG (wide green – range 510 – 550nm). Record observations on a fluorescence worksheet.

6.7.4.2.2 Tape backings and adhesives may be examined in cross-section view or separately, however, questioned and known samples must be examined simultaneously; side-by-side using the stand-alone polarized light microscope with the room lights off.

6.7.4.2.3 Caution should be taken when eliminating samples based upon fluorescence data alone. Detailed notes explaining the basis for the elimination are a must. Eliminations based upon fluorescence will be reported as “due to differences in fluorescence”.

6.7.5 Instrumental Analysis

6.7.5.1 FTIR

6.7.5.1.1 Safety Considerations

6.7.5.1.1.1 Do not look directly into the laser source.

6.7.5.1.1.2 The MCT detector for the microscope accessory must be cooled with liquid nitrogen. Insulated gloves and safety glasses shall be worn when filling the transport Dewar as well as the instrument reservoir.

6.7.5.1.2 Minimum Standards and Controls

6.7.5.1.2.1 Ensure that the QC has been performed.

6.7.5.1.2.2 Replicate analyses may be employed to ensure reproducibility or to demonstrate variability within a sample.

6.7.5.1.3 Analytical Procedures

6.7.5.1.3.1 Remove the adhesive from the backing. Obtain spectra from both sides of the known and questioned tape backings by using the ATR. When more than one layer is indicated by cross-sections and/or ATR, obtain spectra from each layer of a flattened cross-section using the microcompression cell with diamond windows, when possible.

- 6.7.5.1.3.1.1 If a layer is too thin to isolate, obtain data for this layer via a composite with an adjacent layer.
- 6.7.5.1.3.2 Obtain spectra of the known and questioned adhesives/tape adhesives by using either the ATR or the microcompression cell with diamond windows.
- 6.7.5.1.3.3 The spectra for the known and questioned layers are compared. If any meaningful differences are observed in the spectra of the questioned and known sample, the examiner can discontinue the analysis.
- 6.7.5.2 SEM-EDS
- 6.7.5.2.1 If known and questioned tape backings and/or adhesives are generally in good condition, they will be referred for elemental composition comparison.
- 6.7.5.2.2 Both sides of the backing, when more than one layer is indicated, and/or adhesive are mounted on the carbon adhesive tape which is affixed to the aluminum stub.
- 6.7.5.2.3 A representative X-ray spectrum is collected of each sample (K and Q). A hard copy is printed to document the elemental composition.
- 6.7.5.2.4 Comparison of K and Q samples is made on a qualitative basis.
- 6.7.5.3 XRD
- 6.7.5.3.1 This instrumental technique is generally applicable for duct tape comparisons and manufacturer identification of duct tapes. It may also be useful for the comparison of some other types of tapes such as those with adhesives containing fillers (e.g., masking tape).
- 6.7.5.3.1.3 Duct tape samples that are generally in good condition will be referred for XRD analysis.
- 6.7.5.3.1.4 Other tapes with adhesives containing fillers will also be referred.
- 6.7.5.3.1.5 The examiner should consider the use of this technique with adhesives not associated with tapes as well.
- 6.7.5.3.2 Results are included with the Instrument Support Request form.
- 6.7.5.4 References
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- 6.7.5.4.4 “Forensic Characterization of Black Polyvinylchloride Electrical Tape”, Keto, R.O., *Crime Laboratory Digest*, Vol 11 (4), 1984, pp. 71-74.
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- 6.7.5.4.9 “The Forensic Value of Duct Tape Comparisons”, Smith J., *Midwestern Association of Forensic Scientists Newsletter*, Vol 27 (1), 1998, pp. 28-33.
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- 6.7.5.4.12 “The Examination of Pressure Sensitive Adhesive Tapes”, Johnston J. and Serra J., (International Association for Microanalysis) *IAMA Newsletter*, Vol. 5(1), 2005, pp. 19-31.
- 6.7.5.4.13 “PLM Examinations of Clear Polymer Films: Identification of monoaxial and biaxial orientation and other observations”, Smith, J.M. and Weaver, R., *Microscope*, Vol. 52(3/4), 2004, pp. 112-118.
- 6.7.5.4.14 “A New Approach for the Analysis of Duct Tape Backings”, Hobbs, A.L., Gauntt, J., Keagy, R., Lowe, P.C., Ward, D., *Forensic Science Communications*, Vol. 9(1), 2007.
- 6.7.5.5 Documentation
- 6.7.5.5.1 As a minimum, each case file will include individual case notes, all worksheets and hard copies of all spectra generated during the analysis and conditions sheets for instrumental analyses conducted, as applicable.
- 6.7.5.6 Report Wording
- 6.7.5.6.1 If the known and questioned tapes/adhesives can be eliminated based upon any of the testing the report will generally read:
- Item ____ could not be associated with Item ____ due to differences in _____. (e.g., color, physical properties, manufacturing characteristics of the tape backing, chemical properties)
- 6.7.5.6.2 If the known and questioned tapes/adhesives cannot be eliminated based upon any of the testing the report will generally read:

6.7.5.6.2.3 The Item ___ duct tape could not be physically fitted to the Item ___ roll of tape (known). The Item ___ duct tape was consistent with the Item ___ (known) in overall construction and in physical and chemical properties. It was concluded that the Item ___ duct tape could have originated from Item ___ or another source of tape with the same overall construction, physical and chemical properties.

6.7.5.6.2.4 The black electrical tape present on Item ___ was consistent with the Item ___ tape (known) in overall construction and in physical and chemical properties. It was concluded that the Item ___ tape could have originated from Item ___ (known) or another source of tape with the same overall construction, physical and chemical properties.

6.7.5.6.2.5 The Item _____ adhesive recovered from the mouth area of the victim was consistent in physical and chemical properties with the adhesive in Item _____ (known). It was concluded that the Item _____ recovered adhesive could have had a common origin with Item _____ (known) or another adhesive with the same physical and chemical properties.

6.7.5.6.2.6 ...or from a roll manufactured in the same manner.

6.7.5.6.3 Techniques used in the analysis shall be identified in the body of the report. See ¶ 1.9.2 for example wording.

6.7.5.6.4 If foreign fibers were recovered and knowns are being requested for comparison purposes see Section 3.

6.8 Tear Gas and Pepper Sprays

6.8.1 Purpose

To identify tear gas products that may be encountered in casework: 2-chloroacetophenone (CN), o-chlorobenzylidenemalononitrile (CS) and oleoresin capsicum (OC), which contains the active component, capsaicin.

6.8.2 Safety Considerations

6.8.2.1 Tear gas products are irritants, by definition, and will cause physical discomfort if inhaled. If working with spray products or highly concentrated clothing items, perform analysis in a fume hood.

6.8.2.2 Avoid contact with skin and eyes.

6.8.3 Minimum Standards and Controls

6.8.3.1 Treat the questioned samples and any submitted control/standard samples in the same manner.

6.8.3.2 When performing extractions also analyze a blank of the extraction liquid and an unstained portion of the substrate, if possible. If an unstained portion of the substrate is not possible, analyze the extraction solvent as the negative control.

6.8.3.3 When identifying a tear gas component, retention time data for samples and standards are needed from at least the polar or semi-polar GC or GC-MS column, along with the retention time and spectral data from the GC-MS.

6.8.4 Analytical Procedures

6.8.4.1 Extraction of liquids (spray products)

6.8.4.1.1 Weigh the canister to obtain the gross weight as received.

6.8.4.1.1.1 CS and CN

6.8.4.1.1.1.1 Oil based - Spray approximately 1 ml of the liquid into a vial and add about 1 ml of methanol. Analyze by GC and GC-MS. Concentrate by evaporation or dilute with methanol as needed.

6.8.4.1.1.1.2 Alcohol based - Spray approximately 5 ml, if available, into a small beaker and gently evaporate to several drops. The residue is analyzed by GC and GC-MS.

6.8.4.1.1.2 Capsaicin

6.8.4.1.1.2.1 Spray approximately 1 ml of the liquid into a vial and add about 1 ml of methanol. Analyze by GC and GC-MS. Concentrate by evaporation or dilute with methanol as needed.

6.8.4.2 Extraction of clothing items

6.8.4.2.1 View the evidence item(s) under ultraviolet light or by using an alternative light source. Note any fluorescing areas, document as appropriate. If a known source canister is submitted also check the known liquid for fluorescence using ultraviolet light or an alternative light source.

6.8.4.2.1.1 Fluorescence QC procedure:

6.8.4.2.1.1.1 Perform fluorescence QC check prior to using UV source for casework using a suitable fluorescent highlighter. Place a mark on a glass slide using the highlighter to verify proper operation of the UV source and record resulting fluorescence in notes. The prepared slide can be stored out of direct light and can be used for future QC checks. If the QC check fails, troubleshoot equipment and repeat QC check. If the UV source fails to produce fluorescence on the prepared slide(s), take the UV source out of operation until suitable repairs are made.

6.8.4.2.2 If a stained area is visible on the clothing, or becomes viewable by the process described above, remove a portion of it by cutting with scissors or a new scalpel. Remove an equivalent sized portion of an unstained area for use as a control, if necessary.

6.8.4.2.3 If no stains are visible, remove portions of the garment from an area of the clothing where spray was reported to have been administered. Remove an equal sized portion of the clothing from an area of the garment farthest from the sampled area for use as a control, if necessary.

6.8.4.2.4 Extract the cuttings with methanol, using the smallest volume to facilitate complete wetting of the cuttings and subsequent recovery of the methanol. Analyze by GC and GC-MS. Concentrate sample or dilute with methanol as needed.

6.8.5 Report Wording

6.8.5.1 CS and CN:

6.8.5.1.1 The Item _____ canister (was operational and) contained _____. _____, commonly known as _____, is an irritant.

6.8.5.1.2 _____, commonly known as _____, was identified in the Item _____ extract.

6.8.5.1.3 Report CS as o-chlorobenzylidenemalononitrile (CS).

6.8.5.1.4 Report CN as 2-chloroacetophenone (CN).

6.8.5.2 OC:

6.8.5.2.1 Item _____ contained _____. This is consistent with the labeling on the capsicum-based pepper spray container.

6.8.5.2.2 Capsaicin (and dihydrocapsaicin) was (were) identified in the Item _____ extract. This (These) is a(are) component(s) of capsicum-based pepper sprays.

6.8.5.3 If no CS, CN or OC are identified:

6.8.5.3.1 No _____ were identified in the Item _____ extract.

6.8.5.4 Techniques used in the analysis shall be identified in the body of the report. See ¶ 1.9.2 for example wording.

6.8.6 References

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6.8.6.5 Mongan, A.L.; Buel, E. "Identification of Dog Repellent in the Clothes of an Assault Suspect Using Gas Chromatography/Mass Spectrometry"; *Journal of Forensic Sciences*, 1995, 40, 3, 513-514.

6.8.6.6 Gag, J.A.; Merck, N.F. "Concise Identifications of Commonly Encountered Tear Gases"; *Journal of Forensic Sciences*, 1977, 22, 22, 358-364.

6.8.6.7 Illinois State Police, Trace Chemistry Procedures Manual, February 1997.

6.9 Red Phosphorus, Lithium and Iodine

6.9.1 Purpose

To identify iodine, lithium, and red phosphorous that may be encountered in evidence which typically originates from clandestine methamphetamine laboratories.

6.9.2 Safety Considerations

6.9.2.1 Iodine, lithium, and red phosphorus are frequently encountered in reaction mixtures. Care should be taken when handling these samples.

6.9.2.2 Red phosphorus is a combustible solid, that, while stable, may be ignited by heat, friction, physical impact, static electrical sparks, or oxidizing reagents.

6.9.2.3 Red phosphorus should not be stored next to peroxides, hydroxides, halogens or nitric acid.

6.9.2.4 Care should be taken when performing an ignition test for red phosphorus. Only a small amount of sample should be used.

6.9.2.5 Lithium metal is highly reactive and reacts violently with water. Due to hazards associated with lithium metal, it shall not routinely be accepted for examination in the laboratory.

6.9.3 Minimum Standards and Controls

6.9.3.1 Extractions

6.9.3.1.1 Previously cleaned glassware should be rinsed with the extracting solvent.

6.9.3.1.2 A solvent blank shall be run at the same time as the sample extract. The same volume of solvent used for the sample extraction should be used for the blank.

6.9.3.1.3 If the sample extract is filtered or evaporated, the blank shall be handled in a like manner.

6.9.3.2 Analytical Procedures

6.9.3.2.1 Evaluate the submitted items to determine which items to analyze. When working clandestine methamphetamine laboratories, refer to the Code of Virginia, § 18.2-248(J) regarding precursor chemicals to assist in determining the best analytical scheme.

6.9.3.2.2 Characterize the sample using the appropriate techniques (e.g., stereomicroscopy, solubility, miscibility, weighing).

6.9.3.3 Red Phosphorus

6.9.3.3.1 Red phosphorus is the primary component in the striker plate on strike-on-box matches. The color of red phosphorous varies but is most often dark red, purple or purple-gray.

6.9.3.3.2 Ignition Test

6.9.3.3.2.1 Place a small portion of the sample on the frosted labeling surface of a glass slide, and strike a match across the slide.

Record the results in the case notes. Ignition of the match indicates the presence of red phosphorous.

6.9.3.3.2.1.1 A negative control of a match and igniting surface shall be performed prior to the ignition test.

6.9.3.3.2.2 If the match does not ignite, washing the questioned sample may produce a more purified sample that will ignite. Prior to washing, prepare an “as is” portion of the sample for SEM-EDS analysis. Chloroform, acetone, and methanol are possible solvents for washing the sample because red phosphorus is essentially insoluble in these solvents. Rinse the sample multiple times and retry the ignition test once the sample has dried. Record results in case notes. If appropriate, a portion of the washed sample may also be analyzed using SEM-EDS.

6.9.3.4 Iodine

6.9.3.4.1 Iodine is frequently encountered in casework as elemental iodine, stains on coffee filters or other materials, reaction mixtures, or products such as iodine tincture or povidone-iodine solution.

6.9.3.4.2 Elemental iodine is a shiny, dark purple to black nonmetallic crystalline solid that frequently stains whatever it comes in contact with a dark purple, dark orange, brown-yellow, yellow-orange, or a similar color. Elemental iodine readily sublimates to a vapor at room temperature.

6.9.3.4.3 Tincture of iodine is generally composed of an iodide salt (potassium or sodium) and elemental iodine dissolved in ethanol and water. To identify as tincture of iodine, the iodide salt and the ethanol need to be identified.

6.9.3.4.3.1 Physically and chemically consistent with wording may be used when ethanol is not identified but all of the other tests are performed.

6.9.3.4.4 Solubility test

6.9.3.4.4.1 Place a small portion of the solid sample or a portion of the stain separately in acetone and in chloroform. Record the solubility and any color change to the solvent in the case notes. A solution of elemental iodine in acetone will become yellow-orange to dark brown-orange in color. A solution of elemental iodine in chloroform will become purple-pink, pink-purple or violet in color. Stains or reaction mixture solids in solution with chloroform may become yellow to yellow-orange in color.

6.9.3.4.5 Extractions

6.9.3.4.5.1 Samples such as stained coffee filters should be extracted using a solvent unless recoverable material is present. The solvent extract of the sample and the solvent blank shall both be dried down and analyzed using SEM-EDS.

6.9.3.5 Lithium

6.9.3.5.1 Lithium is frequently encountered in casework as lithium salts in reaction products.

- 6.9.3.5.2 Physical characteristics of the sample shall be noted. Proceed to instrumental analysis section.
- 6.9.3.6 Instrumental analysis
- 6.9.3.6.1 For further characterization of the sample, use SEM-EDS, XRD, GC-MS, IC, LIBS, or other technique, as appropriate.
- 6.9.3.6.2 When solid material is present in the sample (excluding neat samples of elemental iodine and reaction products containing lithium), a portion of the “as is” solid should be analyzed using SEM-EDS because many solids encountered in clandestine methamphetamine laboratories are mixtures. If appropriate, a portion of a washed sample may be analyzed using SEM-EDS.
- 6.9.3.6.3 For liquid samples, residues from an evaporated portion of the sample should be analyzed using SEM-EDS.
- 6.9.3.6.4 When extractions are necessary, a portion of the dried solvent extract of the sample and a portion of the dried solvent negative control, or blank, should be analyzed using SEM-EDS.
- 6.9.3.6.5 Red phosphorus shall be identified using SEM-EDS.
- 6.9.3.6.6 Elemental iodine can be identified as iodine using XRD. When sending for instrument support, it is important to indicate that elemental iodine is suspected. Due to the nature of iodine sublimation, a sufficient amount of sample should be sent.
- 6.9.3.6.7 Tincture of iodine may be analyzed using XRD, GC-MS and GC.
- 6.9.3.6.7.1 If needed, confirm the presence of ethanol using GC-MS and GC.
- 6.9.3.6.7.2 Evaporate a portion of the sample (tincture solution) in a watch glass or evaporating dish. As the solvent evaporates, iodide salt will crystallize. Residual iodine may remain in liquid form in the evaporated sample. Acetone and chloroform solubility (with color change observations) can be conducted on a portion of the evaporated sample. The presence of elemental iodine in the evaporated sample is indicated by an orange color in acetone and a violet color in chloroform. The iodide salt is soluble in acetone but is insoluble in chloroform.
- 6.9.3.6.8 For suspected lithium salts, a representative portion of the sample is dissolved in DI water and prepared for IC analysis.
- 6.9.3.6.8.1 If a peak consistent with lithium is present, it shall be confirmed with LIBS.
- 6.9.3.6.8.2 If there are no peaks consistent with lithium, no further analyses are required.

6.9.3.7 Report Wording

6.9.3.7.1 If red phosphorus is present (color and positive ignition test):

Item ___ contained red phosphorus, a listed substance in § 18.2-248(J).

6.9.3.7.2 If phosphorus is present but the ignition test was negative:

Item ___ contained phosphorus.

6.9.3.7.3 If elemental iodine is present:

Item ___ contained iodine, a listed substance in § 18.2-248(J).

6.9.3.7.4 For tincture of iodine samples, if the iodide salt and ethanol have been identified:

Item ___ contained tincture of iodine, a listed substance in § 18.2-248(J).

6.9.3.7.5 For tincture of iodine samples, if the iodide salt has been identified but ethanol has not:

The liquid in Item ___ was physically and chemically consistent with tincture of iodine, a listed substance in § 18.2-248 (J).

6.9.3.7.6 If iodine is present, but the original source cannot be determined:

Item ___ contained iodine, a listed substance in § 18.2-248(J).

The Item ___ extract contained iodine, a listed substance in § 18.2-248(J).

6.9.3.7.7 If a peak consistent with lithium is present in the IC results and lithium is identified by LIBS:

Item ___ contained lithium, a listed substance in § 18.2-248(J).

6.9.3.7.8 If a peak consistent with lithium is present in the IC results and lithium is not identified by LIBS (i.e., less than 3 lithium peaks or negative):

No lithium was identified in Item _____.

6.9.3.7.9 If no red phosphorus, iodine, or lithium is present:

No (red phosphorus, iodine, lithium) was identified in Item _____.

6.9.3.7.10 If all substances listed in § 18.2-248(J) can be eliminated by physical properties and/or chemical testing use:

No substances listed in § 18.2-248(J) were identified in Item _____.

6.9.3.8 Techniques used in the analysis shall be identified in the body of the report. See ¶ 1.9.2 for example wording.

6.9.4 References

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6.9.4.2 The United States Pharmacopeia, Nineteenth Revision, United States Pharmacopeial Convention Inc., Maryland, 1975, pgs. 258-259.

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7 GLASS**7.1 Analytical Approach**

- 7.1.1 Forensic glass examinations usually involve the comparison of a questioned glass sample with a known sample from a broken glass source. The glass samples are analyzed by determining physical properties and refractive index for the known and questioned glass samples. Glass that is macroscopic will be referred to as glass fragments, and glass that is microscopic will be referred to as glass particles. Particle size sometimes precludes the determination of some or all of a glass particle's physical properties. In this situation, physical properties are determined to the extent possible, and conclusions are drawn based on those properties that can be determined.
- 7.1.2 Glass comparison examinations should be considered for fracture match examination, if feasible. Fracture match (or physical fit) provides the only conclusive association between glass samples.
- 7.1.3 Another examination that may be requested is glass identification. A request may be that a submitted sample be identified as glass, or it may be to identify the type of glass in a sample. Physical properties and/or refractive index measurements are determined to the extent necessary to reach a conclusion.
- 7.1.4 Direction-of-impact or sequence-of-impact determinations may be requested for a submitted glass sample. These determinations can only be made from non-tempered glass sources.
- 7.1.4.1 For direction-of-impact, the broken pane is reconstructed and, if possible, the point-of-impact and cracks radiating from that point are identified to determine the direction-of-impact.
- 7.1.4.2 For sequence-of-impact, the glass pane must hold together in order to retain the pattern. Typically, only laminated or wire reinforced panes will retain a pattern suitable for examination. The investigator must collect and preserve as much of the window pane as possible for this determination.
- 7.1.5 Glass sources sometimes have other materials such as paint, decals, or tint films associated with them. These materials may be transferred with glass particles and may increase the evidentiary value of the glass. Comparisons of glass samples such as this should also include comparison of these associated materials. These examinations will be performed in accordance with the applicable Section protocols.

7.2 Recovery of Glass Particles from Clothing and Objects

- 7.2.1 Purpose
- This procedure is necessary to isolate glass particles for characterization and subsequent comparison with known glass samples.
- 7.2.2 Minimum Standards and Controls
- 7.2.2.1 All glassware is cleaned prior to use.
- 7.2.2.2 Processing table/area must be cleaned before and after each use.
- 7.2.2.3 Minute glass particles recovered from debris must be clean in order to perform testing procedures.
- 7.2.2.4 Known glass samples are isolated from items being processed to prevent contamination.
- 7.2.3 Analytical Procedures
- 7.2.3.1 Lay out item(s) to be examined on new, clean paper. Shake and/or scrape the inside of the package over the paper.

- 7.2.3.2 Describe each item to be processed for glass particles. For clothing items, note general type, color(s), patterns and label information, including size and manufacturer. Note the overall appearance of each item (new, worn, clean, soiled, apparent blood stains, fabric separations, etc.).
- 7.2.3.3 For objects such as tools or weapons, note type, color, manufacturer, size and appearance as appropriate. Objects may exhibit scratches, gouges or other damage, which should be noted.
- 7.2.3.4 Using a spatula, scrape clothing item(s) over paper. Scrape inside pockets and cuffs, if present. Personal items (from pockets) should be listed on the examination documentation and retained with originating item. Clothing items from a single individual, with the exception of shoes, may be processed together to yield combined debris from a set of items. Shoes must be processed separate from other clothing, due to the possibility that extraneous glass from the environment may be present. If right and left shoes from a single individual are submitted as separate items, these may be processed together to yield combined debris from the items. It may be necessary to use a small spatula or probe to remove debris from the soles of shoes.

If clothing and shoes are submitted from a particular suspect, the clothing items are considered to be the best evidence for glass examination. If glass particles are recovered from the clothing items and subsequently associated with the known glass source, the shoes may not be necessary for examination. This is determined on a case-by-case basis.

Rigid objects such as tools and weapons are processed by the most appropriate means, depending on the item. Brushing an item over paper using new, clean, disposable paint brushes usually yields the best results. If particles are embedded in an object, probing in gouges may be the most effective means of removal.

- 7.2.3.5 Collect debris and retain in a labeled container (plastic container, evidence fold, etc.). If apparent glass particles are seen visually they may be retained in a separate container.
- 7.2.3.6 Thoroughly examine the recovered debris for glass particles using a stereomicroscope (usually 30X-40X magnification). Describe the overall make-up of the debris. Remove apparent glass particles based on clarity, irregular shape, hardness, freshness of edges, and conchoidal fracture. Place the recovered particles in a labeled depression slide. Recover at least ten (10) particles, if that many can be found. If two or more distinguishable glass sources were broken in the incident and submitted for analysis, the examiner shall attempt to recover additional particles from the collected debris, if possible. The general rule is ten particles per separate known glass sample.
- 7.2.3.7 In a dry mount in the depression slide, examine the recovered particles using a polarized light microscope or stereomicroscope with transmitted illumination and a polarizing attachment. Anisotropic particles, those which exhibit birefringence using crossed polars, are eliminated as glass particles. Particles that exhibit no interference colors using crossed polars are isotropic particles. Isotropic particles having the physical properties indicated above are therefore identified as glass. If the recovered particles are isotropic, indicate in the case notes that the particles were examined using crossed polars and were isotropic.
- 7.2.3.8 Glass fragments and/or particles are cleaned prior to further analysis, as necessary. When glass fragment and/or particles are cleaned, it shall be reflected in the case notes. Select one of the two following methods for cleaning:
- 7.2.3.8.1 Glass particles are transferred to a small labeled beaker (such as the 50 ml size) and 10-20 ml of 5% micro detergent solution is added. The beaker is placed in an ultrasonic cleaner and set to clean for 10 minutes. The particles are washed into a coned filter paper in a funnel with deionized water and rinsed well. The clean glass particles are recovered from the filter paper for comparison to known samples.

7.2.3.8.2 Glass particles are washed directly in the depression slide in a 5% micro detergent solution in an ultrasonic cleaner. The depression slide is placed in a 100mm diameter Pyrex Petri dish. A small amount of deionized water is placed in the Petri dish and in the bottom of the ultrasonic cleaner which is set to clean for 5 minutes. The particles are then transferred to the second depression well on the slide and rinsed in deionized water by 5 minute sonication. The clean glass particles are recovered from the depression slide for comparison to known samples.

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- 7.2.4.6 Collection, Handling, and Identification of Glass, Scientific Working Group for Materials Analysis, Forensic Science Communications, Volume 7, No. 1, January 2005.
- 7.2.4.7 Hamer, Pamela, "Microscopic techniques for glass examination", in Forensic Examination of Glass and Paint, Analysis and Interpretation; Brian Caddy, ed, Taylor and Francis: London and New York, 2001, Ch. 3, Sec 3.1 – 3.5, pp 47-55.
- 7.2.4.8 Thornton, John I., "Interpretation of physical aspects of glass evidence", in Forensic Examination of Glass and Paint, Analysis and Interpretation; Brian Caddy, ed, Taylor and Francis: London and New York, 2001, Ch. 6, Sec. 6.7, p. 114 and Sec. 6.9 pp 116-118.

7.3 Physical Properties

7.3.1 Purpose

This procedure is used to characterize the physical properties of a glass sample or an individual glass fragment for comparison of these properties with other samples or fragments.

7.3.2 Safety Considerations

- 7.3.2.1 When using the UV lamp, safety glasses that provide UV protection should be worn.
- 7.3.2.2 Extreme care should be taken to prevent lacerations from glass samples, especially when working with laminated glass samples.

7.3.3 Minimum Standards and Controls

- 7.3.3.1 Work area must be cleaned before and after each use.

7.3.4 Analytical Procedures

- 7.3.4.1 Glass comparison examinations should be considered for a fracture match examination, when appropriate.
- 7.3.4.1.1 Fracture match (or physical fit) provides the only conclusive association between glass samples.
- 7.3.4.2 Prepare a glass worksheet and record visual observations. These observations include, but are not limited to, color, type, thickness, temper, float properties, shape, inclusions, surface texture and surface markings. Record the amount of glass received. As applicable, this may be accomplished by indicating either the number of fragments/particles/dices of an approximate size (1 fragment ~1 ¼" x 3", 6 fragments ~ 1/2") or by indicating a general description as to amount present (e.g., ~ 1 tablespoon of glass fragments, ~ ¼ cup of glass fragments). Examine a sufficient number of fragments in the sample to determine if multiple broken glass sources may be present in the sample.
- 7.3.4.2.1 Laminated glass, if submitted in its complete form, should be sampled from both layers and treated as two separate samples.
- 7.3.4.3 If two parallel manufactured surfaces are present, measure the thickness of the glass sample using a digital caliper. Take a representative number of measurements to determine if a thickness range exists. The measurements, to include maximum and minimum measurements, shall be recorded on the worksheet to the one hundredth of a millimeter. The digital caliper used will be noted on the glass worksheet.
- 7.3.4.3.1 Float glass samples with thicknesses that differ by greater than 0.15 mm may be eliminated as having originated from the same source. The questioned glass sample is not eliminated if it is within the observed thickness range of the known sample.
- 7.3.4.4 Observe the fractured surfaces of a glass sample for indications of temper. Tempered glass sources typically "dice" when broken, forming approximate cube shaped fragments. The broken surfaces of these fragments are generally perpendicular to the manufactured surfaces. Other characteristics of tempered glass may include symmetrical, or mirror-imaged, ridges on the broken surfaces, or the presence of a "frost line", which generally divides the symmetrical ridges in the broken surfaces. For samples of limited size, examination of the fragments with a polarized light microscope (PLM) using crossed polarized light may be useful to determine temper. Tempered glass exhibits internal stresses that can be observed as interference colors that appear to move through the fragment as the PLM stage is rotated. Based on the presence or absence of these observations, record the sample as "tempered" or "non-tempered" on the glass worksheet.
- 7.3.4.5 Before examining the fluorescence properties of the known glass, perform the handheld UV light source QC check. In a darkened room, examine flat manufactured surfaces using short and long wavelength UV. If a flat glass sample exhibits a marked fluorescence on one side and not on the other while being examined using short wavelength UV, it is determined to be a float glass. Glass samples may also exhibit an overall fluorescence rather than only on the float surfaces. Record fluorescence observations on the worksheet for both long and short wavelengths, including whether the glass sample is float or non-float.
- 7.3.4.6 If the glass sample has a highly reflective but transparent manufactured surface, determine if the glass is from a low emissivity (low-E) source. On the multimeter, select the ohm setting for resistance. Check the multimeter prior to use by touching the probes together to ensure continuity. Record this check on the glass worksheet. Then press the two probes of the multimeter against the glass surface without allowing them to touch each other. If the surface conducts electrical current, the sample is a low-E glass. Low-E glass conducts electrical

current on one manufactured surface but not the other. The multimeter used shall be noted on the glass worksheet.

- 7.3.4.7 If the questioned and known glass samples being compared cannot be distinguished by the physical properties discussed above, the refractive indices of the samples are compared (See Section 7.4).

7.3.5 References

- 7.3.5.1 Koons, Robert D., Buscaglia, JoAnn, Bottrell, Maureen, Miller, E.T. "Forensic Glass Comparisons", in Forensic Science Handbook, Volume 1, Second Edition; R. Saferstein, ed, Pearson Education, Inc.: Upper Saddle River, NJ, 2002, pp. 179-186.
- 7.3.5.2 Nelson, D.F. "The Examination of Glass Fragments" in Methods of Forensic Science; A.S. Curry, ed, Interscience Publishers:London, 1965.
- 7.3.5.3 Collection, Handling, and Identification of Glass, Scientific Working Group for Materials Analysis, *Forensic Science Communications*, Volume 7, No. 1, January 2005.
- 7.3.5.4 Initial Examinations of Glass, Scientific Working Group for Materials Analysis, *Forensic Science Communications*, Volume 7, No. 1, January 2005.
- 7.3.5.5 Dorn, H., Ruddell, D., Heydon, A., Stefanescu, A., Stryjnik, A., Burton, B. D., "Unusual Variation of Thickness Within a Pane of Annealed Float Glass", *Can. Soc. Forensic Sci. J.* Vol. 46, No. 3 (2013), pp. 166-69.

7.4 Refractive Index (RI) Measurement by GRIM3 System

7.4.1 Purpose

This procedure is used to determine the refractive index of a glass sample or individual glass fragments/particles for comparison with other samples or fragments/particles.

7.4.2 Minimum Standards and Controls

- 7.4.2.1 Steel pulverizer and work area must be cleaned before and after each use.
- 7.4.2.2 Annual calibration, a monthly QA check, a day-of-use QA check and a case QA check are required.
- 7.4.2.3 Questioned glass particles recovered from debris from clothing or objects shall be cleaned prior to mount preparation to obtain the most suitable data.

7.4.3 Analytical Procedures

- 7.4.3.1 Start the GRIM3 system and allow it to stabilize for one hour prior to taking measurements.
- 7.4.3.1.1 Open the GRIM3 software and create a data file for the FS case. Select the oil and wavelength being used for the measurement.
- 7.4.3.1.2 The prepared mount is inserted into the hot stage on the phase contrast microscope. The bulk of the particles are located and the microscope is focused on the glass particles.
- 7.4.3.1.3 Using the phase telescope in place of one of the microscope oculars, phase contrast is adjusted by viewing and aligning the phase rings. This is accomplished by pushing in and turning the adjustment knobs for the phase

annulus, which is located on the microscope's condenser assembly. The phase contrast should be checked and adjusted for each prepared mount.

- 7.4.3.1.4 Prepare a mount of fine, crushed glass particles using the appropriate immersion oil. Locke Silicone Oil B is used for most window and container glasses. Locke Silicone Oil C is used with headlight glasses. Locke Silicone Oil A is used for samples with high refractive index that are above the refractive index range of the Locke Silicone Oil B. A cover slip is placed over the oil. The slide is marked for identification purposes.
- 7.4.3.2 The known glass sample is prepared for refractive index determination. The fragment or a portion of the fragment is placed in a pulverizer and crushed.
- 7.4.3.2.1 When five or more known glass fragments are present in a sample, prepare crushed mountings from at least five fragments. Select particles to measure which exhibit edges with the highest degree of contrast. Obtain RI measurements on a minimum of two particles for each crushed mounting for a total of ten measurements per known sample.
- 7.4.3.2.1.1 For samples that exhibit a wide within-sample variation such as tempered glass or other non-annealed glass sources, obtain RI measurements on a minimum of four particles for each crushed mounting for a total of 20 measurements per known sample.
- 7.4.3.2.2 When four or fewer known glass fragments are present in a sample, prepare crushed mountings from each of the fragments. Obtain RI measurements for a total of 10 or 20 particles as noted in ¶¶ 7.4.3.2.1 and 7.4.3.2.1.1. Sample size and examiner discretion will determine how many areas of a fragment will be sampled. Multiple samplings from a single large glass fragment may be necessary.
- 7.4.3.2.3 The case file notes will clearly document how the known glass was sampled.
- 7.4.3.3 Using the appropriate oil, mounts are prepared for questioned macroscopic glass fragments or microscopic recovered glass particles.
- 7.4.3.3.1 For questioned glass fragments, prepare mounts and perform measurements as described for known glass fragments above.
- 7.4.3.3.1.1 A single questioned glass fragment association requires a minimum of four measurements.
- 7.4.3.3.1.2 In the case of a questioned glass sample where the sample is assumed to be homogeneous and the difference in mean refractive index with the known glass is greater than ± 0.0003 , only four measurements are necessary.
- 7.4.3.3.2 For questioned glass particles, perform a measurement on each particle, selecting an edge on the particle which exhibits the highest degree of contrast.
- 7.4.3.3.3 The degree of contrast on the GRIM3 system can be assessed by the "Edge Contrast" value assigned to each measured value. This value is shown in parentheses next to the match temperature measurement. The best value is "99", with the value decreasing as edge contrast quality decreases. Glass particles with an edge contrast value of less than "10", in either the cooling or heating measurement cycles, will not be considered as suitable for comparison purposes. Such glass particles must be saved as "Reject" particles and will not be

associated with a comparison sample regardless of the measured value. The particle may be re-measured on a different edge to obtain an acceptable edge contrast value.

7.4.3.3.4 Multiple individual questioned particles may be mounted together on a single slide. If this is done, rough sketching of the mount is necessary for reference while collecting data and shall be retained with the case file documentation.

7.4.3.3.5 When a single questioned particle is too large to mount without crushing but is too small for determination of physical properties, only one RI measurement is necessary.

7.4.3.4 The refractive index measurement for questioned glass fragments/particles will be compared to the observed refractive index range for the known glass sample(s).

7.4.3.4.1 Questioned glass particles with refractive index values that are within the observed RI range of the known glass sample(s) are considered to be consistent in refractive index.

7.4.3.4.2 Questioned glass fragments with an observed RI range that overlaps with the observed RI range of the known glass sample(s) are considered to be consistent in refractive index.

7.4.3.5 Following the examination, individual questioned glass particles are recovered and retained with the evidence.

7.4.3.5.1 The case file notes will record how the evidence is packaged for return.

7.4.4 References

7.4.4.1 GRIM3 "User Manual 08", September 2006.

7.4.4.2 Locke Scientific "Reference Glasses and Silicone Oils for Refractive Index Determination".

7.4.4.3 Locke, J.; Underhill, M. "Automatic Refractive Index Measurement of Glass Particles"; Forensic Science International, 1985, 27, pp. 247-260.

7.4.4.4 Satterthwaite, M.J.; Harrison, P.H.; Lambert, J.A.; "Glass Refractive Index - Recent Developments in Measurement and Data Handling in UK Forensic Science Service Laboratories", Forensic Science Service, Wetherby Laboratory, West Yorkshire, England.

7.4.4.5 Koons, Robert D., Buscaglia, JoAnn, Bottrell, Maureen, Miller, E.T. "Forensic Glass Comparisons", in Forensic Science Handbook, Volume 1, Second Edition; R. Saferstein, ed, Pearson Education, Inc.: Upper Saddle River, NJ, 2002, pp. 186-195.

7.4.4.6 Glass Refractive Index Determination, Scientific Working Group for Materials Analysis, *Forensic Science Communications*, Volume 7, No. 1, January 2005.

7.4.4.7 Sandercock, P.M.L. "Sample Size Consideration For Control Glass in Casework", Canadian Journal of Forensic Science, Volume 33, No. 4, 2000, pp. 173-185.

7.4.4.8 Hamer, Pamela, "Microscopic techniques for glass examination", in Forensic Examination of Glass and Paint, Analysis and Interpretation; Brian Caddy, ed, Taylor and Francis: London and New York, 2001, Ch. 3, Sec. 3.6, pp 56-64.

7.5 Direction-of-Impact

7.5.1 Purpose

This procedure is used to determine from which side a window pane was broken.

7.5.2 Minimum Standards and Controls

7.5.2.1 The submitted glass fragments taken directly from the window frame must be properly labeled as to inside/outside orientation. This should be accomplished by marking the fragments still in the frame with the correct orientation at the time of collection by the investigator.

7.5.2.2 A sufficient amount of the total glass fragments from the broken pane must be submitted in order to reconstruct the pane and subsequently make an appropriate conclusion with regard to direction-of-impact. This should be accomplished by collecting and submitting all of the larger pieces, including those from the floor, those from the ground and those found in the frame.

7.5.2.3 The broken glass source must be non-tempered. This determination cannot be made on a tempered glass source.

7.5.3 Analytical Procedures

7.5.3.1 Lay out broken pane fragments on paper.

7.5.3.2 Lay fragments in a consistent surface orientation based on float side fluorescence, paint or surface debris if possible.

7.5.3.3 Reconstruct the pane as completely as possible.

7.5.3.4 Determine point(s)-of-impact and attempt to identify radial cracks.

7.5.3.5 If radial cracks can be identified, examine ridges on these cracks. These ridges are perpendicular to the surface opposite the side-of-impact. 4-R Rule: "Ridges on Radial cracks are at Right angles to the Rear."

7.5.3.6 Using the correct inside or outside orientation for the broken pane provided by the investigator, determine the direction-of-impact.

7.5.4 References

7.5.4.1 Koons, Robert D., Buscaglia, JoAnn, Bottrell, Maureen, Miller, E.T. "Forensic Glass Comparisons", in *Forensic Science Handbook*, Volume 1, Second Edition; R. Saferstein, ed, Pearson Education, Inc.: Upper Saddle River, NJ, 2002, pp. 173-176.

7.5.4.2 Nelson, D.F. "The Examination of Glass Fragments" in *Methods of Forensic Science*; A.S. Curry, ed, Interscience Publishers:London, 1965.

7.5.4.3 Glass Fractures, Scientific Working Group for Materials Analysis, *Forensic Science Communications*, Volume 7, No. 1, January 2005.

7.5.4.4 Thornton, John I., "Interpretation of physical aspects of glass evidence", in *Forensic Examination of Glass and Paint, Analysis and Interpretation*; Brian Caddy, ed, Taylor and Francis: London and New York, 2001, Ch. 6, Sec. 6.1 -6.6 pp. 97-114.

7.6 Sequence-of-Impact

7.6.1 Purpose

This procedure is used to determine the sequence in which multiple impacts have occurred in a broken glass pane.

7.6.2 Minimum Standards and Controls

7.6.2.1 This determination can only be made on non-tempered glass sources. The pane must also hold together long enough for the pattern to develop. Typically, only laminated or wire reinforced panes will do so.

7.6.3 Analytical Procedures

7.6.3.1 Determine points-of-impact and identify radial cracks.

7.6.3.2 If multiple points-of-impact can be identified, examine the cracks formed by each impact and attempt to determine the relationship between these impacts. Cracks formed by a later impact will terminate at cracks already formed by an earlier impact.

7.6.3.3 Using these observations, determine the sequence of these impacts.

7.6.4 References

7.6.4.1 Koons, Robert D., Buscaglia, JoAnn, Bottrell, Maureen, Miller, E.T. "Forensic Glass Comparisons", in Forensic Science Handbook, Volume 1, Second Edition; R. Saferstein, ed, Pearson Education, Inc.: Upper Saddle River, NJ, 2002, pp. 176-177.

7.6.4.2 Nelson, D.F. "The Examination of Glass Fragments" in Methods of Forensic Science; A.S. Curry, ed, Interscience Publishers:London, 1965.

7.6.4.3 Glass Fractures, Scientific Working Group for Materials Analysis, *Forensic Science Communications*, Volume 7, No. 1, January 2005.

7.7 Documentation

7.7.1 For glass comparison or identification examinations, all physical properties determined for each item will be listed on a glass worksheet.

7.7.2 When refractive index values are determined by the GRIM3 system, a printed copy of the GRIM3 data and a "GRIM3 Instrumentation" worksheet, which lists the equipment and calibration oils used, will be included in each case file.

7.7.3 For direction-of-impact or sequence-of-impact examinations, photographs and/or sketches will be included.

7.8 Formation of the Opinion in Glass Comparisons

The results of a glass comparison are generally reported using statements selected from the report wording guidelines in Section 7.9. The strength of an associative conclusion between glass samples is determined by factors such as the number of consistent particles found, the number of broken glass sources transferred and the uniqueness of the properties of the glass. The uniqueness of the refractive index of a glass sample is determined by evaluation of the frequency of occurrence of refractive indices from known glass samples encountered in casework. Frequency of occurrence data will only be considered for flat glass sources, since the current collection of refractive index data does not contain a sufficient sampling of other glass types such as headlight or container glass sources.

The DFS frequency of occurrence database shall be updated every twelve months, a hardcopy of the histogram retained, and the electronic database stored on the DFS intranet.

The factors used to form an opinion regarding an association between questioned samples and known glass sources will generally be assessed at the examiner's discretion. For cases where a strong association may be warranted, consultation with another qualified examiner is required and will be reflected in the notes. The number of particles associated and the number of broken glass sources transferred are usually the most important factors for consideration when comparing glass samples. The case notes will reflect the factors contributing to the strength of the conclusion to include the most recent DFS frequency of occurrence data. A hardcopy of the DFS frequency of occurrence data utilized shall be included with the case notes.

7.9 Report Wording

To the maximum extent possible, report wording will be selected from the following statements. **Physical properties should be delineated and include, but are not limited to color, thickness, temper, float properties, type, and curvature.**

- 7.9.1 The techniques (e.g., polarized light microscopy (PLM), ultraviolet light, digital caliper, Glass Refractive Index Measurement System (GRIM3), stereomicroscopy) used for the analysis will be reported using statements such as the following:
- 7.9.1.1 Item _____ was examined using stereomicroscopy, a digital caliper, and the Glass Refractive Index Measurement system (GRIM3). It should be noted that this examination did not include elemental analysis.
- 7.9.1.2 Items _____ and _____ were examined visually....
- 7.9.2 For multiple known samples, use the appropriate statement:
- 7.9.2.1 The Item _____ and _____ glass samples could be distinguished from each other based upon differences in physical properties and/or refractive index.
- 7.9.2.2 The Item _____ and _____ glass samples could not be distinguished from each other by physical properties and/or refractive index.
- 7.9.2.3 Item _____ is a laminated glass composed of two layers which could be distinguished from each other based upon differences in physical properties and/or refractive index.
- 7.9.2.4 Item _____ is a laminated glass composed of two layers which could not be distinguished from each other by physical properties and/or refractive index.
- 7.9.3 For associations of glass samples or fragments, use this statement:
- The Item _____ glass sample/fragments/particle was/were consistent with the Item _____ glass in physical properties and/or refractive index.
- 7.9.4 For association of recovered glass particles (of those examined) from clothing or objects, use this statement:
- (#particles)* of *(total #particles)* glass particles recovered from the combined debris from Items _____ were consistent with the Item _____ glass in refractive index.
- Physical properties could not be determined due to glass particle size.

- 7.9.5 For association of glasses, use the appropriate opinion statement, based on the uniqueness of the glass source or sources:
- 7.9.5.1 Environmental origin could not be eliminated as a possible source of these particles.
- 7.9.5.2 Based upon the fragments/particles examined, it was concluded that these fragments/particles could have originated from the broken glass source represented by Item _____ or another source of broken glass with the same properties/refractive index.
- 7.9.5.3 Based upon the particles examined, it is considered unlikely/remote that these particles originated from any source other than the broken glass source represented by Item _____.
- 7.9.6 For non-associations, use the appropriate statement:
- 7.9.6.1 Based upon the sample/fragments/particle examined, the Item _____ glass sample/fragments/particle could not be associated with the Item _____ glass due to differences in physical properties and/or refractive index.
- 7.9.6.2 Based upon the particles examined, glass particles recovered from the combined debris from Items _____ could not be associated with the Item _____ glass due to differences in refractive index.
- 7.9.7 If no glass particles are recovered, use this statement:
- 7.9.7.1 No glass particles were recovered from the combined debris from Items _____ for comparison to the Item _____ glass.
- 7.9.7.2 No glass particles were recovered from Item _____ for comparison to Item _____.
- 7.9.8 For recovered glass where a known sample is being requested for comparison purposes the report will generally read:

Glass particles were recovered from Item _____. If a possible source of glass is located, resubmit Item _____ along with a sample of the possible glass source for comparison.

7.9.9 Full wording examples - Glass

7.9.9.1 Example 1

Item 5	One (1) green broken bottle neck
Item 6	Green broken glass pieces from inside hotel

RESULTS

Items 5 and 6 were examined using stereomicroscopy, a digital caliper, and the Glass Refractive Index Measurement system (GRIM3). It should be noted that this examination did not include elemental analysis.

The Item 6 glass fragments were consistent with the Item 5 glass in color, thickness, and refractive index. Based upon the fragments examined it was concluded that these Item 6 fragments could have originated from the broken glass source represented by Item 5 or another source of broken glass with the same properties.

The evidence will be available at the Eastern Laboratory after you have received the results of all requested examinations.

7.9.9.2 Example 2

Item 1	Pieces of broken window
Item 15	One (1) Finish Line athletic black T-shirt, size 3XL
Item 16	One (1) pair of black Joe Boxer sweatpants, size XL

RESULTS

Item 1 was examined using a digital caliper, ultraviolet light and the Glass Refractive Index Measurement system (GRIM3). It should be noted that this examination did not include elemental analysis.

Debris recovered from Items 15 and 16 was examined visually and using stereomicroscopy. Glass particles recovered from the debris were further examined using the GRIM3.

Two (2) glass sources were present in Item 1 which could be distinguished from each other based on differences in float properties and refractive index.

Four (4) of twenty-one (21) glass particles recovered from Item 15 were consistent with one of the broken glass sources in Item 1 in refractive index. Eight (8) of twenty-two (22) glass particles recovered from Item 16 were consistent with the other broken glass source in Item 1 in refractive index. Based on the particles examined it was concluded that these recovered particles could have originated from the corresponding broken glass sources in Item 1 or other broken glass sources with the same refractive index. Physical properties could not be determined due to glass particle size.

The evidence is being returned to the Northern Laboratory where it will be available for personal pickup.

8 HAIR**8.1 Analytical Approach**

Items submitted for hair analysis will generally be limited to answering the following questions: Is the evidential material a hair or a fiber? If it is a hair, is it animal or human? If it is a human hair, is it suitable for nuclear DNA (PCR) typing?

8.1.1 Minimum Standards and Controls

- 8.1.1.1 Any mounting media with a stated expiration date will not automatically be discarded after the stated date. As long as the mounting media has not yellowed and continues to “flow” properly, as determined by the examiner, then it may continue to be used.
- 8.1.1.2 Record the general color and approximate length or range of the hair(s).
- 8.1.1.3 Either count the number of hairs present or provide an approximation as to the number present.

8.2 Recovery of Hairs and/or Fibers**8.2.1 Purpose**

To examine evidence to locate, recover and preserve hairs/fibers for identification and/or comparison purposes.

8.2.2 Summary

- 8.2.2.1 Generally speaking, submitting the article(s) of evidence to the laboratory for the examiner to process is the best approach to the recovery of hairs and/or fibers. There are instances where this is not practical or possible, such as recovering hairs and/or fibers from wall-to-wall carpeting, a large piece of furniture, or a vehicle. In these instances, the recovery may be accomplished at the scene and the recovered hairs and/or fibers submitted for examination.
- 8.2.2.2 The order of preference for the recovery of hairs and/or fibers is manual removal with forceps followed by taping with Post-it type notes or another low tack tape. Gentle scraping may be necessary in certain instances. Vacuuming is rarely, if ever, performed because the debris recovered represents far more than recent hair and/or fiber transfers.

8.2.3 Minimum Standards and Controls

- 8.2.3.1 The examiner shall change the examination paper between victim and suspect or scene exhibits. The examiner may change the paper between multiple victim, suspect or scene items, as necessary.
- 8.2.3.2 There should be only one exhibit opened at a time, unless two separate areas exist for this purpose.
- 8.2.3.3 The examiner shall change gloves and clean their tools between examining the evidence from the victim and the evidence from the suspect.
- 8.2.3.4 If possible, the victim's evidence and suspect's evidence should be examined in separate rooms. If this is not possible, then the separation of victim and suspect evidence in time and/or space will be necessary. Document in case file notes.
- 8.2.3.5 Use separate laboratory coats and evidence collection rooms, if available, for examining materials from victim and suspect to prevent possible cross-transfer contamination.

8.2.3.6 Avoid drafts around the examination area.

8.2.4 Analytical Procedure

8.2.4.1 Spread a clean piece of paper on the examination surface.

8.2.4.2 Examine each item of evidence visually or with the aid of an illuminated magnifier, UV light or stereomicroscope.

8.2.4.2.1 If the item being examined contains hairs and/or fibers that are readily visible, collect these hairs and/or fibers with forceps. As hairs and/or fibers are collected, they should be placed in glassine packets or affixed to Post-it type notes.

8.2.4.2.2 Take care with bulky items which require repositioning on the examination table, to avoid the loss of hairs and/or fibers in the repositioning process.

8.2.4.3 Post-it type notes or other low tack adhesive tapes may be used to recover hairs and/or fibers. The adhesive surface is placed on the item being examined and then pulled away. Hairs and/or fibers will adhere to the adhesive on the tape.

8.2.4.3.1 This method may be especially useful on large items or dark-colored items on which hairs and/or fibers of interest may be difficult to see.

8.2.4.3.2 High tack tape can be used to ensure complete recovery of hairs but only for knit articles such as hats, caps, masks, and scarves. If fibers are probative to the case, a high tack tape must not be used.

8.2.4.3.2.1 The only high tack tape approved for use is clear packing tape.

8.2.5 Scraping is generally discouraged as a method of collection for hairs and/or fibers. If scraping is necessary, the item to be examined is suspended above the examination surface and very gently scraped with a spatula. Scraping in a downward direction allows surface hairs and/or fibers to fall onto the examination paper for collection.

8.3 Human and Animal Hair Identification

8.3.1 Purpose

The purpose of human and animal hair identification is to differentiate human hairs from animal hairs.

8.3.2 Safety Considerations

8.3.2.1 The use of xylene or xylene substitute requires caution. The process may be carried out in a well ventilated area or by using a "Nederman" point-of-use vent, if one is available.

8.3.3 Analytical Procedures

8.3.3.1 The hair will be temporarily mounted (in xylene substitute or water) on a slide for microscopic evaluation. Hair permanently mounted in Permount, Protexx or another permanent media may also be evaluated.

- Using a compound microscope, examine the hair.

8.3.3.2 Based upon the microscopic observations, determine if the hair is human or animal and record appropriate observations (e.g., color, banding, scales, medulla, distribution of pigment granules, and root shape).

8.3.4 References

- 8.3.4.1 Appleyard, H.M. Guide to the Identification of Animal Fibers; Wool Industries Research Association: Leeds, England, 1960.
- 8.3.4.2 Brunner, H. and Coman, B. The Identification of Mammalian Hair, Melbourne: Inkate Press Proprietary Ltd., 1974.
- 8.3.4.3 Hicks, John, Microscopy of Hair, FBI Issue 2, January 1977.
- 8.3.4.4 Wildman, A. B. "The Identification of Animal Fibers" *J. Forensic Science Society* 1961. 1 (2), 1-8.
- 8.3.4.5 Deedrick, Douglas W. and Koch, Sandra L., "Microscopy of Hair Part I: A Practical Guide and Manual for Human Hairs", *Forensic Science Communications*, Vol. 6, (1), January 2004.
- 8.3.4.6 Deedrick, Douglas W. and Koch, Sandra L., "Microscopy of Hair Part II: A Practical Guide and Manual for Animal Hairs", *Forensic Science Communications*, Vol. 6, (3), July 2004.

8.4 Evaluation of Human Hairs for Nuclear DNA Suitability

8.4.1 Purpose

An evaluation of human hairs for nDNA suitability is done to determine whether nuclear DNA profiling may be possible. Only hairs which are in an active growth stage (anagen/catagen) or hairs with tissue attached may be suitable for nuclear DNA (PCR) typing.

8.4.2 Safety Considerations

- 8.4.2.1 The use of xylene or xylene substitute requires caution. The process may be carried out in a well ventilated area or by using a "Nederman" point-of-use vent, if one is available.

8.4.3 Analytical Procedure

- 8.4.3.1 Recovered items may be screened using the stereomicroscope to determine if the item is a hair or a hair fragment.
- 8.4.3.1.1 Hair fragments are not suitable for nDNA testing.
- 8.4.3.2 The hair will be temporarily mounted (in xylene substitute or water) on a slide for microscopic evaluation. Hair permanently mounted in Permount, Protexx or another permanent media may also be evaluated.
- 8.4.3.2.1 The hair is examined using a compound microscope.
- 8.4.3.2.2 If the hair is in a telogen growth stage with no tissue attached it is not suitable for nDNA testing.
- 8.4.3.2.3 If the hair is assessed to either be in an anagen/catagen growth stage or if tissue is present, then the examiner will conclude that the hair may be suitable for nuclear DNA (PCR) testing. The hair will be referred to the Forensic Biology (SX) Section.
- 8.4.3.3 The hair must be prepared for transfer to the SX Section.

- 8.4.3.3.1 Remove the hair and place it on a labeled Post-it type note or in a glassine envelope ensuring no mounting media remains on the hair. The Post-it type note is then placed in a sealed and labeled container for transfer.
- 8.4.3.3.2 If Permout or Protexx was applied to the hair, it must be removed. Xylene or Xylene substitute is used to dissolve the Permout or Protexx. The mountant and solvent are removed and the hair processed as above. Alternatively, liquid nitrogen may be used to remove the hair.
- 8.4.3.3.3 Complete a nuclear DNA (PCR) referral form and transfer the original form to the SX Section along with the evidence.

8.4.4 References

- 8.4.4.1 Linch, C.A., Smith, S.L., Prahlow, J.A., "Evaluation of the human hair root for DNA typing subsequent to microscopic comparison", *Journal of Forensic Sciences* 1998; 43 (2), pp. 305-314.
- 8.4.4.2 Linch, C.A., J. A. Prahlow, "Postmortem Microscopic Changes Observed at the Human Head Hair Proximal End," *Journal of Forensic Sciences*, 2001, 46 (1), pp. 15-20.
- 8.4.4.3 Petraco, N., Fraas, C., Callery, F.X., and DeForest, P.R., "The Morphology and Evidential Significance of Human Hair Roots", *Journal of Forensic Sciences*, 1988, 33 (1), pp. 68-76.

8.5 Evaluation of Human Hairs for Mitochondrial DNA Suitability

8.5.1 Purpose

An evaluation of human hairs/hair fragments for mtDNA suitability is done to assist in the determination of which hairs/hair fragments out of multiple specimens may be most suitable for mtDNA testing.

8.5.2 Analytical Procedure

- 8.5.2.1 Human hairs will be temporarily mounted (in xylene substitute or water) on a glass slide for microscopic evaluation. Hairs permanently mounted in Permout, Protexx or another permanent media may also be evaluated.
- 8.5.2.2 Hairs will be viewed using a compound microscope and observable microscopic properties which may include color, length, diameter, size and distribution of pigment granules among others, are recorded.
- 8.5.2.3 Questioned hairs are grouped according to the observable microscopic properties and the grouping information for these hairs is communicated to the mtDNA examiner.

8.5.3 References

- 8.5.3.1 Linch, Charles A., David A. Whiting, and Mitchell M. Holland, "Human Hair Histogenesis for the Mitochondrial DNA Forensic Scientist," *Journal of Forensic Sciences*, 2001, 46 (4), pp. 844-853.

8.6 Documentation

Examiners should have sufficient detail in the case notes related to the characteristics in each hair in order to support their conclusions. Include a copy of the nuclear DNA (PCR) referral form in the case file, as applicable.

8.7 Report Wording

- 8.7.1 Instrumentation and/or methods used in the examination shall be identified in the body of the report. The report will generally read:

The Item ____ hairs and hair fragments were examined using stereomicroscopy and compound microscopy.

- 8.7.2 Select from the following when no hairs are referred for nDNA testing:

8.7.2.1 No hairs suitable for nuclear DNA testing were recovered from Item ____.

8.7.2.2 No hairs were present in Item ____ that were suitable for nuclear DNA testing.

8.7.2.3 The Item ____ hair was not suitable for nuclear DNA testing.

8.7.2.4 The Item ____ hair fragment was not suitable for nuclear DNA testing.

8.7.2.5 Item ____ consisted of numerous hair fragments that were not suitable for nuclear DNA testing.

8.7.2.6 Item ____ contained one (1) animal hair and one (1) animal hair fragment. Animal hairs and animal hair fragments are not suitable for DNA testing by the Virginia Department of Forensic Science.

- 8.7.3 When referring hairs for nDNA testing, the following may be used:

The Item ____ hair may be suitable for nuclear DNA testing and has been referred to the Forensic Biology Section for analysis.

- 8.7.4 When reporting hairs not suitable for nuclear DNA testing and DNA evidence such as a buccal swab has been submitted, inform the Forensic Biology Section that they may terminate the SX exam and use the following statement to terminate the analysis of the item:

Based upon the above analysis, Item ____ was not examined.

- 8.7.5 If a Certificate of Analysis is necessary for mitochondrial DNA grouping and/or suitability, examples of suitable language are provided below:

8.7.5.1 Item 1 pubic hair combings consisted of numerous fibers, animal hairs, animal hair fragments, two human hair fragments (sub-itemed 1A and 1B), and one human hair (sub-itemed 1C). Items 1A, 1B, and 1C may be suitable for mitochondrial DNA testing. Items 1A and 1B are most similar in physical properties and differ from Item 1C.

8.7.5.2 Two hairs recovered from the Item __ pants may be suitable for nuclear DNA testing and have been referred to the Forensic Biology Section for analysis.

One hair fragment recovered from the Item __ pants was not suitable for nuclear DNA testing, however, meets the recommended length necessary for mitochondrial DNA testing suitability.

In addition, there were fibers of various colors and types, animal hairs, animal hair fragments, human hairs, and human hair fragments recovered from the Item __ pants. Animal hairs and animal hair fragments are not suitable for DNA testing by the Virginia Department of Forensic Science. The additional human hairs and human hair fragments were not suitable for either nuclear or mitochondrial DNA testing due to lack of tissue, improper growth stage, and insufficient length.

9 PAINT**9.1 Analytical Approach**

- 9.1.1 The examiner is typically requested to compare a questioned paint sample or questioned paint deposits/smears on an object(s) to known paint samples. Instead of a Q vs K comparison, the examiner may be requested to conduct a make/model determination or to obtain information on an individual paint sample with no known paint sample available.
- 9.1.2 The examiner first conducts a macroscopic examination of the paint evidence. If appropriate, a fracture match comparison can be conducted. If the evidence to be examined is a liquid paint sample, the liquid is applied to clean glass slides and allowed to dry.
- 9.1.3 Paint can be characterized by a number of physical and chemical properties. The sample size and type of paint present in the sample will assist the examiner in determining which subsequent tests will be conducted. Record the approximate amount and/or size of paint sample as received. If a sufficient quantity of questioned paint exists, then the known and questioned paints will be compared and the properties noted in a side-by-side examination as follows: microchemical tests, microsolubility tests, fluorescence microscopy, SEM-EDS, and FTIR. The order of these exams and any additional testing is left to the discretion of the examiner and based upon the evidence at hand.
- 9.1.4 During a microscopic examination, the examiner notes any physical properties which assist in characterizing a paint sample. These properties can include color, type (vehicular/ structural; enamel/lacquer; with/without decorative flake), texture, thickness, layer sequence and weathering. The microscopic examination is conducted separately for known and questioned paint particles. At this point in the examination, fluorescence microscopy may be useful in assessing the paint.
- 9.1.5 For the microsolubility tests, the examiner observes the solubility of individual paint layers in chloroform and acetone. Toluene will also be used if a lacquer paint is indicated. For the microchemical reaction tests, the examiner observes the reactions of individual paint layers with certain chemical reagents: LeRosen and/or Diphenylamine (DPA). DPA is only necessary if a nitrocellulose lacquer is suspected. The use of LeRosen is optional.
- 9.1.6 The examiner shall also analyze paint samples using instrumental techniques. FTIR is a useful technique to compare the organic chemical composition of paint layers. SEM-EDS is useful in determining the elemental chemical composition of paint layers. If K and Q paints are sufficient for organic and elemental characterization, then fluorescence microscopy will also be employed for comparison of the samples.
- 9.1.7 After the analytical data has been obtained, the examiner compares the data for the known and questioned paint samples to evaluate the possible association of the K & Q.
- 9.1.8 When paint particles or smears are present for make/model determination, the examiner must first characterize the paint for suitability of searching. The physical and chemical characteristics of the paint are evaluated to determine if an OEM sequence is present. If an OEM sequence is present, each layer of the paint will be analyzed via FTIR and designated appropriately (OU2, OU1, OT1, OT2, etc.). Appropriate information for each layer is entered into the currently available reference collection of known automotive paints, the Paint Data Query (PDQ) database, and a search is conducted. [Note: pigments present in the topcoat layers should not be included when conducting searches as it may lead to false elimination.] Alternatively, the collected FTIR spectra may be searched against the PDQ entries using KnowItAll spectral search software. If successful, the search results will be reported as to the possible make, model and range of years in which the OEM sequence may have been used. If no OEM sequence is present, report the topcoat color of the paint and the presence of decorative flake, when applicable. If a partial OEM sequence is present, PDQ spectral searches may be attempted. Manufacturers' information and automotive refinish books should be used in conjunction with PDQ results to limit make/model/year results.

- 9.1.8.1 The substrate from which the recovered paint originated should be determined if possible. This can be done using physical characteristics or the chemistry of the paint. Paint from plastic substrates can be searched, however, the results of these searches may not yield suitable results as paint from plastic surfaces are currently underrepresented in the database.
- 9.1.8.2 Generally when performing spectral searching, the layers are searched in the following order: (1) OU1 primer surfacer layer – to determine the possible plants [viewing at least the top 200 hits], (2) OT2 clear/colorless layer – to determine the possible year range with each of the plants, (3) OU2 primer layer – to narrow results or eliminate plants, and (4) OT1 colored layer – to narrow results or eliminate plants. Alternatively, more than one paint layer spectrum may be searched at the same time using KnowItAll software.
- 9.1.8.3 Primer layers, especially the OU1 layer, may be compared with the Munsell color books to determine their Munsell values. These values may be added to a PDQ search to add further discrimination. This is especially useful for unusual, not color coordinated primers.
- 9.1.8.4 The PDQ content manual, internet searches, or contacts with manufacturing representatives may be used to correlate plants/years to makes/models. Refinish books should be searched to confirm the color was produced in the year range by the manufacturer. These books may also allow the examiner to further narrow the year range or determine a possible model if the information is provided. If the model information is not available in the repaint books, internet references, such as the Sherwin-Williams website, may provide it.
- 9.1.8.5 If assistance from the Royal Canadian Mounted Police (RCMP) PDQ Maintenance Team is needed, contact the Trace Evidence PDQ resource person.

9.2 Paint Recovery Techniques

9.2.1 Purpose

To recover foreign paint that might be present on an item of evidence to allow analysis of paint samples.

9.2.2 Minimum Standards and Controls

- 9.2.2.1 The examiner must change the examination paper between victim and suspect or scene exhibits.
- 9.2.2.2 The examiner may change the paper between multiple victim, suspect or scene items, as necessary.
- 9.2.2.3 The examiner must change gloves and clean their tools between examining the evidence from the victim and the evidence from the suspect.

9.2.3 Analytical Procedures

- 9.2.3.1 Thoroughly clean the examination area prior to the evidence being retrieved.
- 9.2.3.2 Remove the item of evidence from the container and place it upon a clean sheet of paper. Note the condition of the evidence. Supporting photographic documentation may be included.
- 9.2.3.3 Conduct a thorough inspection of the item for any visible paint particles/deposits. These are collected in evidence containers which may include plastic or metal boxes, glassine envelopes, weighing paper or a paper evidence fold.
- 9.2.3.4 If no paint particles/deposits are readily observed, the examiner may use a magnifier/viewer or stereomicroscope for searching purposes. If tears or other damage are observed on an item,

these areas may be cut out for further analysis. If particles/deposits are not observed and the evidence consists of textile materials then scraping will be conducted.

9.2.3.5 Scraping is done by holding or suspending the item over a clean sheet of paper and scraping the evidence thoroughly using a clean spatula. Any recovered debris is collected in an evidence container for further analysis.

9.2.3.6 The evidence must be photographed, sketched or otherwise documented to note the locations of any probative tears, paint deposits/smears or other unusual features.

9.2.4 References

9.2.4.1 Thornton, John I., "Forensic Paint Examination", Forensic Science Handbook, Volume 1, Second Edition, Pearson Education, Inc., 2002, pp. 429-478.

9.3 Macroscopic/Microscopic Examination

9.3.1 Purpose

To examine submitted paint evidence on a macroscopic and microscopic scale, noting physical properties, generally as the first step in the characterization and comparison of known and questioned paint samples.

9.3.2 Minimum Standards and Controls

9.3.2.1 Clean tools and work areas are a must.

9.3.3 Analytical Procedures

9.3.3.1 Thoroughly clean the examination area prior to the evidence being retrieved.

9.3.3.2 The sample is examined with a stereomicroscope and the physical properties of each paint sample are noted and recorded. A paint worksheet may be used for this purpose when conducting microsolubility testing. The layer sequence, color, type, texture, thickness and any unusual features are noted for each sample, as appropriate. The examiner can also use the comparison microscope or the polarized light microscope in determining the physical characteristics of a paint sample.

9.3.3.3 Paint particles may be manipulated with fine forceps or other appropriate tool and may be dissected using a fine scalpel blade to allow the examiner to thoroughly characterize the physical properties.

9.3.3.4 Mineral oil or water should be used to compare the K & Q paint particles side-by-side on black and white backgrounds with varying intensities of light.

9.3.3.5 If any meaningful differences are observed between the questioned and known particles, such as color, the examiner can discontinue the analysis.

9.3.4 References

9.3.4.1 "The Evidential Value of Automobile Paint Chips", Ryland, S.G. and Kopec, R.J., *Journal of Forensic Sciences*, Vol 24(1), 1979, pp. 140-147.

9.3.4.2 "Evaluation of Automobile Paint Flakes as Evidence", Gothard, J.A., *Journal of Forensic Sciences*, Vol 21(3), 1976, pp. 636-641.

9.4 Microsolubility/Microchemical Tests

9.4.1 Purpose

Microchemical and microsolubility tests provide indications of the chemical nature of the paint and are used to classify the type of paint present (lacquer/enamel, dispersion/solution/nitrocellulose lacquer type).

9.4.2 Safety Considerations

9.4.2.1 Concentrated acids, solvents and reagent chemicals (e.g., formaldehyde) should be used in a well-ventilated area with proper precautions being exercised to minimize skin contact.

9.4.3 Minimum Standards and Controls

9.4.3.1 The following chemicals should be reagent grade or better:

acetone, chloroform, diphenylamine, concentrated sulfuric acid, formaldehyde, glacial acetic acid, and toluene

9.4.4 Analytical Procedures

9.4.4.1 Place paint particles in a well (or depression) slide, a spot plate or on a microscope slide in the field of view of a stereomicroscope.

9.4.4.2 Apply the reagent or solvent to the particles. The solubility of each layer is noted on the paint worksheet. If LeRosen or diphenylamine are used, the effect of each reagent on the individual paint layers is noted.

9.4.4.3 Sample size and type will assist the examiner in determining which tests will be conducted. Due to subtle differences that may be observed during microchemical comparisons, it is suggested that these be done in a side-by-side fashion.

9.4.4.4 Chloroform and acetone will be used if a sufficient quantity of paint is available. Toluene and diphenylamine will be used as appropriate for distinguishing lacquers. LeRosen is optional.

9.4.4.4.1 For those reagents where the QC check does not give an acceptable result, repeat the QC check with fresh reagent until an acceptable result is achieved.

9.4.4.5 If any meaningful differences are observed between the questioned and known paint particles, the examiner can discontinue the analysis.

9.4.5 References

9.4.5.1 "Application of the LeRosen Test to Paint Analysis", Linde, H.G. and Stone, R.P., *Journal of Forensic Sciences*, Vol 24(3), 1979, pp. 650-655.

9.4.5.2 "Solubility Characterization of Automotive Paints", Thornton, J.I., Kraus, S., Lerner, B. and Kahane, D., *Journal of Forensic Sciences*, Vol 28(4), 1983, pp. 1004-1007.

9.4.5.3 Thornton, John I., "Forensic Paint Examination", in *Forensic Science Handbook*, Volume 1, Second Edition; R. Saferstein, ed, Pearson Education, Inc.: Upper Saddle River, NJ, 2002, pp. 460-462.

9.4.5.4 Scientific Working Group on Materials Analysis (SWGMA), "Forensic Paint Analysis and Comparison Guidelines", *Forensic Science Communications*, Vol. 1 (2), July 1999.

- 9.4.5.5 Forensic Analysis of Paints (2010 Training Materials), F.B.I.
- 9.4.5.6 Forensic Paint Identification and Comparison (2011 Training Materials), Hooke College of Applied Sciences.
- 9.4.5.7 Forensic Examination and Comparison of Paint, Tapes, and Adhesives with a Focus on Interpretation of the Evidence (2011 Training Materials), Florida International University.

9.5 Fluorescence

9.5.1 Purpose

To determine and compare the fluorescence characteristics of paint samples, deposits or smears.

9.5.2 Safety Considerations

- 9.5.2.1 Do not look directly into the fluorescence source.

9.5.3 Analytical Procedure

- 9.5.3.1 Paint samples, deposits or smears are subjected to different wavelengths of light and the resulting fluorescence colors or lack thereof are noted on the fluorescence worksheet.
- 9.5.3.2 Paint samples, deposits or smears may be examined “dry” or mounted in a suitable “non-fluorescent” mounting medium, however, questioned and known samples must be examined simultaneously; side-by-side using the stand-alone polarized light microscope with the room lights off.
- 9.5.3.3 Fluorescence QC procedure:
 - 9.5.3.3.1 Perform fluorescence QC check prior to using the fluorescence microscope for casework. Use the fluorescence QC check sample and record observations on the fluorescence worksheet. If the fluorescence QC check fails to produce acceptable colors or intensity, troubleshoot the fluorescence microscope as appropriate and repeat the QC check. If the QC check continues to fail, take the fluorescence microscope out of service until the problem is resolved.
 - 9.5.3.3.2 Fluorescence cubes to be used are WU (wide UV – range 330 – 385nm), WBV (wide blue violet – range 400 – 440nm), WB (wide blue – range 450 – 480nm) and WG (wide green – range 510 – 550nm). Record observations on a fluorescence worksheet
- 9.5.3.4 Paint smears are usually not homogeneous. Great care should be employed when interpreting the results of the comparison of paint smears with paint particles; some variation in fluorescence properties is expected. In addition, when two paint samples are compared, small differences may be seen if one of the samples has been subjected to sunlight more often than the other sample e.g., paint from a vehicle’s hood compared to paint from the bottom portion of a fender.
- 9.5.3.5 Caution should be taken when eliminating samples based upon fluorescence data alone. Detailed notes explaining the basis for the elimination are a must. Eliminations based upon fluorescence will be reported as “due to differences in fluorescence”.

9.6 Instrumental Analysis

9.6.1 FTIR

9.6.1.1 Safety Considerations

9.6.1.1.1 Do not look directly into the laser source.

9.6.1.1.2 The MCT detector for the microscope accessory must be cooled with liquid nitrogen. Insulated gloves and safety glasses shall be worn when filling the transport Dewar as well as the instrument reservoir.

9.6.1.2 Minimum Standards and Controls

9.6.1.2.1 Ensure that the daily QC has been performed.

9.6.1.2.2 Replicate analyses may be employed to ensure reproducibility or to demonstrate variability within a sample.

9.6.1.3 Analytical Procedures

For the examination of the individual layers of paint in a particle, the examiner must prepare the particle for analysis.

9.6.1.3.1 The sample may be prepared by using a scalpel to remove a very thin cross-section of any/all paint layers. This is followed by further sample flattening using a roller or microcompression cell with diamond windows.

9.6.1.3.2 Alternatively, thin peels or a very low angle cut may be made to expose the paint layers and each layer may be sampled one at a time for use in the microcompression cell.

9.6.1.3.3 The flattened sample is placed on a KBr window or left in the microcompression cell. With the FTIR microscope accessory, the examiner adjusts the aperture edges to mask all but individual layers. Spectra are obtained for each layer, when possible, using standard methods.

9.6.1.3.4 If any meaningful differences are observed in the spectra of the questioned and known samples, the examiner can discontinue the analysis.

9.6.1.4 References

9.6.1.4.1 "FT-Infrared Spectroscopy of Automobile Paints using Infrared Microscopy", Stoecklein, W. and Gloger, M., *Nicolet FT- IR Spectral Lines*, Spring- Summer 1988, pp. 2-6.

9.6.1.4.2 "Forensic Applications of IR Microscopy", Compton, S. and Powell, J., *American Laboratory*, Nov 1991, pp. 41- 51.

9.6.2 Scanning Electron Microscopy/Energy Dispersive X-Ray (SEM-EDS)

9.6.2.1 Safety Considerations

9.6.2.1.1 The EDS detector system may require cooling with liquid nitrogen. Insulated gloves and safety glasses shall be worn when filling the Dewar.

- 9.6.2.2 Minimum Standards and Controls
- 9.6.2.2.1 To minimize take-off angle effects, paint samples should be flat and mounted normal to the electron beam.
- 9.6.2.3 Analytical Procedures
- 9.6.2.3.1 The sample is mounted on a sample stub covered with carbon adhesive tape (flat for single layer paint or on edge for multilayered paint, shavings may also be used). Due to potential heterogeneity within the paint layer, the sample should be at least 2mm in length whenever possible.
- 9.6.2.3.2 A representative X-ray spectrum is collected from each layer of each sample (K and Q). A hard copy is printed to document the elemental composition.
- 9.6.2.3.3 Comparison of K and Q samples is made on a qualitative basis; some variation in elemental concentration is not uncommon.
- 9.6.2.4 References
- 9.6.2.4.1 “Analysis Protocol for Discrimination of Automotive Paints by SEM- EDXA Using Beam Alignment by Current Centering”, Beam, T.L. and Willis, W.V., *Journal of Forensic Science*, Vol 35(5), 1990, pp. 1055- 1063.
- 9.6.2.4.2 Scientific Working Group on Materials Analysis (SWGMAT)., “Standard Guide for Using Scanning Electron Microscopy/X-ray Spectrometry in Forensic Paint Examinations”, *Forensic Science Communications*, Vol. 4 (4), October 2002.

9.7 Documentation

- 9.7.1 At a minimum, each case file will include a paint worksheet(s) (as applicable), any notes detailing physical properties and item description, hard copies of all spectra generated during the analysis and instrumental conditions for all analyses conducted.
- 9.7.2 Make/model determinations:
- 9.7.2.1 For searches using the PDQ software, the coding searched and the resulting number of hits should be included in the case file.
- 9.7.2.2 For searches using the KnowItAll software, spectral evaluation of one paint layer provides a listing of the possible plants of origin. PDQ worksheets may be subsequently used to document narrowing year ranges. Both the listing of the possible plants of origin and the PDQ worksheets must be present in the case file. Alternatively, more than one paint layer spectrum may be searched at the same time using KnowItAll software.
- 9.7.2.3 FTIR spectra of representative hits must be included in the file for direct comparison. At least one spectrum per layer per plant of origin, if available, is needed for case file documentation. If a wide year range is indicated, additional spectra may be printed, when possible, to document these findings.
- 9.7.2.4 Comparable colors (color code and/or name) found when searching the refinish books should be documented.

9.8 Report Wording

9.8.1 Techniques utilized in the analysis shall be listed on the report:

Item ____ was examined using stereomicroscopy, microsolubility tests, microchemical tests, fluorescence microscopy, Fourier Transform Infrared Spectrophotometry (FTIR), and Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS).

9.8.2 For paint cases where multilayered particles with consistent K and Q samples:

The multilayered blue paint particles with decorative flake in Items 1 and 2 were consistent in colors, textures, types, layer sequence, and chemical compositions.

9.8.2.1 “Chemical compositions” is applicable for multilayered paint particles suitable for FTIR and/or SEM-EDS.

9.8.2.2 “Chemical composition” includes both organic and elemental composition. There may be an occasion when one or both of these terms is used in lieu of “chemical” composition.

9.8.3 For paint cases where multilayered paint particles with feathering effects or missing layer structure, but with everything else consistent:

The multilayered gray paint particles with decorative flake in Items 1 and 2 were consistent in corresponding (or respective) colors, textures, types, layer sequence, and chemical compositions.

9.8.4 The use of the term “consistent” must be accompanied by what the particles were “consistent” in (colors, textures, types and so on). This term is generally used when there are no unexplainable differences in the testing conducted on the known and questioned samples. It is recognized that there may be instances when, based upon the data, the term “similar” may be more appropriately substituted for “consistent”. The reason for the use of the term “similar” in lieu of “consistent” must be explained in the report.

9.8.4.1 There may be an occasion when these terms are used in the same sentence:

The multilayered green paint particles in Items 1 and 2 were consistent in colors, textures, types and layer sequence, and were similar in chemical composition because of slight differences in organic composition.

9.8.5 If the particles are different:

9.8.5.1 Based upon the particles examined, the orange paint particles in Items 1 and 2 could not be associated due to differences in color. (or any of the following criteria: type, texture, layer sequence, chemical composition)

9.8.5.2 Based upon the samples examined, green paint smears with decorative flake present on Item 1 could not be associated with the Item 2 green paint with decorative flake due to differences in chemical composition.

9.8.6 If sample size or condition precludes a full examination:

Based upon the particles examined, the red paint particles in Items 1 and 2 were consistent in colors and types (or in whatever tests were conducted). Sample condition precluded a complete analysis.

9.8.7 The sample condition (which includes sample amount) precluded a complete analysis and no conclusion could be drawn from what data does exist:

9.8.7.1 The condition of the Item 1 blue smears precluded a complete analysis and comparison with the Item 2 blue paint.

9.8.7.2 No accurate color comparison could be made with the Item 1 red paint with decorative flake due to the sample condition of the Item 2 smears.

9.8.8 If no paint was recovered:

No paint was recovered from Item 1 for comparison to Item 2.

9.8.9 If no vehicular paint was recovered:

No vehicular paint was recovered from Item _____.

No apparent vehicular paint was recovered from Item _____.

9.8.10 If based upon the analysis of one or more items, the remaining items will not be analyzed use:

Based upon the above results, Items _____ and _____ were not analyzed.

Based upon the above results, no further analysis was conducted on Item _____.

9.8.11 Color and decorative flake presence, when applicable, must be reported when observed. Number may be used in any of the above examples and is especially useful when indicating a limited amount of sample (e.g., "Two minute multilayered red paint particles...").

9.8.12 After the examiner has used one of the paint statements, a conclusion usually follows and ends the reporting results prior to describing the disposition of the evidence on the Certificate of Analysis.

9.8.12.1 Based upon the particles examined, it was concluded that the smears on Item ____ could have originated from the top layer of the Item ____ paint or another source of paint with the same _____ (color, type, composition, etc.)

9.8.12.2 Based upon the particles examined, it was concluded that the Item ____ paint could have had a common origin with Item _____ or another source of paint with the same _____. (colors, textures, types, layer sequence, etc.)

9.8.12.3 Based upon the particles examined, it was concluded that the paints in Items A-Z either originated from the same source or different sources painted in the same manner.

9.8.12.4 Based upon the particles examined, it is considered unlikely that the paint in Item ____ originated from any source other than the _____ vehicle OR originated from any source other than the _____ vehicle represented by Item ____.

9.8.12.5 Based upon the particles examined, it is considered remote that the interchange of paints (or materials) described above occurred in any manner other than the contact of the two vehicles (or objects).

9.8.12.6 Based upon the particles examined, it is considered extremely remote that the interchange of paints (or materials) described above occurred in any manner other than the contact of the two vehicles (or objects).

9.8.13 For samples where a search of the PDQ, paint database was successful:

9.8.13.1 A search of the PDQ (Paint Data Query) database indicated that the Item _____ foreign multilayered red paint with decorative flake was consistent with paint used on the following vehicles:

1998-1999 Toyota Avalon

1998-2001 Toyota Camry

It should be noted that not all makes/models/years of vehicles produced by each manufacturer are present in the PDQ database.

Paint particles recovered from Item ____ were suitable for comparison purposes. If a possible source of paint is located, resubmit Item ____ along with a sample of the possible paint source for comparison.

9.8.14 For samples where an OEM sequence is not present:

9.8.14.1 The red paint (paint particles, paint smears) with decorative flake present (in the debris from, on) Item ____ was consistent with (indicative of) a repaint; therefore, no make/model determination could be made. The red paint (paint particles, paint smears) with decorative flake was (were) suitable for comparison purposes. If a possible source of paint ...

9.8.14.2 Item 1 consisted of a foreign black paint particle that was not suitable (include why) for make/model determination but was suitable for comparison purposes. If a possible source of paint...

9.8.14.3 Item 1 consisted of a two-layered foreign black paint particle that was searched using the PDQ (Paint Data Query) database. The paint particle consisted only of topcoats, therefore the search results did not indicate a possible make/model of origin.

9.8.14.4 Item 1 consisted of a multilayered black paint sequence on a plastic substrate. A search of the PDQ (Paint Data Query) database was conducted, the results of which did not indicate a possible make/model of origin. It should be noted that painted plastic substrates are not well represented in the database.

9.8.15 For recovered paint where samples are being requested for comparison purposes the report will generally read:

Foreign (color) paint (with decorative flake) was recovered from Item __ which was suitable for comparison purposes. If a possible source of paint is located, resubmit Item __ along with a sample of the possible paint source for comparison.

9.8.16 Full Report Wording Examples – Paint:

9.8.16.1 Example 1

Item 3	Paint chips from victim's vehicle
Item 4	Paint chips from suspect's vehicle

RESULTS

Items 3 and 4 were examined using stereomicroscopy, microsolubility tests, microchemical tests, fluorescence microscopy, Fourier Transform Infrared Spectrophotometry (FTIR), and Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS).

The white paint particles in Items 3 and 4 were consistent in colors, textures, types, layer sequence, and chemical compositions. Based upon the particles examined, it was concluded that the paints in Items 3 and 4 either originated from the same source or different sources painted in the same manner.

The disposition of the evidence and the results of other requested examinations are the subject of another report.

9.8.16.2 Example 2

Item 3	Green paint chip found at accident scene
Item 5	Green paint chips from 1999 Dodge truck (passenger door)
Item 6	Green paint chips from 1999 Dodge truck (passenger side, side of bed)
Item 7	Green paint chips from 1999 Dodge truck (passenger side, fender)

RESULTS

Items 3 and 6 were examined using stereomicroscopy, microsolubility tests, microchemical tests, fluorescence microscopy, Fourier Transform Infrared Spectrophotometry (FTIR), and Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS).

The multilayered dark green paint particles with decorative flake in Items 3 and 6 were consistent in colors, textures, types, layer sequence, and chemical compositions. Based upon the particles examined, it is considered remote that the paint in Item 3 originated from any source other than the suspect vehicle represented by Item 6.

Based upon the above results, Items 5 and 7 were not analyzed.

The evidence is being returned to the Northern Laboratory where it will be available for personal pickup.

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10 **PRIMER RESIDUE****10.1 Analytical Approach**

- 10.1.1 One or more Primer Residue Evidence Handling Worksheets are generated. For results data set purposes, each Primer Residue kit is to be considered a separate item. The Primer Residue kit is opened and each of the sample vials are labeled, and the stub surfaces are visually examined. If debris is present go to Section 10.2, Carbon Evaporation or use the ASPEX Explorer in VP mode. Otherwise, proceed to Section 10.3, Automated Scanning Electron Microscope/Energy Dispersive X-ray System (SEM-EDS) Analysis.
- 10.1.2 Minimum Standards and Controls
- 10.1.2.1 Primer Residue kits are ordered by DFS to our specifications. Two percent of the Primer Residue kits in each new lot must pass QC inspection before any kits from that lot are released to User Agencies.
- 10.1.2.1.1 A visual inspection is made of the Primer Residue kits to note whether all components of the kit are present. Any visible debris on the collection surface is noted. Both samples from each kit are analyzed via automated SEM-EDS. A copy of the Primer Residue SEM/EDS Worksheet and Analysis Summary Sheet is retained. Samples from these kits become future negative control samples for automated Primer Residue runs.

10.2 Carbon Evaporation

- 10.2.1 Purpose
- 10.2.1.1 Carbon evaporation, or coating, makes the sample electrically conductive and reduces charging in the SEM. In addition, carbon does not interfere with EDS analysis.
- 10.2.1.2 Special Considerations that should be noted with this technique: Carbon evaporation reduces charging on samples containing visible debris. Therefore, the overall automated SEM-EDS run time may be shortened due to reduction in charging of the sample.
- 10.2.2 Safety Considerations
- 10.2.2.1 Carbon evaporation produces a bright arc at the carbon rod tip source. During evaporation, the carbon rod should only be viewed through an appropriate welder's glass.
- 10.2.3 Minimum Standards and Controls
- 10.2.3.1 A negative control is included with all Primer Residue samples to be coated for a given run. This control is separate from the negative control described in 10.3.3.6.
- 10.2.3.2 A glass slide is used to evaluate the coating.
- 10.2.4 Analytical Procedures
- 10.2.4.1 The bottom of the sample stubs are labeled before coating samples.
- 10.2.4.2 The carbon evaporator operations manual is followed.
- 10.2.4.3 The sample plenary stage is slowly rotated during the coating procedure.

10.2.5 References

- 10.2.5.1 Gabriel, B. L. SEM: A User's Manual for Materials Science. 1985, pp.156 -161. ISBN: 0-87170-202-9.

10.3 Automated SEM-EDS Primer Residue Analysis, ASPEX: VP2000, 3000, and Explorer

10.3.1 Purpose

- 10.3.1.1 Automated SEM-EDS is used to search samples for potential primer residue particles. These instruments search a user defined area at a magnification of 500X on each sample. Generally speaking, a particle characteristic of primer residue is defined as a particle with a spherical or molten appearance (noncrystalline) containing the elements: Pb, Ba and Sb. Particles consistent with primer residue would have similar morphology and typically include 2 of the 3 elements listed above. Other elemental profiles for consistent particles and additional elements allowable in primer residue particles can be found in 10.3.5.2.

- 10.3.1.2 The ASPEX VP2000 and ASPEX Explorer instruments are operated in either high vacuum mode for electrically conductive samples or in variable pressure mode at 0.2 Torr for nonconductive samples.

- 10.3.1.3 This is a sensitive, non-destructive technique used to detect the primer residue component of gunshot residue.

10.3.2 Safety Considerations

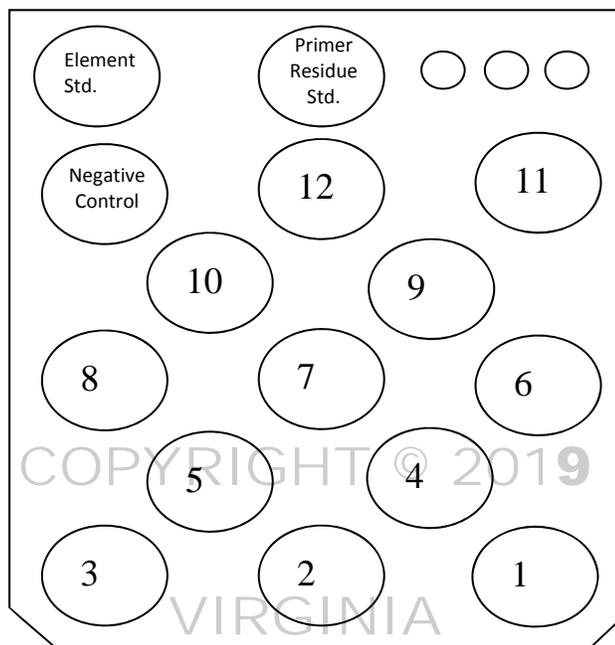
- 10.3.2.1 The SiLi EDS detector system must be cooled with liquid nitrogen. Insulated gloves and safety glasses shall be worn when filling any Dewar.
- 10.3.2.2 During filament replacement the beam power supply located on the back panel of the beam supply under the bias knob is disabled.
- 10.3.2.3 Be aware of elevated temperatures when changing a filament which has been in operation.
- 10.3.2.4 If operating in variable pressure mode, the instrument is changed to high vacuum mode before changing the filament. In addition, the beam is always turned off before changing vacuum modes.

10.3.3 Minimum Standards and Controls

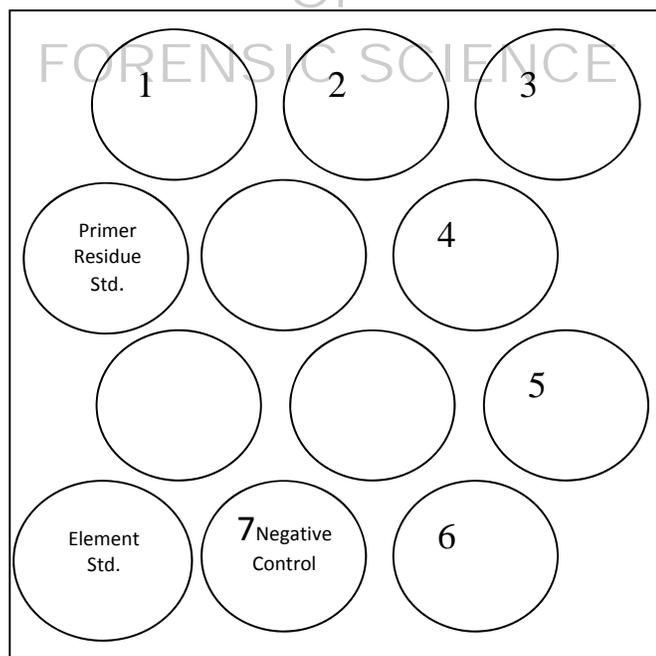
- 10.3.3.1 The EDS detector is electronically calibrated prior to an automated primer residue run. The Cu $L\alpha$ peak should be symmetrical through the peak centroid line as should the Cu $K\alpha$ peak. The centroids of these peaks will be monitored during the automated run. As a matter of QA these peaks should be within 0.03 KeV of the nominal values. FWHM of Cu $K\alpha$ will also be monitored during the automated run. It should not exceed 170 eV. If it is not possible to obtain the appropriate values, recalibrate the EDS detector and rerun the samples. If it is still not possible to obtain the appropriate values, contact ASPEX service.
- 10.3.3.2 Before an automated primer residue run begins, the SEM filament must be saturated and aligned with the manual gun centering knobs on a copper standard where beam damage will not occur. Also, the filament is electronically centered by monitoring the BSED brightness and contrast of the copper standard in the line scan mode. Filament centering is adjusted in the advanced filament application.
- 10.3.3.3 A positive control is a sampling device containing known primer residue particles.

- 10.3.3.4 The backscatter detector thresholds are set to detect primer residue particles in the 1 micron size range.
- 10.3.3.5 A QC analysis is run on the ENFSI 2008 proficiency sample at the beginning of the run, then on the positive control after 3 samples have been examined, and on the same area on the positive control at the end of the run. After the run is complete, data from the ENFSI 2008 proficiency sample is mapped. A total of 90% or greater of the synthetic gunshot residue particles, down to and including particles as small as 1.2 microns must be found for the QA to pass. In addition, 10 particle images and their corresponding spectra from each of the positive controls are generated showing primer residue particles found in the 1 micron size range. The sheets containing the ten images and spectra and the ENFSI 2008 proficiency sample map are stored in a QC notebook in the SEM lab. If a primer residue particle in the 1 micron size range is not found on the positive control or if the ENFSI 2008 proficiency sample map does not pass the QA criteria listed above, and any case sample does not contain particles characteristic of primer residue, the backscatter detector threshold must be readjusted and the samples rerun.
- 10.3.3.6 In every automated SEM-EDS primer residue run, a negative control is analyzed just before the final positive control field. The negative control is the first sample to be loaded in the sample holder. The negative control sample is discarded after the automated primer residue run is complete.
- 10.3.3.7 Approximately once a month the photographic unit of the SEM is checked using an ASTM measurement standard and a high resolution, gold islands on carbon sample. Apertures are replaced if the image cannot be stigmated.
- 10.3.4 Analytical Procedures
- 10.3.4.1 A primer residue analysis setup is performed by setting up or using an existing .afa file.
- 10.3.4.2 A new automated run is setup. DHD_.zrr (the most recent file available) and gsrchem139.vcf files are selected when using the ASPEX VP 2000 instrument. 25KVDHD9.zrr and gsr25sdd.vcf files are selected when using the ASPEX 3000 instrument. 25KVDHD9.zrr and GSR25OMs.vcf files are selected when using the ASPEX Explorer instrument. Once the automated run is complete the QC report is printed and saved.
- 10.3.4.3 Samples are loaded into the SEM as described on the Primer Residue Worksheet SEM/EDS. Sample location and placement is indicated on the stage map diagrams below. The ENFSI 2008 proficiency sample will be located next to the primer residue reference standard in the permanent sample holder bar.

10.3.4.3.1 ASPEX 3000 and Explorer Stage Map



10.3.4.3.2 ASPEX VP2000 Stage Map



10.3.4.4 The SEM is pumped down to operational vacuum.

10.3.4.5 The filament is saturated at 20 KV when using the ASPEX VP2000 instrument and at 25 KV when using the ASPEX 3000 and Explorer instruments. The filament is allowed to stabilize up to one hour.

- 10.3.4.6 When using the ASPEX VP2000 instrument the beam is placed on a copper standard and the spot size adjusted to achieve approximately 1,500 CPS with a process time of 25.6 μ sec at 20KV. A spectrum from 0 to 10.24 KeV is collected and calibrated using the EDS detector calibration software. When using the ASPEX 3000 instrument the beam is placed on a copper standard and the spot size adjusted to achieve approximately 20,000 CPS with a process time of 3.2 μ sec at 25KV. A spectrum from 0 to 10.24 KeV is collected and calibrated using the EDS detector calibration software. When using the ASPEX Explorer instrument the beam is placed on a copper standard and the spot size adjusted to achieve approximately 30,000 CPS with a process time of 3.2 μ sec at 25KV. A spectrum from 0 to 10.24 KeV is collected and calibrated using the EDS detector calibration software.
- 10.3.4.7 The backscatter electron detector is set to detect particles approximately 1 micron or greater in diameter in the following manner: After the objective lens is degaussed using the beam blank function at 1000X in an area where beam damage will be minimized, the stage is driven to the QA field on the primer residue positive control sample. An area is located where particles in the 1micron size range are visible at 1000X in the right hand image. This image is focused and stigmated using the mini imaging and medium scan speed mode. Imaging is then switched to normal and high scan speed.
- 10.3.4.8 The objective lens is degaussed at 1000X and each sample to be run is then checked using a three point focus system through the stage setup program. It is imperative that the sample coordinate positions are defined in exactly the same order as listed on the primer residue setup sheet. Areas of the stub where the aluminum surface is exposed should be avoided.
- 10.3.4.9 The magnification is returned to 100X. The area containing the particles in the 1 micron size range on the primer residue control sample is focused as described in Step 10.3.4.7. If setting up a new .afa file, the quality check is reviewed and set to check copper before the first, after the last and between every sample.
- 10.3.4.10 The backscatter detector thresholds are set to detect particles in the 1 micron size range. Typically, this involves low brightness and high contrast settings.
- 10.3.4.11 The automated run is started. Data is stored in the next sequential run file.
- 10.3.4.12 The ENFSI 2008 proficiency sample map and the primer residue control sample field is monitored to ensure particles in the 1micron size range are being detected. If they are not, the backscatter image and/or focus must be readjusted.
- 10.3.4.13 The Primer Residue Worksheet SEM/EDS is completed listing the SEM-EDS operating conditions used in the automated run.
- 10.3.4.14 Each sample surface is searched until 10 potential particles characteristic of primer residue are found or until the entire predesignated area is searched or 10,000 particles have been analyzed.
- 10.3.4.15 When the automated run is finished (typically the following day) a calibration is done on copper to check the EDS detector at 20KV, as described in 10.3.4.6, for the ASPEX VP 2000 and ASPEX 3000 instruments. The ASPEX Explorer instrument is calibrated at 25KV as described in 10.3.4.6.
- 10.3.4.16 The operator performs a manual confirmation at 20KV of the potential primer residue particles detected during the automated run on the ASPEX VP2000 and ASPEX 3000 instruments. The manual review on the ASPEX Explorer instrument is conducted at 25 KV.

10.3.5 References

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- 10.3.5.2 ASTM E 1588-10 Standard Guide for Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive Spectroscopy.
- 10.3.5.3 Basu, S., "Formation of Gunshot Residues," Journal of Forensic Sciences, Vol. 27, 1982, pp. 72-91.
- 10.3.5.4 DeGaetano, D. H., Siegel, J. A., and Klomparens, K. L., "A Comparison of Three Techniques Developed for Sampling and Analysis of Gunshot Residue by Scanning Electron Microscopy and Energy Dispersive X-Ray Analysis," Journal of Forensic Sciences, Vol. 37, 1992, pp. 281-300.
- 10.3.5.5 Garofano, L., Capra, M., Ferrari, F., Bizzaro, G. P., DiTullio, D., Dell'Olio, M., and Ghitti, A., "Gunshot Residue Further Studies on Particles of Environmental and Occupational Origin," Forensic Science International, Vol 103, 1999, pp. 1 – 21.
- 10.3.5.6 Mosher, P. V., McVicar M. J., Randall E. D., and Sild, E. H. "Gunshot Residue-Similar Particles Produced by Fireworks", Canadian Society of Forensic Science Journal, Vol. 31 (2) 1998, pp. 157-168.
- 10.3.5.7 Nesbitt, R. S., Wessel, J. E., and Jones, P. F., "Detection of Gunshot Residue by Use of the Scanning Electron Microscope," Journal of Forensic Sciences, Vol. 21, 1976, pp. 595-610.
- 10.3.5.8 Sild, E. H. and Pausak, S., "Forensic Applications of SEM/EDX," Scanning Electron Microscopy, Vol. 2, 1979.
- 10.3.5.9 Wolten, G.M., Nesbitt, R.S., Calloway, A.R., Loper, G.L. and Jones, P.F., "Final Report on Particle Analysis for Gunshot Residue Detection," Report ATR-77(7915)-3. The Aerospace Corp., Sept. 1977.
- 10.3.5.10 Wright, D., and Trimpe, M., "Summary of the FBI Laboratory's Gunshot Residue Symposium May 31- June 3, 2005", Forensic Science Communications, Vol. 8, (3) 2006.
- 10.3.5.11 Gunshot Residue V2.00 from 1.12 Documentation, Operator's Manual. R. J. Lee Instruments Limited. 515 Pleasant Valley Rd., Trafford, PA 15085.
- 10.3.5.12 Guide for Primer Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray. <http://www.swggsr.org/FINAL%20GUIDE11-29-11.pd>.

10.4 Documentation

- 10.4.1 Elements detected as potential primer residue particles and the diameter of the particles are documented on the Particle Summary Report. This report is generated by the operator after reviewing the electronic images and spectra of potential primer residue particles captured by the instrument during the automated analysis. Whenever possible 10 potential particles characteristic of and/or consistent with primer residue are selected for manual review.
- 10.4.1.1 Particle classifications on the Particle Summary Report are preliminary due to the short X-ray acquisition times during the automated run. The operator will indicate the actual particle classification on the Particle Summary Report after performing the manual review. Particles that are confirmed as characteristic of or consistent with primer residue are indicated with a check mark.

- 10.4.1.2 Additions or corrections to the Particle Summary Report are made by the examiner at the time of the manual review.
- 10.4.1.3 Any changes not made at the time of the manual review must be initialed and dated at the time of the change.
- 10.4.2 The original Primer Residue SEM/EDS Worksheet will be retained in the case file associated with the 1st case sample in the automated run.
- 10.4.3 A representative photograph and corresponding EDS spectrum for each confirmed primer residue particle is generated for the case file. EDS spectra should be obtained at a minimum of 1K counts full scale or 99 seconds Live Time whenever possible.
- 10.4.4 Confirmed particles from the representative particles are saved to the hard drive.
- 10.4.5 The Primer Residue Evidence Handling Worksheet is used to provide documentation for the results reported in the Certificate of Analysis. "Boiler plate" wording on the Worksheet that is not applicable may be struck through with a single line and the entire paragraph bracketed and initialed by the examiner.
- 10.4.6 The QA output must include a copy of the ENFSI 2008 proficiency sample map or positive control field before and after the casefile sample(s). A copy of the negative control must also be included in the QA binder.
- 10.4.7 An Analysis Summary Report for each of the case samples analyzed is also included in the case file.
- 10.4.8 The PRIMER RESIDUE ANALYSIS INFORMATION FORM that is provided in the Primer Residue kit is retained by the laboratory and kept in the case file.
- 10.4.9 The outside of the Primer Residue kit is photocopied and retained in the case file.
- 10.4.10 If the PRIMER RESIDUE ANALYSIS INFORMATION FORM indicates that the amount of time between the shooting event and the sampling for a living individual is in excess of 8 hours the samples will not be analyzed unless extenuating circumstances exist.

10.5 Cartridge Case Residue Examination

- 10.5.1 Purpose
 - 10.5.1.1 Occasionally potential primer residue particles may be encountered that have an unusual elemental composition that may be consistent with a particular type of ammunition or of a non-firearms source. In such cases it is advisable to compare cartridge case residue to particles found on the hands for elemental consistency. Because cartridge cases are frequently heavily loaded with primer residue, special precautions must be taken for this type of exam. The overall examination is described below and is similar to the examination described in Sections 10.3 and 10.4 with the following notable exceptions.
- 10.5.2 Minimum Standards and Controls
 - 10.5.2.1 Collection of residue should be done by the TE Section before the FX Section analyzes the cartridge cases.
 - 10.5.2.2 Evidence is opened on a cleaned, paper covered lab bench outside of the SEM room.
 - 10.5.2.3 Gloves are worn and changed between cartridge cases.

10.5.3 Analytical Procedures

- 10.5.3.1 The headstamp information is documented.
- 10.5.3.2 A bamboo skewer cleaned with methanol is used to scrape the inner sides of the cartridge case and primer cup.
- 10.5.3.3 The type of primer used, Boxer (one flash hole) or Berdan (two flash holes) is documented.
- 10.5.3.4 The cartridge case is inverted and tapped over top of an unused sampling device with protective covering removed.
- 10.5.3.5 Using fine forceps, the protective covering is replaced over the residue pressing it into the tape surface. The protective covering is then removed and discarded.
- 10.5.3.6 The sampling device is inverted and tapped over a trash can to remove any remaining loose particles.
- 10.5.3.7 The sampling device is labeled.
- 10.5.3.8 The cartridge case residue is examined in the ASPEX VP 2000 using a “Cartridge case” .afa and rule file appropriate for the number of cartridge case samples being examined. The ENFSI 2008 proficiency sample is not required; the positive control sample is sufficient. The automated examination is programmed to stop after 1000 particles have been examined on each sample. Cartridge case residue samples from only one FS lab # are run at one time.
- 10.5.3.9 Follow 10.4.1-10.4.7 for documentation.
 - 10.5.3.9.1 10 representative particles with their corresponding photos and spectra are saved for each type/manufacturer of primer encountered.
 - 10.5.3.9.2 A copy of the negative control is not included in the case file or QA binder as indicated in 10.4.6, as one is not necessary for cartridge case examination.
- 10.5.3.10 After the manual review is complete the samples are removed from the sample chamber and the sample chamber is detached from the SEM.
- 10.5.3.11 The sample chamber is thoroughly cleaned in a hood.
- 10.5.3.12 A “clean chamber check” negative control sample is examined in the ASPEX VP 2000 using a “clean chamber check” .afa and rule file. A copy of the “clean chamber check” data will be included in the QA binder. The Primer Residue Worksheet SEM/EDS for the clean chamber check is kept in the FS lab# file associated with the cartridge cases examined.

10.6 Report Wording

10.6.1 Primer Residue Kit / Inanimate Object Samples

10.6.1.1 Number of Particles

- 10.6.1.1.1 When one or two particles are found on the sample, the number and type (e.g., characteristic, consistent) will be listed as part of the results statement.
- 10.6.1.1.2 When three or more characteristic or consistent particles are found on a sample, it is not necessary to list the total number of particles found as part of the results statement.

10.6.1.1.3 If three or more characteristic particles are found on a sample, additional consistent particles found on the sample need not be reported.

10.6.1.2 To the maximum extent possible, report wording will be selected from the following:

10.6.1.2.1 Item _____ was examined using Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS).

Particles characteristic of primer residue are those that have a spherical or molten appearance (noncrystalline) and contain the elements lead, barium and antimony. Although rare, there are sources other than the discharge of a firearm that are capable of forming particles of this type.

Particles consistent with primer residue have the same appearance and typically include two of the three elements listed above. This type of particle is commonly associated with the discharge of a firearm but could also originate from numerous non-firearm sources.

10.6.1.2.2 No primer residue particles were found in the areas examined on the sample(s) in Item _____ marked right/left hand.

The absence of primer residue on the hands is consistent with an individual not having fired a weapon. A negative result could also occur from circumstances such as: washing the hands, wiping the hands, wearing gloves, sweating profusely, environmental factors including wind and rain, bloody hands, excessive debris on the sample, greater than 4 to 6 hours passing between firing and sampling, or the weapon not producing primer residue on the hands when discharged.

10.6.1.2.3 Two (2) particles characteristic of primer residue were found in the areas examined on the sample(s) in Item _____ marked right/left hand. One (1) particle consistent with primer residue was found in the areas examined on the sample(s) in Item _____ marked right/left hand.

Primer residue can be deposited on the hands by circumstances such as: firing a weapon, handling a weapon, being in the proximity to the discharge of a weapon or coming into contact with an object that has primer residue on it. The examination itself cannot determine the relative likelihood of these listed circumstances.

10.6.1.2.4 Particles characteristic of primer residue were found in the areas examined on the sample(s) in Item _____ marked right/left hand.

Primer residue can be deposited on the hands by circumstances such as: firing a weapon, handling a weapon, being in the proximity to the discharge of a weapon or coming into contact with an object that has primer residue on it. The examination itself cannot determine the relative likelihood of these listed circumstances.

10.6.1.2.5 Particles consistent with primer residue were found in the areas examined on the sample(s) in Item _____ marked right/left hand.

Primer residue can be deposited on the hands by circumstances such as: firing a weapon, handling a weapon, being in the proximity to the discharge of a weapon or coming into contact with an object that has primer residue on it. The examination itself cannot determine the relative likelihood of these listed circumstances.

- 10.6.1.2.6 Particles characteristic of primer residue were found in the areas examined on the sample(s) in Item _____ marked inanimate object 1/inanimate object 2.

Primer residue can be deposited on the surface of an inanimate object by circumstances such as: the object being in the proximity to the discharge of a weapon or coming into contact with another object that has primer residue on it. The examination itself cannot determine the relative likelihood of these listed circumstances.

- 10.6.1.2.7 It was indicated on the Primer Residue Analysis Information Form that the amount of time between the shooting event and the sampling for Item _____ was approximately _____ hours and _____ minutes. Item _____ was not analyzed. It should be noted that the optimal window of opportunity for sampling the hands of a living individual lasts for up to 4 to 6 hours after the shooting event.

- 10.6.1.2.8 The sample in Item _____ marked right/left hand was not necessary for examination.

10.6.2 Cartridge Case Residues

- 10.6.2.1 Report the type of particles found on the samples and the unusual elements observed. Compare and contrast them with the type of residue found in the spent cartridge case(s) associated with the case. Typical report wording examples are as follows:

- 10.6.2.1.1 Examples of an alleged .22 caliber shooting case:

The samples in Item _____ and residues collected from Items _____ were examined using Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS).

Particles characteristic of primer residue are those that have a spherical or molten appearance (noncrystalline) and contain the elements lead, barium and antimony. Although rare, there are sources other than the discharge of a firearm that are capable of forming particles of this type.

Particles consistent with primer residue have the same appearance and typically include two of the three elements listed above. This type of particle is commonly associated with the discharge of a firearm but could also originate from numerous non-firearm sources.

Particles characteristic of primer residue were found in the areas examined on the samples in Item _____ marked right hand and left hand. One (1) of the particles characteristic of primer residue found in the area examined on the sample in Item _____ marked left hand contained tin. One (1) particle consistent with primer residue containing lead and barium was found in the area examined on the sample in Item _____ marked right hand. Two (2) particles consistent with primer residue containing barium and aluminum were found in the area examined on the sample in Item _____ marked left hand.

Particles consistent with primer residue containing lead and barium were found in the residues collected from the Item _____ cartridge cases.

The majority of the primer residue population from the samples in Item _____ marked right hand and left hand was inconsistent with the primer residue composition found in the residues collected from the Item _____ cartridge cases.

Primer residue can be deposited on the hands by circumstances such as: firing a weapon, handling a weapon, being in the proximity to the discharge of a weapon or coming into contact with an object that has primer residue on it. The examination itself cannot determine the relative likelihood of these listed circumstances.

10.6.2.1.2 Examples of primer residue found on a victim's hands where three types of ammunition were involved:

The samples in Item _____ and residues collected from Items _____ were examined using Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS).

Particles characteristic of primer residue are those that have a spherical or molten appearance (noncrystalline) and contain the elements lead, barium and antimony. Although rare, there are sources other than the discharge of a firearm that are capable of forming particles of this type.

Particles consistent with primer residue have the same appearance and typically include two of the three elements listed above. This type of particle is commonly associated with the discharge of a firearm but could also originate from numerous non-firearm sources.

Particles characteristic of primer residue were found in the area examined on the sample in Item _____ marked left hand. Particles consistent with primer residue were found in the area examined on the sample in Item _____ marked right hand. It should be noted that particles containing zirconium, barium and tin with or without lead were found on both the right hand and left hand sampling devices. In addition, there were characteristic particles and consistent particles found that contained aluminum on both the right hand and left hand sampling devices.

Particles containing zirconium, barium and tin with or without lead were found in the residues collected from the Item _____ cartridge cases.

Particles characteristic of primer residue and consistent with primer residue, many containing aluminum, were found in the residues collected from the Item _____ cartridge cases.

Particles consistent with primer residue containing lead, barium, silicon, calcium and potassium with or without tin were found in the residues collected from the Item _____ cartridge cases. This type of particle was not observed in the areas examined on the samples in Item _____ marked right hand or left hand.

Primer residue can be deposited on the hands by circumstances such as: firing a weapon, handling a weapon, being in the proximity to the discharge of a weapon or coming into contact with an object that has primer residue on it. The examination itself cannot determine the relative likelihood of these listed circumstances.

11 LAMP FILAMENTS

11.1 Introduction

The primary purpose of a lamp filament analysis is to determine whether the lamp was lighted or unlighted at the moment of impact. Fortunately, the filaments of incandescent lamps often give the key to answering the question. The stresses generated on a filament by the accelerations and sudden stops of a collision produce deformation and fracture phenomena with characteristic differences between hot and cold filaments. Further evidence may arise if the glass bulb is broken during the accident. If the filament is hot, it will be oxidized and it may melt any broken glass that comes in contact with it.

11.1.1 The investigator may be able to provide information useful to the examination such as:

- All lamps located in the damaged area of the vehicle
- Accident reports and photographs depicting the damaged area(s) of the vehicle(s)
- The socket position on the vehicle from which the lamp is removed and the function of the lamp.

11.1.2 Lamps should be identified with a unique item number. Lamps should be described as to the type of lamp and, whenever possible, specify the specific location on the vehicle from which it was obtained. The terms “passenger” and “driver” side provide a clearer description than “right” and “left” side of the vehicle.

Example: #1 head lamp – passenger side, outside lamp
#2 parking lamp – driver side

11.1.3 Lamp examinations can also be conducted on other types of vehicles and objects. Bicycles, farm tractors, caution signs, traffic lights and the like may yield evidence helpful to an investigation. Any lamp having a tungsten filament, which has been involved in a collision or has been impacted, may yield some helpful information; however, there may not always be sufficient information to make a definitive conclusion.

11.2 Safety Considerations

Examiner should:

- Use caution in examining lamps and testing circuits.
- Never look with the naked eye directly into a lamp which is lighted. Caution should be used as the light emitted during white heat can injure the eyes.
- Ordinary sunglasses are not dark enough. Use a piece of exposed film.
- Broken bulbs have very sharp edges and can produce severe cuts.
- Electrical circuits may be energized and could produce electrical shocks.

11.3 Equipment

- Stereomicroscope
- Comparison microscope
- Photographic equipment
- Charcoal starter
- Scribe
- A volt-ohm meter (multimeter) to measure resistance of the wire
- Batteries or a 6 and/or 12 volt battery charger for testing reference lamps and/or for experimentation
- Hand tools
- Other equipment as needed

11.4 Minimum Standards and Controls

Known lamp standards may be used for comparisons. These lamps may be purchased from any retail lamp distributor.

All reported vehicle lamp conclusions will be verified by an examiner qualified to perform technical review of the case file who will conduct the verification from the original evidence. The examiner must view the lamps microscopically as part of the verification. The examiner's case file notes will be initialed and dated by the second examiner who will include a notation to indicate that the vehicle lamp conclusion was verified.

11.5 Procedure

11.5.1 The evidence will be marked in accordance with the QM. A systematic approach should be used for the vehicle lamp examination, with findings and observations recorded in the notes.

11.5.2 General Lamp Examination Procedure

11.5.2.1 Classify lamp as to the brand, type, base arrangement, electrical rating (voltage/wattage), and purpose in vehicle, if possible.

11.5.2.2 Document the physical appearance of the lamp through photographs or sketches and notes, to include the following:

- physical condition of the glass (breakage/discoloration).
- physical condition of the base (type of metal, markings present).
- physical appearance of the filament(s) to include the amount of distortion/stretching
- presence or absence of any discoloration of the metal, oxide deposits
- presence or absence of age sag, pitting of the metal
- presence of cold fracture
- presence of molten or melted glass
- any other physical phenomena observed

11.5.2.3 It may be necessary to open the lamp, as described in ¶ 11.5.3 or 11.5.4.

11.5.2.4 Prior to using the volt-ohmmeter, verify proper operation by placing the volt-ohmmeter in the resistance mode to measure continuity. Touch probes together and verify a reading of "0". If the volt-ohmmeter reads "0" it may be used to test for continuity in lamp filaments. Document in case notes that the volt-ohmmeter is functioning properly.

11.5.2.5 If observations are inconsistent, it may be advantageous to use comparison lamps to show similarities in lamp configurations and conduct experiments to simulate characteristics observed in the evidence.

11.5.3 Small Lamp Examination Procedure

Opening small lamps is a relatively simple procedure, but care must be used with broken glass. Continuity testing with a volt-ohm meter shall be conducted before opening any lamps.

11.5.3.1 Wear protective eye coverings

11.5.3.2 Using a scribe, etch the bulb to stress the glass.

11.5.3.3 Place a piece of plastic wrap around the glass.

11.5.3.4 Place the lamp bulb right side up in the jaws of a vise, while holding onto the base of the lamp.

- 11.5.3.5 Slowly and gradually close the vise jaws on the glass bulb until the pressure exerted on the glass causes it to break.
- 11.5.3.6 Carefully remove the lamp from the vise.
- 11.5.3.7 Turn the lamp upside down over a piece of paper to catch any material that has fallen into the base.
- 11.5.3.8 Remove any sharp edges of glass with a pair of pliers.
- 11.5.3.9 Compare the current condition of the filament to how it appeared prior to opening the lamp.
- 11.5.3.10 An alternate method of opening a small lamp may be utilized using a glass tube cutter. Care must be taken to reduce the possibility of damaging the filaments and internal lamp parts.
- 11.5.3.11 Record the method of opening, findings and observations in the notes by documenting and/or photographing.

11.5.4 Sealed Beam Lamp Examination Procedure

The following method has been used successfully for opening sealed beam lamps, and is preferred over tapping methods as it reduces the possibility of damaging the filaments.

- 11.5.4.1 Using a carbide scribe, make a score mark around the lugs on the reflector in a circular pattern. It is helpful to use a circular pattern that coincides with the charcoal starter's radius.
- 11.5.4.2 Hold the hot charcoal starter loop on the scored circle.
- 11.5.4.3 A drop of water may be placed where the reflector and the charcoal starter meet. The reflector should crack along the scored line with a distinct click.
- 11.5.4.4 Rotate the lamp slowly to continue the crack around the scored line.
- 11.5.4.5 Once the crack has gone completely around the reflector, remove the heat.
- 11.5.4.6 Grasp a lug with pliers and tap the reflector on the scored, cracked line with a small hammer or pliers to separate the part of the reflector attached to the filaments from the lens.

11.6 Interpretation of Results and Reporting

All interpretations of results to include lamp description, observations and conclusions shall be recorded in the notes.

- 11.6.1 Description of the lamp: This gives information about the lamp as to its type and description. It may also offer the position of the lamp as it was in the vehicle.
- 11.6.2 Techniques: Techniques utilized in the examination shall be listed on the report.
 - 11.6.2.1 Item _____ was examined visually and using stereomicroscopy and compound microscopy.
 - 11.6.2.2 Item _____ was examined using a multimeter, stereomicroscopy, compound microscopy, and Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry (SEM-EDS).
- 11.6.3 Observations: In the body of the "Certificate of Analysis" under the "Results" heading, the appearance of the lamp, the functionality of the filament(s) and both normal and abnormal characteristics are reported

- 11.6.3.1 Items ___ and ___ are dual filament, dual purpose lamps. Generally, when used in the rear of a vehicle, one filament serves as the taillight and the other filament serves as the brake, turn indicator, and/or hazardous warning signal.

Examination of Item ___ revealed the glass envelope to be intact and the filaments to be operational. The physical appearance of each filament was considered normal.

Examination of Item ___ revealed the glass envelope to be intact and slightly darkened due to age. The filaments were operational and exhibited stretching and distortion.

- 11.6.3.2 The Item ___ type 3457AK lamp is a dual filament, dual purpose lamp with operational filaments. Generally, when this lamp is used in the front of the vehicle, one filament serves as the parking light and the other filament serves as the front turn signal. The parking light filament exhibited significant distortion and stretching. The turn signal filament was considered normal in appearance.

- 11.6.3.3 Item ___ contained a damaged portion of a sealed beam headlamp. The Item ___ lamp is a dual filament, dual purpose lamp. Generally, when this lamp is used in the front of the vehicle, one filament serves as the low beam and the other filament serves as the high beam.

The Item ___ glass envelope was broken and missing with only pieces of the reflector remaining. The support posts were bent and damaged, therefore, the filaments are not in normal operating condition. The low beam filament was distorted, mechanically stretched, and discolored with molten and adhering glass. The low beam filament was fractured with tapered and/or rounded ends in two locations. The high beam filament was separated from the support posts and was flattened, blackened, and fractured.

- 11.6.3.4 Item ___ consisted of a [light-emitting diode (LED) / high-intensity discharge (HID)] lamp. [LED / HID] lamps do not contain filaments and therefore are not suitable for on/off determination.

- 11.6.3.5 When a filament is continuous, but envelope conditions would cause burnout:

Examination of the [Item#] lamp revealed it to be capable of conducting electrical current; however, the lamp is not operational due to the condition of its envelope.

- 11.6.4 Conclusions: Additionally, in the “Results” section of the report, the conclusions, based on the observations reported, are stated.

- 11.6.4.1 Examination of the [Item#] lamp revealed physical characteristics consistent with it having been lighted when it was subjected to impact shock and the glass envelope was broken (damaged). See Criterion I

- 11.6.4.2 Examination of the [Item#] lamp revealed physical characteristics consistent with it having been lighted when it was subjected to impact shock (damaged). See Criterion II

- 11.6.4.3 Examination of the [Item#] lamp revealed physical characteristics consistent with it not having been lighted when it was subjected to impact shock (damaged). See Criterion III

- 11.6.4.4 Examination of the [Item#] lamp revealed insufficient distortion/stretching to definitively determine the “on” or “off” condition at the time of vehicle impact. (damaged). See Criterion IV

- 11.6.4.5 Examination of the [Item#] lamp revealed it to be normal, and therefore it was not possible to determine the “on” or “off” condition at the time of vehicle impact. (lamp is normal, with no apparent damage) See Criterion IV

11.6.4.6 General Criteria for Lamp Examination Conclusions

11.6.4.6.1 Criterion I: The bulb was "on" ... (glass broken).

Physical characteristics would include an oxidized filament (discolored), possibly with beaded ends, some degree of filament distortion, possibly molten or welded glass on the filaments, possibly white tungsten oxides present on the inner surface of the bulb and/or on the colder surfaces within the bulb (posts, other filament); note that mechanical stretching may occur when glass is broken, so at least one of the other characteristics should be present to make this determination.

11.6.4.6.2 Criterion II: The bulb was "on" ... (glass not broken).

Physical characteristics include some significant degree of distortion and/or stretching of one or both filaments.

11.6.4.6.3 Criterion III: The bulb was not "on" ...

The filament has been cold fractured or there is a normal burnout. The absence of physical characteristics such as oxidation of the filament, melted glass, filament distortion cannot definitively determine the "off" condition, when the glass is broken, although these characteristics would be expected to be observed if a lamp was lighted at time of impact and the glass was broken. The absence of the characteristics associated with 11.6.4.6.1 and 11.6.4.6.2 above are only an indication that the bulb was not lighted at the time that the glass was broken.

11.6.4.6.4 Criterion IV: Insufficient characteristics to determine the "on" or "off" condition.

The lamp appears normal, or there is insufficient distortion to make a determination.

11.7 Full Report Wording Examples

11.7.1 Example 1

Item 1	One (1) 1157 lamp
Item 2	One (1) 2057 lamp

RESULTS:

Items 1 and 2 were examined visually and using stereomicroscopy, compound microscopy, and a multimeter.

Items 1 and 2 are dual filament, dual purpose lamps. Generally, when used in the rear of a vehicle, one filament serves as the taillight and the other filament serves as the brake, turn indicator, and/or hazardous warning signal.

Examination of Items 1 and 2 revealed the glass envelopes were intact. The brake/turn/hazard filament in each lamp was operational. The taillight filament in Item 1 was operational but the taillight filament in Item 2 was not operational due to cold fracture. The taillight filaments exhibited slight distortion, however, less distortion than that exhibited by the brake/turn/hazard filaments.

The physical appearance of these lamps was consistent with the brake/turn/hazard filaments having been lighted and the taillight filaments not having been lighted when, at some time, these lamps were subjected to impact shock.

11.7.2 Example 2

Item 1A	One (1) type 3057 dual filament tail lamp from top right position rear of vehicle
Item 1B	One (1) type 194 single filament marker lamp from center right position rear of vehicle
Item 1C	One (1) type 3057 dual filament backup lamp from bottom right position rear of vehicle
Item 1D	One (1) type 3057 dual filament tail lamp from top left position rear of vehicle

RESULTS:

Items 1A, 1B, 1C, and 1D were examined visually and using compound microscopy.

Examination of the above listed lamps revealed the following:

Item 1A – The glass envelope was intact and darkened due to age. Both the taillight filament and the brake/turn filament were operational. The taillight filament exhibited stretching and distortion. The brake/turn filament was considered normal in appearance. The physical appearance of Item 1A was consistent with the taillight having been lighted when, at some time, this lamp was subjected to impact shock. It was not possible to determine the “on” or “off” condition of the brake/turn filament at the time of vehicle impact.

Item 1B – The glass envelope was intact and darkened due to age. The filament was operational and exhibited stretching and distortion. The physical appearance of Item 1B was consistent with this lamp having been lighted when, at some time, it was subjected to impact shock.

Item 1C – The glass envelope was intact. Both filaments in this lamp were operational. The physical appearance of each filament was considered normal. It was therefore not possible to determine the “on” or “off” condition of this lamp at the time of vehicle impact.

Item 1D – The glass envelope was intact and darkened due to age. The taillight filament and the brake/turn filament were both operational. The taillight filament exhibited distortion which is consistent with it having been lighted when, at some time, this lamp was subjected to impact shock. The brake/turn filament exhibited some distortion which is indicative that this filament could have been lighted; however, the degree of the distortion was insufficient to definitively determine that this filament was lighted when, at some time, this lamp was subjected to impact shock.

11.7.3 Example 3

Item 6	One (1) type 1157 lamp
Item 7	One (1) type 1157 lamp

RESULTS:

Items 6 and 7 were examined visually and using stereomicroscopy.

Item 6 and 7 are Chinese manufactured type 1157 dual filament, dual purpose lamps. Generally, when used in the rear of the vehicle, one filament serves as the taillight and the other filament serves as a brake, turn indicator, and/or hazardous warning.

Examination of Item 6 revealed the glass envelope to be broken, and for the most part, missing. The filaments were not in normal operating condition. The support posts were damaged. The taillight filament exhibited stretching, distortion, oxidation, molten glass, fused glass, and a fracture. The brake/turn/hazardous warning filament exhibited stretching, distortion, and mild oxidation.

Examination of Item 7 revealed the glass envelope to be intact and the filaments to be operational. The taillight filament exhibited stretching and distortion. The physical appearance of the brake/turn/hazardous warning filament was considered normal.

The physical characteristics of Items 6 and 7 taken together was consistent with the taillights having been lighted and the brake/turn/hazard filaments not having been lighted when, at some time, these lamps were subjected to impact shock and the glass envelope of Item 6 was broken. The Item 6 taillight filament was subsequently fractured.

11.8 References

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12 FOURIER TRANSFORM INFRARED SPECTROPHOTOMETER (FTIR)**12.1 Overview**

The QC checks will be performed prior to using the instrument and any accessories for casework. A sample log will be maintained that includes at a minimum the date, the initials of the examiner, the FS Lab# or other identifying information and the method of analysis: Bench, Microscope or ATR. Every six months, copy data files to a writeable CD/DVD or the Department intranet and retain.

12.2 Bench QC

12.2.1 Day-of-Use

- 12.2.1.1 The bench shall be aligned using the “Align Bench” function. Place a checkmark in the respective column in the logbook to record when this is done. If the bench has previously been aligned during the given week, the bench does not need to be re-aligned.
- 12.2.1.2 Run Val-Pro iS50 System Qualification Test. This test checks a number of different performance characteristics including collecting polystyrene and NG11 glass spectra, checking the peak-to-peak noise using samples mounted on a wheel, which is replaced prior to expiration of its calibration certificate. If all tests do not pass, troubleshoot and re-run Val-Pro. Do not proceed until this issue is resolved. All copies of the Val-Pro report, including those with notations of “Fail”, will be generated, initialed and retained. Record the date and initials of the person performing the test in the logbook.

12.3 Microscope Accessory QC

12.3.1 Day-of-Use

- 12.3.1.1 Bench QC procedures shall be performed prior to using this accessory.
- 12.3.1.2 All of the lights (transmittance, reflectance and aperture) will remain on and the detector will be cooled for a minimum of thirty minutes prior to conducting the QC checks. Insulated gloves and safety glasses shall be worn when filling the transport Dewar as well as the instrument reservoir.
- 12.3.1.3 The alignment of the optical path of the microscope shall be checked and corrected as necessary.
- 12.3.1.4 The noise level of the scope will be measured at the 2700-2600 cm^{-1} region of the spectrum with the following instrument parameters:
- Scans = 200
 - Resolution = 4 cm^{-1}
 - Gain = 1.0
 - Optical velocity = 1.8988
 - Aperture = 32

For the MCT-A detector, the Peak-to-Peak noise value should be $< 0.041\%T$, if not, perform maintenance. The noise value will be recorded in the log book, along with the date and initials of the person performing the test.

For the MCT-B detector, the Peak-to-Peak noise value should be $< 0.264\%T$, if not, perform maintenance. The noise value will be recorded in the log book, along with the date and initials of the person performing the test.

12.3.1.5 A spectrum of the polystyrene test film will be taken with the following instrument parameters:

- Scans = 128
- Resolution = 4 cm^{-1}
- Gain = 1.0
- Optical velocity = 1.8988
- Aperture = 32

The value of the 1601 cm^{-1} peak will be recorded in the log book, along with the date and initials of the person performing the test. If the value has changed $\pm 0.2 \text{ cm}^{-1}$ from the most recent value obtained, then troubleshoot and re-analyze the polystyrene test film. Do not proceed until this issue is resolved. A copy of the polystyrene spectrum with wavenumbers displayed to two decimal places will be generated, initialed and retained.

12.4 ATR Accessory QC

12.4.1 Integrated iS50 ATR

Day-of-Use

12.4.1.1 Bench QC procedures shall be performed prior to using this accessory.

12.4.1.2 Run the Val-Pro ATR Qualification Test. This test checks peak location and optical resolution using a polystyrene standard, which is replaced prior to calibration certificate expiration. If all tests do not pass, troubleshoot and re-run Val-Pro. Do not proceed until this issue is resolved. All copies of the Val-Pro report, including those with notations of "Fail", will be generated, initialed, and retained. Record the date and initials of the person performing the test in the logbook.

12.4.1.3 Clean the diamond crystal and anvil surface before and after analysis.

12.4.2 Golden Gate ATR

Day-of-Use

12.4.2.1 Bench QC procedures shall be performed prior to using this accessory.

12.4.2.2 When the ATR accessory is used instead of the integrated iS50 ATR, it must be placed into the bench sample compartment, and the throughput checked by testing the Smart Accessory. The system must display a check mark to proceed.

12.4.2.3 Clean the diamond crystal and anvil surface before and after analysis.

12.4.2.4 A spectrum of the backing of a designated adhesive tape will be taken with the following ATR instrument parameters:

- Scans = 32
- Resolution = 4 cm^{-1}
- Gain = Autogain
- Optical velocity = 0.4747

Print the spectrum with no ATR correction with wavenumbers displayed to zero decimal places (units) and compare to that previously obtained with the accessory, looking at any significant changes to the %Reflectance, the position of the peaks and the resolution of peaks. If there is a significant change in the data, perform maintenance. The printed spectrum of the tape backing will be initialed and retained.

12.5 Casework Analysis

12.5.1 Introduction

- 12.5.1.1 The spectra for samples are compared to other samples (K and Q) or to standards. The resolution of standard reference spectra will be equal to or better than the resolution of the sample spectra. Data will be displayed to zero decimal places (units).
- 12.5.1.2 If any meaningful differences are observed in the spectra of the questioned and known sample, the examiner may discontinue the analysis.
- 12.5.1.3 A new background spectrum shall be collected either before or after every casework sample.
- 12.5.1.4 Data manipulation, including baseline correction, is discouraged and smoothing of data is prohibited. If baseline correction is conducted, a copy of the original, uncorrected spectrum will be included in the case file.
- 12.5.1.5 While the examiner must always carefully assess the sampling method selected and the data obtained, the examiner must be particularly cautious when using the ATR Accessory. ATR is a surface analysis technique and, as such, may not be suitable for all types of samples. Paint samples require particular attention as the surface spectrum may differ from the spectrum of the individual layer when using the microscope accessory. Additionally, it is important that the anvil not touch the crystal surface or interference bands from the anvil crystal will appear in the data. For this reason, many fibers do not have a large enough diameter to be analyzed via the ATR Accessory. Assess the %R and if it is poor, the sample, as analyzed, may not be covering a sufficient amount of the ATR crystal. If this is the case, either use more sample and re-analyze or select a different sampling method.
- 12.5.1.6 Sample preparation techniques are dependent on the type of sample to be analyzed. The most frequently used sample preparation techniques are listed below.

12.5.2 Sample Preparation

12.5.2.1 Pellet Technique

- 12.5.2.1.1 Infrared grade KBr must be kept dry by storing it in a desiccator.
- 12.5.2.1.2 Infrared grade KBr and the sample each must be finely ground. The KBr and sample are mixed by grinding with a mortar and pestle in an approximate ratio of 100 parts KBr to 1 part sample.
- 12.5.2.1.3 The mixture is placed in a pellet press to prepare the pellet.
- 12.5.2.1.4 Alternatively, a KBr-only pellet may be pressed or a commercially available KBr window may be used to place a drop of sample in solvent onto the KBr. The solvent is evaporated and the sample is analyzed as a thin film on KBr.

12.5.2.2 Microcompression Cell with Diamond Windows

- 12.5.2.2.1 Clean the diamond windows before and after analysis.
- 12.5.2.2.2 Place the sample between the windows and hand tighten the cell to compress the sample.
- 12.5.2.2.3 To decrease the interference fringes seen in the spectra, KBr or AgCl may be placed between the diamond windows, compressed with the sample and used to

obtain the background spectrum. Alternatively, one window may be removed after compression and before analysis.

- 12.5.2.2.4 Ensure that the appropriate setting on the objective and/or condenser compensation ring(s) is used. After making adjustments to the compensation ring(s), it is important to check the optical path alignment as manipulation of the compensation ring(s) may shift the alignment.

12.5.2.3 ATR Accessory

- 12.5.2.3.1 Clean the diamond crystal and anvil surface before and after analysis.
- 12.5.2.3.2 For solid samples, cover the diamond portion of the crystal with sample. Press the anvil against the sample by turning the anvil screw clockwise until it spins without further tightening. Exercise caution during this maneuver as with some samples (e.g., polystyrene bead) the anvil screw may not spin freely and attempts to further tighten it may result in damage to the accessory.
- 12.5.2.3.3 For liquid samples, place a drop or two of liquid directly onto the ATR crystal using enough sample to cover the crystal completely. If the sample is volatile, place the cover over the sampling area to prevent evaporation during analysis. The anvil is not used during analysis.
- 12.5.2.3.4 If the sample requires an extraction, the sample in an organic solvent may be dropped onto the crystal and allowed to evaporate to form a film. The bridge is not lowered during analysis. An o-ring may be used to contain the liquid as it is placed on the crystal.
- 12.5.2.3.5 Samples are compared to other samples (K and Q) or to standards by comparing the raw ATR data of each. However, the ATR correction should be performed prior to searching libraries containing transmission spectra.

12.5.2.4 Reflectance

- 12.5.2.4.1 Clean the first order mirror slide before and after analysis.
- 12.5.2.4.2 Place the sample on the mirrored slide paying attention to which side is first-order.
- 12.5.2.4.3 Using the 'Scope Transmittance 128' experiment file, open the Experiment Setup menu. In the Bench tab, change the sample compartment option from 'μScope %T' to 'μScope %R'.
- 12.5.2.4.4 Change the Continuum microscope mode to Reflection using the buttons on the base of the microscope.
- 12.5.2.4.5 Focus on the sample surface and collect the sample spectrum.
- 12.5.2.4.6 Move onto the mirror and collect the background spectrum.

12.5.2.5 Vapor Technique

Standard gas cells are used for vapor samples. Evacuate the gas cell, by pulling room air through, and obtain the background spectrum. Fill the gas cell and obtain the sample spectrum. "Clean" the gas cell by evacuating it following sample analysis.

12.6 Instrumental Conditions

12.6.1 The following instrumental conditions are used unless otherwise noted in the case file documentation.

Experiment	Bench	Scope		ATR
Detector:	DTGS	MCT-A	MCT-B	DTGS
Spectral range (cm ⁻¹):	4000-400	4000-650	4000-400	4000-400
Beam splitter:	KBr	KBr	KBr	KBr
Source:	IR	IR	IR	IR
Gain:	Auto	Auto	Auto	Auto
Optical velocity (max.):	0.4747	1.8988	1.8988	0.4747
Aperture:	100	32	32	230
Resolution (cm ⁻¹):	4	4	4	4
Number of sample scans:	32	128	300	32
Number of background scans:	32	128	300	32
Background collection:	Before Sample	After Sample	After Sample	Before Sample
Final Format:	% Transmittance	% Transmittance	% Transmittance	% Reflectance
Correction & Attenuation:	None	None	None	None
Zero filling:	None	None	None	None
Apodization:	Happ-Genzel	Happ-Genzel	Happ-Genzel	Happ-Genzel
Phase correction:	Mertz	Mertz	Mertz	Mertz

12.7 Maintenance

12.7.1 Maintenance and/or repairs to the FTIR system, computer or peripherals will be performed as needed and summarized in the log book to include the date and initials of the person making the entry.

12.8 Documentation

12.8.1 FTIR spectra will be printed as follows:

- Split at 2000 cm⁻¹
- X-axis limits will be appropriate to the experiment spectral range listed above
- The spectrum will occupy the maximum amount of space on the page as possible
- One spectrum per page

12.8.2 FTIR spectra will be labeled as follows:

- FS Lab #, Item # and how prepared (e.g., diamond cell, KBr pellet, liquid, vapor, etc.)
- Detector type (i.e., MCT-A vs. MCT-B) should also be noted, if more than one is available
- Note if acquired with the bench or microscope (this may appear in the case notes in lieu of on the individual spectrum)
- Date and time
- Filename (optional)

13 GAS CHROMATOGRAPH (GC)

13.1 Materials

13.1.1 Capillary Columns

- 13.1.1.1 All routine methods use flexible fused silica capillary columns containing a bonded, cross-linked stationary phase. The stationary phase is chosen to affect needed resolution. Examples include 5% phenylmethyl silicone (i.e., HP-5) and polyethylene glycol (i.e., DBWAX). The normal general purpose column has a 0.25 µm film thickness and 0.25 mm internal diameter.
- 13.1.1.2 For some general chemical examinations, it may be necessary to use an alternative column for specialized analysis or if more resolving power is required, or an additional, different diameter and/or phase column can be temporarily attached at the end of the existing column by using an appropriate connector. Guidance from the instrument operator is recommended.

13.1.2 Additional Instrument Parameters

- 13.1.2.1 The carrier gas is normally high purity helium at a flow rate of 0.5 to 3 mL/min.
- 13.1.2.2 When using the FID detector, helium makeup gas is supplied in order to support gas flow to provide optimal detector sensitivity.
- 13.1.2.3 Split/splitless liners designed specifically for use with the particular instrument should be used.

13.2 Maintenance

- 13.2.1 The GC septum and injection port glass liners are changed as needed.
- 13.2.2 If using a regulator, the pressure of the compressed gas cylinder providing research grade helium to the system should be checked and the tank changed as needed (at a minimum of ~250 psi).
- 13.2.3 If using a hydrogen generator, the water level is checked and water is added to approximately the fill line as needed.
- 13.2.4 Gas line moisture traps and oxygen scrubbers are changed as needed.
- 13.2.5 Record any maintenance performed in the instrument logbook, date and initial.
- 13.2.6 Data files shall be copied to long term storage media or the Department intranet annually, at a minimum. Files in long term storage shall be retained for at least six years.
- 13.2.7 When noticeable changes (i.e., decreased resolution, decreased sensitivity) occur in the instrument's performance as noted in check samples or standard samples, the following items shall be checked as necessary: septum, injection port liner, column nut, column, and autosampler. Any maintenance or repair will be recorded in the instrument logbook.

13.3 QC Procedures

13.3.1 Day-of-Use

- 13.3.1.1 The Polar Check Sample (PCS) is injected to monitor the resolution and sensitivity of the instrument when a polar column is installed. For non-polar columns, the Resolution Test Mixture (RTM) is injected to monitor the resolution and sensitivity of the instrument.

13.3.1.2 Polar Check Sample (PCS) QC mix: (Polar Columns)

To 300 mL of carbon disulfide, add 3 drops each of ethyl benzene, limonene, α -pinene, β -pinene, toluene, 1,2,4-trimethylbenzene, *o*-xylene, *m*-xylene, and *p*-xylene.

13.3.1.3 Resolution Test Mixture (RTM): (Non-polar Columns)

1,2,4-Trimethylbenzene, *m*-Ethyltoluene, *o*-Ethyltoluene, *p*-Xylene, Toluene, n-Hexane (C6), n-Octane (C8), n-Decane (C10), n-Dodecane (C12), n-Tetradecane (C14), n-Hexadecane (C16), n-Octadecane (C18), n-Eicosane (C20)

13.3.1.4 Acceptance Criteria:

13.3.1.4.1 Polar Check Sample: The check sample chromatogram is compared to previous ones, if available, for relative sensitivity, resolution, and baseline profile. The abundance from the baseline to the valley between the *p*-xylene and *m*-xylene peaks must be no greater than approximately 10% of the abundance of the weaker peak. Retention times must be within 0.033 minutes from the previous PCS check unless extenuating circumstances exist. If the check sample chromatogram is not acceptable, do not use the instrument until corrected.

13.3.1.4.2 Resolution Test Mixture: The retention times for the 1,2,4-Trimethylbenzene (1,2,4-TMB) peak and the hexadecane (C16) peak must be within 0.033 minutes from the previous RTM QC check.

13.3.1.5 Check sample chromatograms are retained for six years.

13.3.1.6 Whenever a new column is installed, it shall be checked with an appropriate check sample.

13.4 Analysis of Casework Samples

13.4.1 Routinely, 1 μ L injection volumes are used. However, injection volumes may range from a needle dip to 3.0 μ L for diluted standards and case samples and up to 2.0 mL for headspace samples. Samples may be concentrated to avoid injecting higher volumes.

13.4.2 It is encouraged that general chemical case samples be characterized as much as possible prior to injection into the instrument.

13.4.2.1 Samples may be diluted or dissolved in an appropriate solvent.

13.4.2.2 Depending on the nature of the sample, some samples must be cleaned up by extraction.

13.4.2.3 Some compounds do not chromatograph well, requiring derivatization to help effect good chromatographic peak shape.

13.4.3 Standards/references used in the comparison shall be run on the same day as the sample whenever possible, except for fire debris. "Same day" is defined as an approximate 24 hour period. Circumstances may exist where the sample and reference cannot be run on the same day; under these circumstances, the Section Supervisor may approve this minor deviation.

13.4.4 Appropriate standards/reference samples and blanks shall be run.

13.4.4.1 The blank, standard, and sample must be run under the same chromatographic conditions and data acquisition parameters, as appropriate.

13.4.4.2 A blank, run prior to each sample, shall be appropriate for the sample being run, i.e., an appropriate solvent or the solvent(s) used to dissolve or dilute the case samples, room air for

headspace injections, or the clean needle to be used for needle dip scenarios. Additional blanks may be run at the examiner's discretion.

- 13.4.4.3 When running multiple samples, a blank shall be run between sample injections. An extraction blank may be used as a blank that is run between sample injections.
- 13.4.5 The date, the initials of the examiner, the FS Lab#, sequences and/or samples (including standards) are maintained in a sample log.
- 13.4.5.1 Sequencing via autosampler should be utilized whenever practical.
- 13.4.5.2 If individual samples are not recorded in the logbook, completed sequence logs shall be printed and retained.
- 13.4.6 Integrated retention times for analytes must agree with the standard within 0.033 minutes.

13.5 Documentation

- 13.5.1 Sample chromatograms are compared to known standard chromatograms according to retention times and overall patterns.
- 13.5.2 To include in the case file:
- 13.5.2.1 Chromatogram of blank showing the entire range demonstrating that there are no interfering peaks in the region of interest.
- 13.5.2.2 Chromatograms of both sample and standard with integrated retention times.
- 13.5.2.3 If a method other than those listed in 13.6 is used, document the method parameters used (e.g., print the method).
- 13.5.3 The minimum requirements for labeling data is as follows:
- FS Lab #
 - Item Number
 - Examiner's handwritten initials
 - Injection volume, unless noted in the GC-FID method below
 - Injection solvent

13.6 Generally Used GC-FID Methods

- 13.6.1 Using autosampler

Method Name	PAUTO1-50-240	PAUTO2-20-240	PAUTO1-50-160	PAUTO2-20-160
Injection Type	1 µL autosampler	2 µL autosampler	1 µL autosampler	2 µL autosampler
Column	Carbowax	Carbowax	Carbowax	Carbowax
Oven Parameters	40°C (2 min hold) Ramp 15°C/min to 240°C (0.67 min hold)	40°C (2 min hold) Ramp 15°C/min to 240°C (0.67 min hold)	40°C (2 min hold) Ramp 15°C/min to 160°C (0.5 min hold)	40°C (2 min hold) Ramp 15°C/min to 160°C (0.5 min hold)
Inlet/Detector Temp	235°C / 245°C	235°C / 245°C	235°C / 245°C	235°C / 245°C
Column Flow	1.8 mL/min	1.8 mL/min	1.8 mL/min	1.8 mL/min
Split Ratio	Split 50:1	Split 20:1	Split 50:1	Split 20:1

13.6.2 Manual injections

Method Name	PMAN1-50-240	PMAN2-20-240	PMAN1-50-160	PMAN2-20-160
Injection Type	Manual	Manual	Manual	Manual
Column	Carbowax	Carbowax	Carbowax	Carbowax
Oven Parameters	40°C (2 min hold) Ramp 15°C/min to 240°C (0.67 min hold)	40°C (2 min hold) Ramp 15°C/min to 240°C (0.67 min hold)	40°C (2 min hold) Ramp 15°C/min to 160°C (0.5 min hold)	40°C (2 min hold) Ramp 15°C/min to 160°C (0.5 min hold)
Inlet/Detector Temp	235°C / 245°C	235°C / 245°C	235°C / 245°C	235°C / 245°C
Column Flow	1.8 mL/min	1.8 mL/min	1.8 mL/min	1.8 mL/min
Split Ratio	Split 50:1	Split 20:1	Split 50:1	Split 20:1

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14 GAS CHROMATOGRAPH-MASS SPECTROMETER (GC-MS)**14.1 Materials**

14.1.1 Capillary Columns

- 14.1.1.1 All routine methods use flexible fused silica capillary columns containing a bonded, cross-linked stationary phase. The stationary phase is chosen to affect needed resolution. Examples include methylsilicone (i.e., HP-1MS) and 5% phenylmethyl silicone (i.e., HP-5). The normal general purpose column has a 0.25 μm film thickness and 0.25 mm internal diameter.
- 14.1.1.2 Fire Debris cases shall be examined using a HP-1MS column or equivalent, 30 m X 0.25 mm ID X 0.25 μm film thickness.
- 14.1.1.3 For some general chemical examinations, it may be necessary to use an alternative column for specialized analysis or if more resolving power is required, or an additional, different diameter and/or phase column can be temporarily attached at the end of the existing column by using an appropriate connector. Guidance from the instrument operator is recommended.
- 14.1.1.4 The carrier gas is normally high purity helium at a flow rate of 0.5 to 3 mL/min.
- 14.1.1.5 Split/splitless liners designed specifically for use with the particular instrument should be used.

14.2 Maintenance

- 14.2.1 GC septum and injection port glass liners are changed as needed.
- 14.2.2 If using a regulator, the pressure of the compressed gas cylinder providing research grade helium to the system should be checked and the tank changed as needed (at a minimum of ~250 psi).
- 14.2.3 Gas line moisture traps and oxygen scrubbers are changed as needed.
- 14.2.4 Record any maintenance performed in the instrument logbook, date and initial.
- 14.2.5 When noticeable changes (i.e., air leaks, decreased resolution, decreased sensitivity) occur in the instrument's performance as noted in the autotune, standard samples, or check samples, the following items shall be checked as necessary: septum, injection port liner, column nut, GC-MS interface nut, column, autosampler, and MS source. Any maintenance or repair will be recorded in the instrument logbook.
- 14.2.6 Data files shall be copied to long term storage media or server monthly. Method and macro files shall be copied to long term storage or server semi-annually, at a minimum. Files in long term storage shall be retained for at least six years.

14.3 QC Procedures

14.3.1 Day-of-Use

- 14.3.1.1 The GC-MS shall be tuned using autotune (ATUNE) with perfluorotributylamine (PFTBA).
 - 14.3.1.1.1 The peak width at half height (pW50) will be changed from the factory setting of 0.6 to a value of 0.5.
 - 14.3.1.1.2 The autotune report must be examined to ensure that all parameters are within an acceptable range. If the autotune report is not acceptable, do not use the instrument until this condition is corrected. An acceptable tune will be noted in

the logbook with the date and initials of the approving examiner. Hard copies of tunes shall be retained for six years.

Autotune Acceptance Criteria:

TUNE PARAMETER	SPECIFIC PARAMETER	ACCEPTANCE RANGE
Peak widths	0.50 Da	± 0.05 Da (spread between values ≤ 0.05)
Mass assignment	69.00, 219.00, 502.00 Da	± 0.10 Da
Isotope Ratios	Ratio of mass 70 to 69	0.5 – 1.6%
Isotope Ratios	Ratio of mass 220 to 219	3.2 – 5.4%
Isotope Ratios	Ratio of mass 503 to 502	7.9– 12.3%

14.3.1.2 A background spectrum shall be collected for the GC-MS.

14.3.1.2.1 The background spectrum must be examined to ensure that all parameters are within an acceptable range. If the background spectrum is not acceptable, do not use the instrument until this condition is corrected. An acceptable background will be noted in the logbook with the date and initials of the approving examiner. Copies of backgrounds shall be retained for six years.

14.3.1.3 The Resolution Test Mixture (RTM) shall be injected to monitor GC-MS performance. This test mixture shall be a certified reference material containing the following compounds:

1,2,4-Trimethylbenzene, m-Ethyltoluene, o-Ethyltoluene, p-Xylene, Toluene, n-Hexane (C6), n-Octane (C8), n-Decane (C10), n-Dodecane (C12), n-Tetradecane (C14), n-Hexadecane (C16), n-Octadecane (C18), n-Eicosane (C20)

14.3.1.3.1 RTM acceptance criteria: The retention times for the 1,2,4-Trimethylbenzene (1,2,4-TMB) peak and the hexadecane (C16) peak must be within 0.033 minutes of the retention times from the previous RTM QC check. If the RTM is not acceptable, do not use the instrument until this condition is corrected

14.3.1.4 The Oxygenate Check Sample (OCS) (1:2:1 acetone, ethanol and methylethylketone in CS₂) shall be injected to monitor GC-MS performance. This will serve as a check of the resolution and proper functioning of the instrument.

14.3.1.4.1 OCS acceptance criteria: The abundance from the baseline to the valley between the acetone and ethanol peaks must be no greater than approximately 10% of the abundance of the acetone peak. If the OCS is not acceptable, do not use the instrument until this condition is corrected.

14.3.1.5 Record RTM and OCS runs in instrument log. RTM and OCS QC data is retained for six years.

14.4 Analysis of Casework Samples

14.4.1 The maximum allowable temperature program ramp rate for reproducible retention times is 30°C/min for Agilent GC models 6890 and higher.

- 14.4.2 In most instances, injection is made in the split mode at a split ratio of either 50:1 or 20:1. Splitless injections may be used when it is necessary to increase the amount of analyte delivered to the column and the detector.
- 14.4.2.1 Generally, a 50:1 split ratio is used for diluted standards and “strong” case samples, while a 20:1 split ratio is used for weaker case samples. Splitless injections may be used for even weaker samples. Routinely, a starting place for injections would be: 1.0 μL at 50:1 for diluted standards and “strong” case samples, and 2.0 μL at 20:1 for extracted case samples.
- 14.4.2.2 Changing the sample concentration, using larger injection volumes, or using SIM may be helpful when case samples are weaker than identification in full scan mode allows. Before using SIM, confirm that there is an indication that the compound(s) of interest are present at the appropriate retention time(s) with the appropriate major ions.
- 14.4.3 Normal injection volumes for diluted standards and case samples range from a needle dip to 3.0 μL . For headspace samples, injection volumes of up to 2.0 mL may be used. Samples may be concentrated to avoid injecting higher volumes.
- 14.4.4 It is encouraged that general chemical case samples be characterized as much as possible prior to injection into the instrument.
- 14.4.4.1 Samples may be diluted or dissolved in an appropriate solvent.
- 14.4.4.2 Depending on the nature of the sample, some samples must be cleaned up by extraction.
- 14.4.4.3 Some compounds do not chromatograph well, requiring derivatization to help effect good chromatographic peak shape.
- 14.4.5 Standards/references used in the comparison shall be run on the same day as the sample whenever possible, except for fire debris. “Same day” is defined as an approximate 24 hour period. Circumstances may exist where the sample and reference cannot be run on the same day; under these circumstances, the Section Supervisor may approve this minor deviation.
- 14.4.6 The injection order when running samples with standards should be either “standard, blank, sample(s)” or “blank, sample(s), standard.”
- 14.4.6.1 Appropriate standards/reference samples shall be run.
- 14.4.6.2 The blank, standard, and sample must be run under the same chromatographic conditions and data acquisition parameters, as appropriate. Additionally, sample concentrations should be approximately the same concentration as the standard.
- 14.4.6.3 At a minimum, an appropriate blank must be run prior to each sample. Additional blanks may be run at the examiner’s discretion.
- 14.4.6.4 When running multiple samples, a blank shall be run between sample injections. An extraction blank may be used as a blank that is run between sample injections.
- 14.4.6.5 A blank, run prior to each sample, shall be appropriate for the sample being run, i.e., an appropriate solvent or the solvent(s) used to dissolve or dilute the case samples, room air for headspace injections, or the clean needle to be used for needle dip scenarios. Additional blanks may be run at the examiner’s discretion.
- 14.4.6.6 Disposable syringes are used for headspace injections.

- 14.4.7 The date, the initials of the examiner, the FS Lab#, sequences and/or samples (including standards) are maintained in a sample log.
- 14.4.7.1 Sequencing via autosampler should be utilized whenever practical. If individual samples are not recorded in the logbook, completed sequence logs shall be printed and retained.
- 14.4.8 The identification of a compound must be based on a comparison of the retention time and the mass spectrum of an actual standard reference material. Integrated retention times for analytes must agree with the standard within 0.033 minutes.

14.5 Documentation

- 14.5.1 The Total Ion Chromatogram (TIC) for case samples is compared to the TIC for known reference samples according to retention times, mass spectra, and overall patterns.
- 14.5.1.1 No rigid mass spectral probability based match criteria are defined to identify a compound. Flexibility is given to the experienced interpreter because rigid criteria can lead to misidentification as well as under-identification. The identification will be based on a number of factors, such as retention time, unique ions, ion abundance, S/N and literature references as well as probability based matched scores.
- 14.5.2 To include in the case file:
- 14.5.2.1 TIC of blank run before the sample showing the entire range demonstrating that there are no interfering peaks in the region of interest.
- 14.5.2.1.1 If an extraction blank is used, include the extraction blank TIC showing no peaks in the region of interest at a scale of very low abundance or a printout of a time block around the retention time of interest showing that the extracted ions of the compound of interest are not present.
- 14.5.2.2 Sample's TIC, background subtracted mass spectrum, and normalized tabulation. The library printout may be included.
- 14.5.2.3 Standard's TIC, background subtracted mass spectrum, and normalized tabulation.
- 14.5.2.4 An air peak may be verified on screen and a notation hand-written on the sample's TIC. Alternatively, the TIC and spectrum of the air peak may be printed; however, it is unnecessary to print the tabulation and library search.
- 14.5.2.5 If a method other than those listed in 14.6 is used, document the method parameters used (e.g., print the method).
- 14.5.2.6 The minimum requirements for labeling data are as follows:
- FS Lab #
 - Item Number
 - Examiner's handwritten initials
 - Injection volume, unless noted in the GC-MS method below
 - Injection solvent

14.6 Generally Used GC-MS Methods

Method Name	Split Ratio	Injection Type	Initial Oven Temp / Hold Time	Temp Ramp	Temp / Hold Time	Temp Ramp	Final Oven Ramp / Hold Time	Inlet Temp	Transfer Line Temp	Column Flow	Scan Conditions (m/z range)	Solvent Delay
AUTF1-50 AUTF1-50CS2	50:1	ALS - 1µL	40°C / 1.5 min	20°C/min	140°C / 0 min	30°C/min	300°C / 5.17 min	290°C	300°C	■	14-200 @ 0 min; 14-400 @ 2 min; 14-600 @ 12 min	
AUTF1-50_OCS	50:1	ALS - 1µL	40°C / 1.5 min	20°C/min	140°C / 0 min			290°C	300°C	■	14-200 @ 0 min; 14-400 @ 2 min	
AUTF2-20 AUTF2-20CS2	20:1	ALS - 2µL	40°C / 1.5 min	20°C/min	140°C / 0 min	30°C/min	300°C / 5.17 min	290°C	300°C	■	14-200 @ 0 min; 14-400 @ 2 min; 14-600 @ 12 min	
AUTF2-20_OCS AUTF2-20_OCSNoCS2	20:1	ALS - 2µL	40°C / 1.5 min	20°C/min	140°C / 0 min			290°C	300°C	■	14-200 @ 0 min; 14-400 @ 2 min	
HDSPCE_40ISO	50:1	Manual	40°C / 5 min					290°C	300°C	1.5 mL/min	14-600	
HEADSPAC	20:1	Manual	35°C / 4 min	10°C/min	100°C / 0 min			290°C	300°C	▲	10-150	
GenScrn_1A	50:1	ALS - 1µL	60°C / 1 min	30°C/min	300°C / 6 min			290°C	300°C	1.5 mL/min	14-600 @ 1.8 min	1.8 min
GenScrnHDSPCE_1M	50:1	Manual	40°C / 0.5 min	20°C/min	100°C / 2 min	30°C/min	130°C / 1 min	290°C	300°C	1.5 mL/min	14-600	
LACH2-20	20:1	ALS - 2µL	200°C / 2 min	15°C/min	300°C / 3.33 min			290°C	300°C	●	14-200 @ 0 min; 14-400 @ 2 min; 14-600 @ 12 min	
LUBRICANTS	10:1	ALS - 1µL	40°C / 2 min	20°C/min	290°C / 5 min			290°C	300°C	1.8 mL/min	14-550	
SUGARTMS_1A	50:1	ALS - 1µL	150°C / 1 min	30°C/min	300°C / 4 min			290°C	290°C	1.2 mL/min	14-600 @ 2.3 min	2.3 min
SUGARTMS_3A	50:1	ALS - 3µL	150°C / 1 min	30°C/min	300°C / 4 min			290°C	290°C	1.2 mL/min	14-600 @ 2.3 min	2.3 min
SMOKY	16.66:1	ALS - 2 µL	50°C / 2 min	20°C/min	100°C / 0.5 min	25°C/min	300°C / 2 min	240°C	300°C	1.2 mL/min	14-400 @ 3.3 min; 14-600 @ 10 min	3.3 min

▲ = 0.6 mL/min (0 min hold), Ramp 20 mL/min to 1.2 mL/min (9.97 min hold), Ramp 20 mL/min to 1.8 mL/min (0 min hold)

■ = 1.8 mL/min (2 min hold), Ramp 20 mL/min to 1.2 mL/min (9.97 min hold), Ramp 20 mL/min to 1.8 mL/min (0 min hold)

● = 1.8 mL/min (2 min hold), Ramp 20 mL/min to 1.2 mL/min (7.97 min hold)

15 GLASS REFRACTIVE INDEX MEASUREMENT SYSTEM (GRIM3)**15.1 Logs**

- 15.1.1 Record any maintenance performed in a log, date and initial.
- 15.1.2 A separate sample log will be maintained that includes at a minimum the date, the initials of the examiner, the FS Lab# or other identifying information.

15.2 Calibration

- 15.2.1 Calibration of the instrumentation is performed on an annual basis for the silicone oil and interference filter being used in casework. The calibration data is used by the GRIM3 system to determine refractive index (RI) at the given filter wavelength. The system is calibrated with the 589 nm interference filter using Locke Scientific Silicone Oil B and the Locke Scientific B Series of glass standards (B2 –B4; B7- B12) using the declared RI values. Five readings from five glass particles are obtained from one prepared slide of each of the Locke Scientific B Series glass standards used. The calibration correlation value must be less than or equal to -0.99990. The dRI must be between -10 and +10 inclusive for each standard glass analyzed. New slides of the Locke B standards will be prepared annually.
- 15.2.2 The calibration of the GRIM3 system using the Locke B oil will be checked using Locke glass standards B3, B6 and B11 taking five readings from five glass particles from one prepared slide. The mean of the five readings will be compared to the declared certificate value for the B3, B6 and B11 glasses. This value must be +/- 0.00010 of the declared certified value.
- 15.2.3 If headlight glass is encountered, the GRIM3 is calibrated at 589 nm using Locke Scientific Silicone Oil C and the Locke Scientific C Series of glass standards using the RI values from the table in the Locke manual that are closest to the match temperature of the glasses used. Five readings from five particles are obtained from one prepared slide of each of the appropriate glass standards. The calibration correlation value must be less than or equal to -0.99990. The dRI must be between -10 and +10 inclusive for each standard glass analyzed. Prepare glass standard slides, as appropriate. This calibration for headlight glass is good for one year from the date it was obtained.
- 15.2.4 The calibration of the GRIM3 system using the Locke C oil will be checked with NIST SRM 1820 taking five readings from five glass particles from one prepared slide. The mean of the five readings will be compared to the certificate value for the NIST SRM 1820. This value must be +/- 0.00010 of the certificate value for the SRM. New slides of the NIST SRM 1820 will be prepared as needed and may be used for up to one year from preparation. Since the use of this oil is non-routine, the data will be photocopied and retained in the case file. Additionally, the original data will be printed and retained in the QA binder for the equipment.
- 15.2.5 If glass is encountered which has a very high refractive index that is above the range of the Locke Scientific Silicone Oil B, the GRIM3 can be calibrated at 589 nm using Locke Scientific Silicone Oil A and the Locke Scientific A Series and B Series of glass standards, as appropriate, using RI values from the table in the Locke manual that are closest to the match temperature of the glasses used. Five readings from five particles are obtained from one prepared slide of each of the appropriate glass standards. The calibration correlation value must be less than or equal to -0.99990. The dRI must be between -10 and +10 inclusive for each standard glass analyzed. Prepare glass standard slides, as appropriate. This calibration for glass with a very high refractive index is good for one year from the date it was obtained.
- 15.2.6 The calibration of the GRIM3 system using the Locke A oil will be checked using Locke glass standard A4. Locke glass standard A4 will be prepared as needed and may be used for up to one year from preparation. Five readings from five particles are obtained from one prepared slide of each of the appropriate glass standards. The mean of the five readings will be compared to the RI value from the table in the Locke manual for A4 and must be +/- 0.00010 of the RI value from the table in the Locke manual for A4. Since the use of this oil is non-routine, the data will be photocopied and retained in the case file. Additionally, the original data will be printed and retained in the QA log for the equipment.

15.2.7 The calibration data will be printed and retained in a log which will be maintained with the equipment.

15.3 Monthly QA

15.3.1 The following monthly QA procedures must be performed prior to use in casework. If the system is not used for casework during a given month, these procedures do not have to be performed until the next time casework is performed. A notation shall be placed in the sample log in the event the instrument is not used for casework during any given month.

15.3.2 The Mettler hot stage on the GRIM3 system will be inspected internally for residual mounting oil. In addition, the glass slide that is positioned in the hot stage under the objective opening will also be inspected. Set the hot stage temperature to a low setting and clean the hot stage and glass slide with methyl or isopropyl alcohol as needed.

15.3.3 The accuracy of the GRIM3 system will be checked using Locke glass standards B3, B6 and B11 in the Locke Silicone Oil B. Five readings from five particles are obtained from one prepared slide of each of the appropriate glass standards. The mean of the five readings will be compared to the certificate value for the B3, B6 and B11 glasses. These values must be +/- 0.00010 of the declared certified value.

15.3.4 When the monthly QA check is performed, the data will be printed and retained in a log which will be maintained with the instrument. No QA data will exist for months in which no casework was performed.

15.4 Day-of-Use QA

15.4.1 The Day-of-Use QA will be performed each day prior to running case samples. The Day-of-Use QA is not necessary when completed on the same day as the Monthly QA. The calibration will be checked using Locke glass standard B6 in the Locke Silicone Oil B. Five readings from five particles are obtained from one prepared slide. The mean of the five readings will be compared to the certificate value for the B6 glass. This value must be +/- 0.00010 of the declared certified values for the B6 glass. Include the data for this QA check with each case file analyzed that day.

15.5 Case QC Check

15.5.1 For each case examined with a single known glass sample, a single glass standard is analyzed using the appropriate wavelength and oil. The Locke glass standard selected for this QC check will be that which is closest to the refractive index value of the known glass. If multiple known glass samples of different refractive indices are present, then multiple glass standards may be necessary. Five readings from five particles are obtained from one prepared slide of each of the appropriate glass standards. The mean of the five readings will be compared to the certificate value for the glass standard(s). This value must be +/- 0.00010 of the declared certified value.

16 ION CHROMATOGRAPH (IC)

- 16.1** Record any maintenance performed in a log, date and initial.
- 16.2** A separate sample log will be maintained that includes at a minimum the date, the initials of the examiner, the FS Lab# or other identifying information.
- 16.2.1 Sequencing via autosampler should be utilized whenever practical.
- 16.2.2 Sequences and/or samples (including standards) shall be recorded in the instrument logbook.
- 16.2.2.1 If individual samples are not recorded in the logbook, completed sequence logs shall be printed and retained.

- 16.3** The Dionex reference standard for the anion system consists of fluoride, chloride, nitrate, sulfate, and phosphate ions. Nitrite, chlorite and chlorate will be added when necessary. The reference standard for the cation system consists of lithium, sodium, ammonium, potassium, and calcium. A perchlorate reference standard is prepared in-house and used when applicable.

The approximate concentrations of each are as follows: (Note: ppm = mg/L)

Anions

Fluoride - 20 ppm
 Chloride - 30 ppm
 Nitrate - 100 ppm
 Phosphate - 150ppm
 Sulfate - 150 ppm
 [Nitrite ~ 100 ppm]
 [Chlorite ~ 100 ppm]
 [Chlorate ~ 100 ppm]

Anions

Perchlorate – variable

Cations

Lithium - 50 ppm
 Sodium - 200 ppm
 Ammonium - 400 ppm
 Potassium - 200 ppm
 Magnesium - 200 ppm
 Calcium - 1000 ppm

- 16.4** The system is configured with the necessary eluent, regenerant, columns, suppressors, and detector as established by Dionex Corporation.
- 16.5** Prior to each day's use, the IC system is initially flushed until an acceptable and stable baseline is established. The appropriate reference standard is injected until system stability is demonstrated by two successive, reproducible chromatograms. These chromatograms are evaluated subjectively on the basis of relative retention times, peak resolution, baseline stability, and peak heights. The following parameters are checked and recorded in a logbook: date, column used, system pressure and background conductivity. The retention times of key components (chloride, nitrate, sulfate, and phosphate) are also recorded.
- 16.6** If an acceptable and stable baseline and/or two successive, reproducible chromatograms cannot be achieved, then do not use the instrument. Call Dionex for repair.
- 16.7** All appropriate reference standards and blanks are run with the casework. The appropriate reference standard is run both prior to and following all casework on the day of use.
- 16.8** Corrective procedures are performed on the unit as required through monitoring the parameters and performance.
- 16.9** A service contract is maintained through the manufacturer, and the instrument receives an annual diagnostic check, including the electronics.

17 MICROSPECTROPHOTOMETER (MSP)

17.1 Overview

The QC checks will be performed prior to using the instrument for casework. A sample log will be maintained that includes at a minimum the date, initials of the examiner and the FS Lab# or other identifying information. On a semi-annual basis, copy data files to a writeable CD/DVD and retain.

17.1.1 Day-of-Use QC Check

17.1.1.1 The lamp power supply will remain on for a minimum of thirty minutes prior to conducting the QC checks.

17.1.1.2 Wavelength Checks will be performed using the following instrument parameters:

- AutoCalibration-Transmission tool
- Focused on an ink-free area on the reference slide
- Objective: 10X
- Aperture: 4
- Holmium Oxide & Didymium Filters

The wavelength checks must be within the manufacturer's established calibration limits to pass. The results shall be saved, printed, initialed, and retained.

17.1.1.3 Photometric Checks will be performed using the following instrument parameters:

- AutoCalibration-Transmission tool
- Focused on an ink-free area on the reference slide
- Objective: 10X
- Aperture: 4
- Neutral Density Filters: 0.1, 0.5, and 1.0

The photometric checks must be within the manufacturer's established calibration limits to pass. The results shall be saved, printed, initialed, and retained.

17.1.1.4 UV Peak Check includes manually checking two certified peaks in the UV region of the Holmium Oxide filter using the following instrument parameters:

- Wavelength range: 240-300 nm
- Resolution factor : 0
- Aperture: 4
- Measurements taken in Absorbance

The peak values are compared to the certified values to ensure the instrument is within established manufacturer's calibration limits. A copy of the spectrum with the two peaks displayed will be generated, initialed, and retained in the MSP QC Notebook. If the UV Peak check has been previously performed during the given calendar month, the UV Peak Check does not need to be redone.

17.1.1.5 If the obtained values for the Holmium oxide, Didymium, and neutral density filters do not fall within the current NIST certified reference value ranges, perform Köhler illumination adjustment on the microscope and repeat the instrument wavelength check, photometric check, and UV Peak check. **DO NOT PROCEED WITH CASEWORK SAMPLES** until all of the filter peak mark values are within an acceptable range of the NIST certified reference values. Call Craic Technical Support as needed.

17.2 Analysis of Casework Samples

- 17.2.1 Prior to using the MSP, evidence samples have already been visually and/or microscopically compared and found to have no meaningful, or unexplainable, differences in the side-by-side comparison of color. The QC checks using the Holmium oxide, Didymium, and neutral density filters have been completed prior to, and on the same day, as casework MSP spectra are obtained.
- 17.2.2 Spectra are collected considering the following:
- 17.2.2.1 Two objectives are available for use (10X and 40X). The selected objective size is dependent upon the size of the colored material being examined.
 - 17.2.2.2 All casework samples will be collected with a resolution factor of 4.
 - 17.2.2.3 The appropriate sampling aperture size should be selected such that it is completely contained in the area of the sample to be measured.
 - 17.2.2.3.1 The aperture size used for data collection will be recorded on the spectra.
 - 17.2.2.4 Samples shall be mounted using quartz slides and cover slips if they are to be compared in the UV region. Samples shall be mounted in glycerin, xylene substitute or water.
 - 17.2.2.5 While the spectral range extends from 240 nm to 925 nm, generally above 800 nm is too noisy for comparative purposes.
 - 17.2.2.6 Fiber spectra will include the UV with the wavelength range of 240-800 nm.
 - 17.2.2.7 Autoset Optimize sets the integration time for the instrument while Dark Scan establishes and adjusts for instrument noise.
 - 17.2.2.7.1 If the aperture size is changed, a new optimization and dark scan is necessary.
 - 17.2.2.7.2 Repeat Autoset Optimize and Dark Scan periodically during lengthy sample measurement sessions.
 - 17.2.2.8 Reference Scan measures and compensates for the light transmitting/absorbing effect of the light source, optics, glass slide, cover slip, and mounting media.
 - 17.2.2.8.1 Generally, whenever the sample is moved out of the field of view to another target location on the same or on a different slide, the Reference Scan function should be repeated.
- 17.2.3 Spectra, along with spectral averages (optional) shall be printed and initialed as needed for case file.
- 17.2.4 The individual sample spectra and spectral averages (optional) are compared between samples (i.e., K and Q). If any meaningful differences are observed, the examiner may discontinue the analysis.

17.3 Maintenance

- 17.3.1 Maintenance and/or repairs will be performed as needed and summarized in the logbook to include the date and initials of the person making the entry.
- 17.3.2 Always handle the Holmium oxide, Didymium, and neutral density filters by the edges to avoid fingerprints on the surface. These filters were received with NIST certified values and are sent for re-certification prior to the expiration of their certificates.

- 17.3.3 Anytime the software is reloaded onto the computer or the filters are recertified, ensure the correct filter values are being used.
- 17.3.4 In order to preserve the life of the xenon bulb, reduce the number of times the lamp is turned on. When shutting down, turn off the lamp power supplies first, followed by the microscope power, detector chiller, and computer in no particular order.

17.4 Reference

- 17.4.1 CRAIC QDI 2010 Microspectrometer User's Manual, Version 2.7, CRAIC Technologies, Sam Dimas, CA, 2002-2007.

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18 SCANNING ELECTRON MICROSCOPE-ENERGY DISPERSIVE X-RAY ANALYSIS (SEM-EDS)

18.1 General

- 18.1.1 A service contract may be maintained for each instrument which includes comprehensive annual preventive maintenance (PM) visits. Copies of the paperwork from the annual PM visit, if applicable, are retained in the maintenance log.
- 18.1.2 Prior to use, the calibration of the x-ray analyzer is checked with a copper standard.
- 18.1.2.1 If the calibration check fails, attempt to check again. If the calibration check still fails, then call ASPEX service.
- 18.1.3 The liquid nitrogen Dewar on the x-ray detector is filled at least twice a week to insure that it does not go dry.
- 18.1.4 The filament assembly is replaced and gun cap cleaned if necessary when the filament burns out. Be aware of elevated temperatures when changing a filament which has been in operation.
- 18.1.4.1 During filament replacement on the ASPEX Instruments be sure to disable the beam power supply located on the back panel of the beam supply under the bias knob.
- 18.1.5 Change apertures as indicated by stigmation.
- 18.1.6 The mechanical vacuum pump oil is changed annually.
- 18.1.7 The resolution is checked monthly by taking a 25,000x micrograph of an appropriate standard.
- 18.1.8 Service, maintenance and other calibration data are logged and kept in the SEM laboratory.

18.2 ASPEX Daily Operations Log

A bound log is kept for documenting the daily operations of the ASPEX Instruments. This log is separated into columns that include appropriate parameters and a comments section. Comments refer to filament changes, overnight runs and equipment adjustments as they arise.

18.3 SEM-EDS System Maintenance Schedule

- 18.3.1 Fill the Dewar with liquid nitrogen as needed.
- 18.3.2 Change the mechanical pump oil every 12 months or when it becomes cloudy or low.
- 18.3.3 Change apertures as indicated by stigmation.

18.4 ASPEX Emergency Shutdown Procedures

- Turn off filament.
- Minimize perception software.
- Shutdown Windows 2000.
- Turn off main, power and pulse proc. toggle switches on SEM.

18.5 ASPEX EDS System

18.5.1 Prior to use calibration

18.5.1.1 Use a Cu Standard.

18.5.1.1.1 When using the ASPEX or VP2000 instrument the beam is placed on a copper standard and the spot size adjusted to achieve approximately 1,500 CPS with a process time of 25.6 μ sec at 20KV. A spectrum from 0 to 10.24 KeV is collected and calibrated using the EDS detector calibration software. When using the ASPEX 3000 instrument the beam is placed on a copper standard and the spot size adjusted to achieve approximately 20,000 CPS with a process time of 3.2 μ sec at 25KV. A spectrum from 0 to 10.24 KeV is collected and calibrated using the EDS detector calibration software. When using the ASPEX Explorer instrument the beam is placed on a copper standard and the spot size adjusted to achieve approximately 30,000 CPS with a process time of 3.2 μ sec at 25KV. A spectrum from 0 to 10.24 KeV is collected and calibrated using the EDS detector calibration software.

18.5.1.1.1.1 If the calibration fails, attempt again. If the calibration still fails, then call ASPEX service.

18.5.1.1.2 During automated GSR runs and prior to analyzing instrument support samples the centroids of these peaks will be monitored. The Cu $K\alpha$ centroid should be 8.04 ± 0.03 KeV. The Cu $L\alpha$ centroid should be 0.93 ± 0.03 KeV. FWHM of Cu $K\alpha$ will also be monitored during the automated run. It should not exceed 170 eV.

18.5.1.1.2.1 If it is not possible to obtain the appropriate values, recalibrate the EDS detector and rerun the samples. If it is still not possible to obtain the appropriate values, contact ASPEX service.

18.5.1.1.3 A hard copy of the QC data collected during automated GSR runs and instrument support analysis will be kept in a QC notebook.

18.6 SEM-EDS Primer Residue Kit – QC

18.6.1 Prior to the manufacture of the PR kits a sample of the carbon tape to be used is analyzed to ensure no primer residue particles are present. Inform the manufacturer that the tape is acceptable so they may begin production of the kits. If the tape is not acceptable, request that the manufacturer send another tape sample for testing.

18.6.2 Each box of kits from the manufacturer must have a lot number associated with it. Do not accept the kits for testing without a lot number.

18.6.3 Select 2% of the kits received from the manufacturer for predistribution testing.

18.6.3.1 Inspect the kits to ensure all components are present.

18.6.3.2 Visually inspect the sample stubs. The stubs should be completely covered with carbon conductive tape.

18.6.3.3 Run an automated primer residue analysis on one sample from each kit to ensure no primer residue particles are present.

18.6.3.4 If all of the above checks pass, then inform the DFS Stockroom that the Primer Residue kits are available for distribution. Otherwise, do not release the kits for distribution.

18.6.4 The QC'd kits are retained and used as negative controls for automated primer residue runs.

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19 X-RAY POWDER DIFFRACTOMETER (XRD)**19.1 Logs**

- 19.1.1 Record any maintenance performed and calibration check data in a log, date and initial.
- 19.1.2 A separate sample log will be maintained that includes at a minimum the date, the initials of the examiner, the FS Lab# or other identifying information.

19.2 Monthly QC

An alumina standard (NIST reference standard) is run monthly. The intensity in counts per second and the d -spacings are recorded at generator settings of 45 kV and 40 mA for the 43.35 and 76.86 peaks. The values obtained at these two angles must fall within 0.03 of these stated values for the instrument to be used in casework. If values fall outside this range, do not use the instrument for casework. Call a service technician. When intensity values for the 43.35 peak and the 76.86 peak fall below 1400 counts per second and 850 counts per second respectively, the x-ray tube must be replaced.

19.3 Day-of-Use QC

- 19.3.1 A silicon dioxide (SiO_2) reference is run on the day of use prior to case work. The intensity in counts per second and the d -spacings are recorded at generator settings of 45 kV and 40 mA for the 26.63 peak in SiO_2 . The value obtained at this angle must fall within 0.03 minutes of the stated value for the instrument to be used in case work. If values fall outside this range, do not use the instrument for casework. Call a service technician. The intensity in counts per second is compared to the values previously recorded for each recent day of use. If the operator notes a sudden decrease in intensity or questions these comparative values, the alumina standard should be run.
- 19.3.2 Resolution is checked by running a silicon dioxide (SiO_2) reference at generator settings of 45 kV and 40 mA . Enhancement of the 67° - 69° range of the SiO_2 pattern should reveal five distinguishable peaks. If five distinguishable peaks are obtained, then a check mark is placed in the appropriate column on the log sheet. If the five peaks are not distinguishable, do not use the instrument for casework. Call a service technician.
- 19.3.3 The standard method with instrument conditions and settings is:

Panalytical Empyrean
Goniometer: Theta/Theta
Generator: Spellman
X-ray tube: Cu
Used radiation: K-Alpha
Detector: X'Celerator

Measurement program: Explosives

Start angle (°2Theta):	5
End angle (°2Theta):	75
Step size (°2Theta):	0.0083556
Time per step(s):	1.905
Generator kV:	45.0
Generator mA:	40.0

- 19.3.4 A case file will not require an XRD conditions sheet as long as the data is acquired using the standard method. If modifications to the standard method are made, an XRD conditions sheet showing the changes must be included with each case analyzed using the modified method.

20 LASER INDUCED BREAKDOWN SPECTROSCOPY (LIBS)**20.1 Introduction**

20.1.1 Laser induced breakdown spectroscopy is used to analyze suspected clandestine laboratory samples for the presence of lithium.

20.2 Logs and Worksheets

20.2.1 Record instrument utilization and associated maintenance in the designated log book.

20.2.2 A QA worksheet shall be maintained electronically for day-of-use instrument QA. A copy of the QA worksheet shall be included in the case file.

20.3 Safety Considerations

20.3.1 Ensure the Quatro JSL Series HEPA collection system is powered on prior to instrument utilization. The HEPA collection system removes any particulate matter that may be formed during analysis.

20.3.2 The RT100 LIBS utilizes a Class 4 laser product when interlocks are engaged. The RT100 LIBS shall be interlocked at all times for user safety. When interlocks are compromised a Class 4 high power laser is exposed. Extreme care must be taken when servicing the laser and shall only be performed by qualified trained personnel.

20.3.3 To ensure proper cooling during laser utilization, verify the water reservoir on the laser is approximately 80% full prior to use.

20.4 Day-of-Use Quality Assurance

20.4.1 NIST standard reference material 1831 soda lime sheet glass shall be analyzed in replicates of ten on the day-of-use prior to casework. The following instrumental method shall be used for analysis:

Instrumental Method

Parameter	Setting
Spot Size	100 μm
Spectrometer Delay	1 μsec
Laser Out Put	100 %
Repetition Rate	10 Hz
Warm-up Shots	100
Number of Shots	50
Accumulation of Data	On
Shot Location	Single Location

20.4.2 Elemental ratios shall be extrapolated from the acquired data and evaluated to meet acceptance criterion. Data files shall be in .csv format and evaluated using the quality assurance macro. The analyst shall input analyst name and date in the QA Worksheet and review the worksheet for pass/fail results of respective elemental ratios. A total of 64 elemental ratios and associated wavelengths shall be evaluated to ensure proper instrumental performance. At minimum, a total of sixty out of sixty-four elemental ratios must be within their respective acceptance criterion to pass the daily quality assurance check. The wavelengths utilized for the elemental ratio determinations must also be within ± 0.12 nm of the *TrueLIBS* wavelength for daily quality assurance acceptance. For a detailed list of wavelengths and acceptance criterion see Section 20.7.

20.4.3 If the acquired ratios fall outside of the designated acceptance range, repeat analysis. If acquired ratios fall outside of the designated acceptance range after second analysis, do not use instrument for casework. Call a service technician or perform necessary instrument maintenance.

- 20.4.4 The wavelengths evaluated using the NIST SRM 1831 soda lime sheet glass evaluate each spectrometer channel within the instrumentation with the exception of Channel 6 (884-1040 nm). The spectrometer channels are:

Spectrometer Configuration

Channel	Wavelength Range (nm)
Channel 1	190-309
Channel 2	309-460
Channel 3	460-588
Channel 4	588-692
Channel 5	692-884
Channel 6	884-1040

Channel 6 covers the 884 to 1040 nm wavelength range. No spectral lines within this channel are used for analysis and subsequently no spectral lines within this range are utilized for instrument quality assurance.

20.5 Analytical Procedures

20.5.1 Sample Preparation

- 20.5.1.1 The sample preparation technique utilized may vary depending on the consistency of evidentiary material. Double-sided tape, adhesive dots, and clear nail polish are approved sample preparation matrices. The Department *Reagent Worksheet* shall be used to record sample preparation matrices.
- 20.5.1.1.1 The matrix utilized for sample preparation and blank analysis shall come from the same source (e.g., bottle of nail polish, package of adhesive dots). If multiple matrix sources are utilized, blank analyses of each lot of matrix shall be completed.
- 20.5.1.1.2 The individual sample report for the initial blank shall be included in the case file.
- 20.5.1.1.3 Blank matrix shall be evaluated before and after the analysis of evidentiary samples. Individual sample reports for blanks run between samples do not need to be included in the case file. Instead, an image will be generated and placed in the case file showing the list of data files generated for that day. The examiner shall indicate that the blanks have been evaluated and are free from carryover.
- 20.5.1.1.4 The blank matrix analyzed after evidentiary samples must have a consistent spectrum with the blank matrix evaluated before evidentiary sample(s) to indicate the sample chamber is free from carryover.
- 20.5.1.1.4.1 If the blank matrix sample analyzed after evidentiary sample(s) contains carryover from the evidentiary sample, the sample chamber shall be purged with 1.0 L/min helium and/or wiped down with isopropanol. After purging/cleaning the sample chamber, the blank matrix shall be re-evaluated to demonstrate that all sources of carryover have been removed. If the source of carryover cannot be removed, remove instrument from service until issue has been corrected.
- 20.5.1.1.4.2 If carryover is detected, individual sample reports must be generated and placed in the case file for the blank showing

carryover and the blank showing that the carryover has been remediated.

20.5.1.1.5 Double-sided tape

20.5.1.1.5.1 Adhere double-sided tape to glass slide.

20.5.1.1.5.2 Apply representative sample of evidentiary material onto double-sided tape for analysis.

20.5.1.1.6 Adhesive dots

20.5.1.1.6.1 Adhere adhesive dot to glass slide.

20.5.1.1.6.2 Apply representative sample of evidentiary material onto adhesive dot for analysis.

20.5.1.1.7 Clear nail polish

20.5.1.1.7.1 Apply approximately 1-2 drops of clear nail polish to glass slide as needed.

20.5.1.1.7.2 Mix representative sample of evidentiary material with clear nail polish to produce a homogenous slurry.

20.5.1.1.7.3 Allow sample to dry prior to analysis.

20.5.2 Instrumental Parameters

The instrumental parameters described herein are optimal instrumental conditions. The laser intensity and number of warm-up shots shall not be changed. The number of laser shots, spectrometer delay, laser spot size, repetition rate, chamber gas composition and flow rate, as well as laser position may be optimized for individual analysis. The sample instrumental method shall be utilized for both blank samples and evidentiary samples within an analysis.

Prior to analysis, the instrument shall be powered on and allowed at minimum forty-five minutes to warm-up the system.

20.5.2.1 Optimized Instrumental Parameters:

Optimized Instrumental Parameters - Lithium

Laser Shots	5
Laser Intensity	70%
Laser Spot Size	135 µm
Spectrometer Delay	0.1 µsec
Repetition Rate	5 Hz
Chamber Gas	Ambient
Laser Position	Single Point
Warm-up Shots	100
Data Accumulation	On

20.5.2.2 Optimized Spectral Lines

20.5.2.2.1 In general, identified spectral lines should have acceptable peak shape and a signal to noise ratio greater than 3:1 for identification.

20.5.2.2.1.1 In some circumstances, doublet peaks may occur due to the inherent nature of self-quenching of some spectral lines. Self-quenched peaks shall be considered to have acceptable peak shape.

20.5.2.2.2 At a minimum, three spectral lines must be identified to confirm the presence of lithium. The spectral lines include the most abundant lines for lithium. Other spectral lines are delineated within the validation summary.

Spectral Lines (nm)

Target Element	Spectral Lines (nm)
Lithium	323, 413, 610, 670, 812

20.5.2.2.3 The Applied Spectra Axiom software contains a *TruLIBS™* database as well as a NIST atomic emission line database. These databases include information regarding the wavelengths and associated intensities for elements which can be utilized to further indicate the presence of additional elements present in an evidentiary sample.

20.5.3 Instrument Operation

20.5.3.1 Power On Sequence

20.5.3.1.1 Power on Quatro JSL Series HEPA collection system.

20.5.3.1.2 Turn on laser power supply.

20.5.3.1.3 Depress 'On' button located on the front panel of the instrument.

20.5.3.1.4 Open Axiom instrument software.

20.5.3.2 Allow at minimum, 45 minutes for the instrument to warm-up prior to sample analysis.

20.5.3.3 Press 'Door Locked' button in software to disengage interlock system.

20.5.3.4 Remove sample chamber from instrument load door. Place sample in sample chamber and return to instrument. Close load door.

20.5.3.5 Press 'Door Open' button in software to activate interlock system. The laser will not initialize unless interlock system is engaged.

20.5.3.6 Analyze sample using appropriate instrumental parameters.

20.5.3.7 Upon completion of sample analysis, press 'Door Locked' button in software and remove sample from sample chamber.

20.5.3.8 Power Down Sequence

20.5.3.8.1 Press 'Exit' in Axiom software to exit program.

20.5.3.8.2 Depress 'Off' button on the front panel of the instrument.

20.5.3.8.3 Turn off laser power supply.

20.5.3.8.4 Power off Quatra JSL Series HEPA collection system.

20.6 Quality Assurance Acceptance Criteria

%CV Acceptance Criteria For Wavelength Ratios

Elemental Ratio	Wavelength Ratio	% Coefficient of Variation
Al/Mg	309.284/383.829	< 5%
Al/Mg	309.284/516.732	< 5%
Al/Mg	309.284/517.268	< 5%
Al/Mg	309.284/518.361	< 5%
Al/Mg	394.400/383.23	< 5%
Al/Mg	394.400/383.829	< 5%
Al/Mg	396.152/383.829	< 5%
Mg/Sr	383.23/407.771	< 5%
Mg/Sr	383.829/407.771	< 5%
Al/K	308.215/766.490	< 10%
Al/Mg	308.215/518.361	< 10%
Al/Na	308.215/588.995	< 10%
Al/Na	308.215/819.483	< 10%
Al/Ti	308.215/336.121	< 10%
Al/Mg	309.284/383.23	< 10%
Al/Na	309.284/588.995	< 10%
Al/Na	309.284/589.593	< 10%
Al/Ti	309.284/336.121	< 10%
Al/Ca	394.400/373.687	< 10%
Al/Mg	394.400/517.268	< 10%
Al/Ti	394.400/336.121	< 10%
Ca/Sr	373.687/407.771	< 10%
Ca/Mg	393.366/518.361	< 10%
Ca/Na	393.366/588.995	< 10%
Ca/Na	396.847/330.237	< 10%
K/Mg	766.490/518.361	< 10%
K/Na	766.490/589.563	< 10%
K/Na	766.490/819.483	< 10%
K/Mg	769.896/516.732	< 10%
K/Na	769.896/819.483	< 10%
Li/Ti	610.354/336.121	< 10%
Mg/Ti	383.23/336.121	< 10%
Mg/Ti	383.829/336.121	< 10%
Mg/Na	516.732/588.995	< 10%
Mg/Na	516.732/819.483	< 10%
Mg/Na	518.361/819.483	< 10%
Mg/Ti	518.361/336.121	< 10%
Sr/Ti	407.771/334.904	< 10%
Sr/Ti	407.771/336.121	< 10%
Al/Ca	308.215/317.933	< 20%
Al/Li	308.215/610.354	< 20%
Al/K	309.284/766.490	< 20%
Al/Li	309.284/610.354	< 20%
Al/Mg	309.284/280.27	< 20%
Al/K	394.400/766.490	< 20%
Al/Na	394.400/819.483	< 20%
Al/Na	396.152/819.483	< 20%
Ca/Mg	315.887/517.268	< 20%
Ca/Na	315.887/588.995	< 20%
Ca/Mg	317.933/383.829	< 20%
Ca/Mg	317.933/518.361	< 20%
Ca/Na	317.933/589.593	< 20%

Elemental Ratio	Wavelength Ratio	% Coefficient of Variation
Ca/Ti	317.933/336.121	< 20%
Ca/Mg	373.687/517.268	< 20%
Ca/Na	373.687/588.995	< 20%
Ca/Ti	393.366/334.904	< 20%
Ca/Na	396.847/819.483	< 20%
Li/Mg	610.354/383.829	< 20%
Li/Na	610.354/330.237	< 20%
Mg/Na	279.553/589.593	< 20%
Mg/Na	280.27/588.995	< 20%
Mg/Na	383.829/819.483	< 20%
Mg/Na	516.732/330.237	< 20%
Na/Ti	819.483/334.904	< 20%

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21 QUALITY ASSURANCE**21.1 Introduction**

- 21.1.1 The purpose of this section is to provide a uniform Quality Assurance (QA) Program for the Section for equipment and supplies that are not mentioned in previous sections. It is to establish a baseline or reference point of reliability and system performance.
- 21.1.2 TE examinations typically are comprised of identifications and/or comparisons between known and questioned samples. Certified reference materials are currently not available for most comparisons or identifications, therefore, commercially available products and reference materials are purchased as necessary.
- 21.1.2.1 Traceability has been established for certain measuring equipment including: balances, check weights, digital calipers, gauge blocks, and Eppendorf variable pipettes. These measuring devices shall be calibrated by an outside vendor that is accredited to ISO/IEC 17025:2005 and whose scope of accreditation covers the calibration performed.
- 21.1.3 It is expected that the examiner will report any unacceptable or anomalous behavior of any analytical systems immediately to either their Section Supervisor or the individual primarily responsible for a particular instrument. It is further expected that appropriate measures which ensure resolution of the issue will be carried out ASAP and that these will be properly documented.

21.2 Balances

- 21.2.1 Balances shall be checked prior to use in casework, however, if the balance has been previously checked during the given month, the balance does not need to be re-checked. The calibration check is performed using calibrated, traceable check weights and is dependent upon the range of the balance. Record the weights in the Balance QC Log.
- 21.2.1.1 Use the following check weights depending on balance class:
- 21.2.1.1.1 4-place analytical balance:
- 0.1000 (± 0.0003) gram
 - 5.0000 (± 0.0005) gram
 - 100.0000 (± 0.0010) gram
- 21.2.1.1.2 3-place toploading balance:
- 1.000 (± 0.002) gram
 - 10.000 (± 0.005) gram
 - 100.000 (± 0.005) gram
- 21.2.1.1.3 2-place toploading balance:
- 5.00 (± 0.02) grams
 - 100.00 (± 0.03) gram
 - 1000.00 (± 0.05) gram
- 21.2.1.2 The calibration check data is recorded on the *Balance QC Log* and is kept with each balance. The person performing the calibration check shall initial and date the form.
- 21.2.1.3 If a result from the calibration check is outside of the acceptable range, ensure that the balance is level and free of debris, and recheck the balance a second time.

- 21.2.1.4 If a result is outside of the acceptable range after rechecking, the balance shall immediately be taken out of service until maintenance and/or calibration are performed by an approved vendor.
- 21.2.1.5 Weights used to check balance accuracy shall be re-certified every three years by an ISO/IEC 17025:2005 accredited vendor whose scope of accreditation covers the certification performed.
- 21.2.1.6 Balances shall be calibrated annually by an outside vendor that is accredited to ISO/IEC 17025:2005 and whose scope of accreditation covers the calibration performed. Record the service call on the *Balance QC Log* that is kept with each balance.
- 21.2.2 Service receipts and miscellaneous balance maintenance comments are retained with QA records that are kept with each balance.
- 21.2.3 An examiner may use a calibrated balance from another Section in the Department. The examiner's case notes will reflect the identification of the balance and its location, for example: Controlled Substances (DX) Section, Sartorius, DFS XXX.
- 21.2.4 When recording weights obtained on a TE balance, the examiner shall record the weight to the readability of the balance. Weights shall not be rounded.
- 21.2.5 The Department has developed processes and collected data for the Uncertainty of Measurement program for the DX Section.
- 21.2.5.1 When reporting critical weights in a Certificate of Analysis, use a balance in the DX Section where the Uncertainty of Measurement (UoM) has already been determined. Complete an Instrument Support (IS) Request Form and arrange with an examiner in the DX Section to conduct the weighing. The TE examiner will retain custody of the evidence during the weighing process. Ensure that the identifying information for the balance and the determined weight(s) are recorded on the IS form by the DX examiner. In addition, the DX examiner shall complete the Controlled Substances Weighing Worksheet. The TE examiner shall include the Support Request Form and Weighing Worksheet in the case file and report UoM as described below. Refer to section 32 of the Controlled Substances Procedures Manual for UoM information and reporting requirements.
- Reporting Example:
- Item 1 weighed 25.93 ± 0.05 grams. Measurement uncertainty of weight measurements is reported at a 95.45% level of confidence.
- 21.2.5.2 It is rare that critical weights would be necessary. An example is when the weight of explosive powders is necessary to be reported for a Federal prosecution.

21.3 Chemicals and Reagents

- 21.3.1 The minimum acceptable quality level of chemicals and reagents (non-critical) has been determined through experience and these appear in each applicable section of this manual. The supplier of these chemicals and reagents is any supplier that can provide the specified quality level that meets the criteria for the specific extraction or test. Generally speaking, pre-approved manufacturers/vendors/suppliers are:
- Alltech Associates (Grace – Discovery Sciences)
 - J.T. Baker
 - Burdick & Jackson
 - Fisher
 - Mallinckrodt
 - Sigma-Aldrich and its subsidiaries

- VWR
- Government Scientific
- AccuStandard
- Cerilliant

- 21.3.1.1 The Section Supervisor, or in their absence the Chemistry Program Manager, will be responsible for approving the purchase of a chemical or reagent from a manufacturer/vendor/supplier that is not listed above.
- 21.3.2 Chemicals and solvents used in qualitative reagents should be of at least ACS reagent grade.
- 21.3.3 Solvents used for extractions or to dissolve samples or standards or as standards should be a high quality, low residue solvent (e.g., HPLC grade, OMNISOLV, OPTIMA).
- 21.3.4 Water used in reagent preparation or as an extraction solvent should be either deionized (DI) or reverse osmosis (RO).
- 21.3.5 Composition of prepared quality assurance check mixtures including, but not limited to, PCS and OCS shall be documented.
- 21.3.6 All chemicals and commercial reagents are labeled when received with the date received and the initials of the individual receiving them, and again when opened.
- 21.3.6.1 All chemicals and commercial reagents are placed onto an inventory list maintained either within the Section or within the laboratory.
- 21.3.6.2 Working bottles of chemicals and commercial reagents will be labeled at a minimum with the name of the chemical or commercial reagent, the manufacturer and lot number and the date dispensed into the working bottle.
- 21.3.6.3 All chemicals and commercial reagents are replaced when they fail to perform adequately under controlled conditions. Chloroform, acetone, sulfuric acid, nitric acid, and hydrochloric acid dispensed into working bottles for microchemical tests will be replaced approximately every three months.
- 21.3.7 All bottles of stock reagents prepared in-house must be labeled with the identity of the reagent, the date of preparation and the initials of the person who prepared the reagent. Reagent bottles for individual use must be labeled with the identity of the reagent and the preparation date.
- 21.3.7.1 The Department *Reagent Worksheet* shall be used to record reagent preparation. This will include the quality check to ensure that the reagent is functioning properly when it was prepared.
- 21.3.7.2 A stock bottle of reagent may continue to be used until the aliquoted reagent fails the quality check. If the quality check fails, it is the examiner's responsibility to immediately dispose of the remaining reagent and to communicate this to all examiners with working bottles of the reagent.
- 21.3.7.3 After the initial preparation, reagents will be checked at least once per each case analyzed. The results of the QC check are recorded in the case notes. Do not use the reagent if the quality check fails. A notation such as "QC ✓" is sufficient to document the reagent reacted as expected.
- 21.3.8 All other reagents used in non-routine examinations, such as general chemical type cases, are prepared as necessary and are tested with knowns and blanks, the results of which are recorded in the case file notes.

21.3.8.1 The *Reagent Worksheet* shall be used to record reagent preparation if the reagent will be retained for future use.

21.3.8.2 If a single-use reagent, a *Worksheet* is not necessary.

21.3.9 Test strips are checked with appropriate standards and results are recorded in case notes. Test strips may continue to be used past their stated expiration date as long as the QC check is positive.

21.3.10 Storage Requirements

21.3.10.1 Store Silver Nitrate reagent in the dark.

21.3.11 Reagent Recipes for Routine Examinations

Aqueous Aniline Sulfate: Dissolve 5.0 g aniline sulfate in 100 ml deionized water. (Ref. 2)

Barium Chloride: Dissolve 5.0 g barium chloride in 100 ml deionized water (5% solution). (Ref.1)

Brucine: Dissolve 5.0 g brucine sulfate (brucine sulfate 7-hydrate) in 100 ml concentrated sulfuric acid. (Ref. 1)

Diphenylamine: Dissolve 0.68 g diphenylamine in 45 ml concentrated sulfuric acid. Place in ice bath and cautiously add 22.5 ml glacial acetic acid. (Ref. 6)

LeRosen: Mix 75 ml of concentrated sulfuric acid in 1.5 ml of 37% formaldehyde. (Ref. 3, pp.137)

1-Naphthol: Dissolve 15 g 1-naphthol in 100 ml of ethanol (Ref. 2)

Nessler's: Dissolve 20 g potassium hydroxide in 50 ml deionized water. Dissolve 10 g mercuric iodide and 5 g potassium iodide in 50 ml deionized water. Mix together. (Ref. 4)

Silver Nitrate: Dissolve 3.75 g silver nitrate in 75 ml deionized water. (Ref. 1)

Sodium Hydroxide: 2.0 N: Dissolve 6.0 g NaOH in 75 ml deionized water. (Ref. 4, pp. 57, 64)

Saturated Sodium Hydroxide: Dissolve 10g NaOH in 100 ml deionized water (Ref. 2)

Sulfuric Acid (75%): Mix 56.25 ml concentrated sulfuric acid into 18.75 ml deionized water. (Ref. 7)

Triphenyl Selenium Chloride: Saturated solution in 60 ml deionized water. (Ref. 5)

21.3.12 Reagent Recipes for Non-Routine Examinations

Fehling's: Dissolve 7.5 g cupric sulfate in 100 ml deionized water. Dissolve 35 g sodium tartrate and 25 g potassium hydroxide in 100 ml deionized water. Mix equal volumes of each solution. (Ref. 3, pp.131)

Nitron: Dissolve 3.75 g nitron (diphenylenedianilohydrotriazole) in 75 ml of 88% formic acid. (Ref. 1)

Triphenyltetrazolium Chloride: Dissolve 0.38 g triphenyltetrazolium chloride in 75 ml deionized water. (Ref. 3, pp. 338)

21.3.13 References

- 21.3.13.1 Parker, R.G., Stephenson, M.O., McOwen, J.M, Cherolis, J.A., “Analysis of Explosives and Explosive Residues. Part 1: Chemical Tests”, *Journal of Forensic Sciences*, 1975, 20 (1), pp. 133-140.
- 21.3.13.2 “Spot Tests, Systematic Analysis Of Low Explosives”, Bureau of Alcohol, Tobacco, and Firearms, Rev 6/88.
- 21.3.13.3 Anger, V., and Feigl, F. Spot Tests In Organic Analysis 7th ed., Elsevier Publishing Company: Amsterdam, The Netherlands, 1966.
- 21.3.13.4 Jungreis, Ervin; “Spot Test Analysis, Clinical, Environmental, Forensic, and Geochemical Applications”, Volume 75, 1985, pp.57-58.
- 21.3.13.5 Anger, V., and Feigl, F., Spot Tests in Inorganic Analysis, 6th ed., Elsevier Publishing Company: Amsterdam, The Netherlands, 1972, p.184.
- 21.3.13.6 Thornton, John I., “Forensic Paint Examination,” Forensic Science Handbook, Volume 1, Prentice-Hall, Inc., 1982, p.550.
- 21.3.13.7 Hall, David, “Practical Fiber Identification”, Textile Engineering Department, Auburn University, 1976, p.36.

21.4 Critical Supplies

- 21.4.1 Critical supplies used within the Section include carbon disulfide and pentane used for fire debris extractions and primer residue kits which are manufactured to DFS specifications and provided to law enforcement agencies throughout the Commonwealth.
- 21.4.2 Generally speaking, suppliers of carbon disulfide and pentane which have previously been checked and deemed to be acceptable are those which are used for future orders. Pre-approved manufacturers/vendors/suppliers are: Fisher, J.T. Baker, Burdick & Jackson, Mallinckrodt and VWR.
- 21.4.2.1 As a check, an appropriate amount of solvent from each lot is concentrated by evaporation and analyzed by GC-MS. The resulting data is maintained in an appropriate location within the Section. Do not use the solvent until this check is acceptable (no major interfering peaks in the region of interest). See Section 4.1.11 for more information regarding the quality checks.
- 21.4.3 Primer residue kits are only ordered through the company who has been awarded the contract for their manufacture. The tape used to prepare the kit sampling devices as well as the completed kits are checked prior to release for distribution. See Section 18.6.
- 21.4.4 Standards used for identification in casework are considered critical supplies and shall be purchased from manufacturers/vendors/suppliers approved by the Chemistry Program Manager.
- 21.4.4.1 The following manufacturers/vendors/suppliers are pre-approved:
- Alltech Associates (Grace – Discovery Sciences)
 - Fisher
 - Kodak
 - Mallinckrodt
 - Pierce Chemical
 - Sigma-Aldrich and its subsidiaries
 - VWR

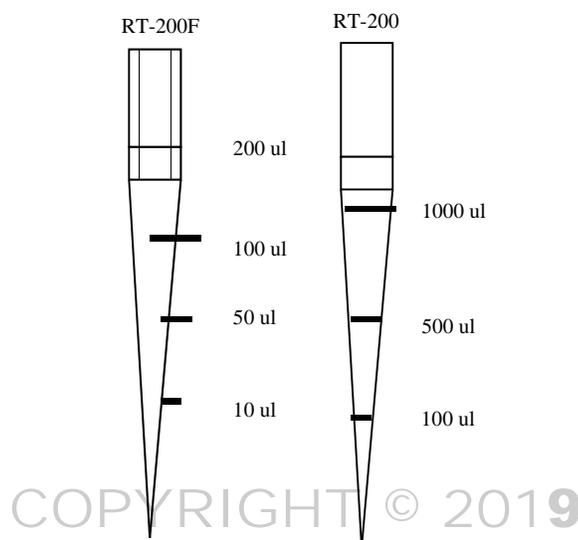
- 21.4.4.2 Standards used for identification purposes are verified each time an examiner analyzes them with case evidence.
- 21.4.4.2.1 It is the examiner's responsibility to ensure that the standard being analyzed agrees with known published data or that the data is consistent with the unique compound represented from both a chemical and data interpretation perspective.
- 21.4.4.3 The Section Supervisor, or in their absence the Chemistry Program Manager, will be responsible for approving the purchase of a chemical or reagent from a manufacturer/vendor/supplier that is not listed above.
- 21.4.4.4 All standards used for identification purposes will be identified to a unique source to include at a minimum the manufacturer and lot number of the material.

21.5 Eppendorf Pipettes

- 21.5.1 The Section has a number of fixed volume Eppendorf pipettes that are not used for critical measurements in analytical work. These pipettes do not require a calibration check of the accuracy and precision of the volume that they dispense.
- 21.5.2 The Eppendorf variable pipettes are used in preparing standards and samples for analysis by Ion Chromatography (IC). Any of these solutions or dilutions is meant to only be comparative in nature on a semi-quantitative basis.
- 21.5.2.1 Annually – Eppendorf variable pipettes shall be calibrated by an outside vendor that is accredited to ISO/IEC 17025:2005 and whose scope of accreditation covers the calibration performed.
- 21.5.2.2 As Needed – If an Eppendorf variable pipette has been dropped or otherwise damaged, the calibration of the pipette will be checked using graduated Rainin Pipette RT tips. Verify the pipetting volumes of the pipette as specified in the Pipette Verification Table below. The pipette tips are guaranteed to be accurate to $\pm 3\%$ at the specified reference lines. Should the pipette be visibly incorrect according to the reference line, the pipette will be sent to the manufacturer for repair.
- 21.5.2.3 As Needed – Clean and check the sealing system as outlined in the pipette's instruction manual.
- 21.5.2.3.1 Once the pipette has been reassembled after cleaning, conduct a calibration check using graduated Rainin Pipette RT tips. Verify the pipetting volumes of the pipette as specified in the Pipette Verification Table below. The pipette tips are guaranteed to be accurate to $\pm 3\%$ at the specified reference lines. Should the pipette be visibly incorrect according to the reference line, the pipette will be sent to the manufacturer for repair.

Pipette Verification Table

Pipette	Tip RT-200F			Tip RT-200		
	10 μ L	50 μ L	100 μ L			
10-100						
100-1000				100 μ L	500 μ L	1000 μ L



21.6 Digital Calipers

Digital calipers shall be calibrated every three (3) years by an outside vendor that is accredited to ISO/IEC 17025:2005 and whose scope of accreditation covers the calibration performed. Gauge blocks used to check digital caliper accuracy shall be re-certified every three (3) years by an ISO/IEC 17025:2005 accredited vendor whose scope of accreditation covers the certification performed.

- 21.6.1 Digital calipers shall be checked prior to use in casework, however, if the caliper has been previously checked during the given month, the caliper does not need to be re-checked. Calibration checks are made with the following five (5) certified steel standard gauge blocks: 0.050", 0.250", 0.500", 1.000" and 2.000".
- 21.6.2 The measured values are recorded on a log sheet which is stored with the digital calipers.
- 21.6.3 The measured values shall be within: ± 0.02 mm or 0.001".
- 21.6.4 If the values are outside the appropriate range for a given gauge block, then measure again. If the values are still outside the appropriate range, then the digital caliper must be sent to a vendor that is accredited to ISO/IEC 17025:2005 and whose scope of accreditation covers the calibration performed for recalibration.
- 21.6.5 If any digital caliper is dropped or otherwise subjected to extremes of temperature, shock or contamination, then the calibration will be checked prior to use in casework measurements.

21.7 Microscopes

- 21.7.1 All the microscopes used within the Section are cleaned and serviced every two years, and as needed. A log documenting cleaning and service is maintained.
- 21.7.2 Lenses are cleaned during use as needed. These include condenser lenses, objective lenses and eyepieces. Miscellaneous microscope maintenance comments are recorded as appropriate.
- 21.7.3 Setting up Köhler illumination is one of the most widely accepted techniques of ensuring that a microscope is correctly aligned, that it has high intensity homogenous illumination and that it is focused correctly. It should be noted that this does not apply to stereomicroscopes.

- 21.7.3.1 Depending on a particular microscope and its design, setting up Köhler illumination may be possible in full or only in part. Many modern microscopes are fitted with fixed light sources, which mean that it is not possible to center or focus the image of the lamp filament like in conventional microscopes. Also, many modern microscope designs include one or more diffuser filters between the lamp and the condenser. In such cases, even if the microscope is fitted with an adjustable lamp, an image of the filament cannot be seen. In these microscopes only partial Köhler illumination can be obtained.
- 21.7.3.2 Köhler illumination is a multi-step process, which should be performed or checked every time the microscope is used. Some of the steps should even be checked more often while working.

21.8 pH Meter

- 21.8.1 The pH meter is calibrated prior to each use using a 3-point calibration method. Refer to the individual pH meter's instrument manual for these procedures.
 - 21.8.1.1 The reference buffers chosen should bracket the expected pH value range of the sample(s) to be analyzed, if possible.
 - 21.8.1.2 The electrode slope must be within the range: 92%-102%. While this is the range cited for 2-point calibrations, it will also be applied here to the 3-point calibration method.
 - 21.8.1.3 Record the pH values on the pH meter calibration log sheet. The pH values must be within ± 0.1 units of the pH value stated on each individual reference buffer's labeling.
 - 21.8.1.4 If the calibration values are within the accepted limits, the pH meter is ready to use for samples.
 - 21.8.1.5 If the calibration values are not within the accepted limits, rerun and/or troubleshoot as appropriate.
- 21.8.2 Rinse the electrode with deionized water or the buffer, standard or sample, as appropriate.
 - 21.8.2.1 Inspect the electrode for scratches, cracks or salt crystal deposits prior to each use. Clean or replace the electrode in response to the discovery of these deficiencies, as appropriate.
 - 21.8.2.2 Do not wipe the pH electrode glass bulb.
 - 21.8.2.3 Short term storage is defined as being less than one week.
 - 21.8.2.4 For short term storage: Store in an electrode storage bottle containing pH storage solution or in a covered bottle of filling solution. Covered glassware containing sufficient storage or filling solution to cover the sensing element may be used.
 - 21.8.2.5 For long term storage: Fill the reference chamber with filling solution and cover the fill hole. Put a few drops of pH storage solution into an electrode protective cap and cover the sensing element and reference junction. Alternatively, store in a covered bottle of filling solution. Some models require covering the sensing surface with the protective cap and storing dry.
 - 21.8.2.6 If the reference chamber dries out during storage, rinse with deionized water several times using a plastic pipette. Follow by rinsing several times with filling solution. Refill the reference chamber, cover the fill hole, and soak the electrode in storage or filling solution for at least one hour.
 - 21.8.2.7 Do NOT store the electrodes in deionized water.

- 21.8.3 Reference buffers shall be replaced when they expire.
- 21.8.3.1 Choose pH buffers that are no more than three pH units or no less than one pH unit apart.
 - 21.8.3.2 Keep the buffer bottle tightly sealed and free of contamination.
 - 21.8.3.3 Do not reuse an aliquot of buffer or return it to the original bottle.
 - 21.8.3.4 Pure water samples (e.g., deionized water blanks) may result in noisy electrode response and may require a long stabilization period particularly if used after a buffer with high ionic strength. The sample may also be contaminated if adequate rinsing is not performed.
 - 21.8.3.5 Refer to the pH meter instrument manuals for recommendations of good laboratory practices, correct applications, problem samples and troubleshooting.

21.9 Refrigerators and Freezers

- 21.9.1 At the beginning of each work week, check and record the temperatures on all refrigerators currently in use for storing evidence. The temperature of the refrigerator should be between 2-8° C. If the temperature is outside this range, document in case file notes and immediately notify the Section/Group Supervisor who will remedy this situation. The thermometers used for the temperature check will be NIST traceable and their certificates stored with the refrigerator/freezer log. Thermometers shall be replaced prior to their calibration due date (typically every two years).
- 21.9.1.1 If evidence is submitted frozen and must be stored frozen, check and record the temperatures on all freezers currently in use for storing evidence. The temperature of the freezer should be below -10°C.
- 21.9.2 Some standards and reagents are labeled with specific storage temperatures and should be stored accordingly. Some reference materials and reagents last longer when refrigerated. Because all standards, reagents and reference materials are checked with each case, a check of the storage refrigerator/freezer temperature is not required.

21.10 Thermometers and Ovens

- 21.10.1 The temperature of ovens used in passive adsorption-elution fire debris extractions (charcoal strip) will be checked prior to use with casework and the value recorded on a log sheet kept with the ovens.
- 21.10.1.1 Each oven should have its own external thermometer. The oven temperature should read 65° C \pm 2.0° C. If the temperature does not read as specified, then the thermostat will be adjusted to obtain the correct temperature. The oven temperature will be re-read and this value recorded. If the correct temperature cannot be achieved, the oven will be removed from service until it can be repaired.
 - 21.10.1.2 The thermometers used for the temperature check will be NIST traceable and their certificates stored with the oven log. Thermometers shall be replaced prior to their calibration due date (typically every two years).
 - 21.10.1.3 Record the serial number of the NIST traceable thermometers corresponding to each oven on the oven log.
- 21.10.2 Any maintenance or repair will be documented in a log kept with the oven.

21.11 [Handheld UV Source](#)

21.11.1 Fluorescence QC Procedure:

21.11.1.1 Perform fluorescence QC check prior to using UV source for casework using a suitable fluorescent highlighter. Place a mark on a glass slide using the highlighter to verify proper operation of the UV source and record resulting fluorescence in notes. The prepared slide can be stored out of direct light and can be used for future QC checks. If the QC check fails, troubleshoot equipment and repeat QC check. If the UV source fails to produce fluorescence on known fluorescent surfaces, take the UV source out of operation until suitable repairs are made. For glass analyses please see the “Glass” section of this manual.

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22 COMMONLY USED ABBREVIATIONS

The following is a list of abbreviations commonly used by examiners in the Section. This list has been generated to assist in the interpretation of case file notes. While as comprehensive as possible, the list may not be complete. The abbreviations are appropriate written in either lower or upper case and they are appropriate with or without punctuation such as periods. It is the examiner's responsibility to ensure that there is no confusion in the note-taking when using abbreviations. Common symbols may also be used to represent their common context.

@	at
~ or ≈	approximately; equivalent or similar
Δ	change(s); change in; heat
⇒ or →	contains; containing; reaction goes to; taken to dryness
≠	does not equal; not same as
↑	high
↑↑↑	very, many, abundant
“ ”	labeling; anything enclosed in quotes is word for word from the package labeling
μ or μm	micron; micrometer (applies to microgram and microliter as well; a lower case u is equivalent)
μchems	microchems (microchemical tests)
μscopic	microscopic
- or θ	negative
	parallel
⊥	perpendicular
+ or ⊕	positive
↓	low
↓↓↓	very few, sparse
∴	therefore; thus
λ	wavelength
1,2,4-TMB	1,2,4-trimethylbenzene
IPS	phase separating filter paper (e.g., Whatman IPS)
ACA	Assistant Commonwealth's Attorney
AH	animal hair
app	apparent
ASV	auto sampler vial
BC	bar code
B/E	back end
bf	body filler
bl'd	blood
BPB	brown paper bag
BM	best match
BSE	backscattered electron
BX	box
CA	Commonwealth's Attorney
CB	call back
c/c	clear/colorless
Char	characteristic
CLP	one plastic container which contained one glass container which contained
C of A or CoA	Certificate of Analysis
comp	compensator
cont.	container
conv. bag	convenience bag
conv. pkg.	convenience package
Cryst	crystalline
c.s. or c/s	cross section
CSD	County Sheriff's Department
CSO	County Sheriff's Office
ct	can't tell

D	dissolve
DF	decorative flake
dH ₂ O or DI water	deionized water
dk	dark
dia.	diameter
DC or (DC)	diamond cell
DPA	diphenylamine
DMF	dimethylformamide
d/t	due to
dws	deep welled slide
eff.	effervescence
EN, ENV	envelope
esc	escape
ext	extract
FA	falls apart
F/E	front end
FDCS	Fire Debris Check Sample
f/n or fn	finger nail(s), rfn, lfn = right/left fingernails
frag(s)	fragment(s)
fx'd	fractured
G	glass
H ⁺	acid
h	horizontal
H/F	hairs/fibers
H/F/K	hairs/fibers/known
hort.	horizontal
h/s	heat sealed
I	insoluble
i.d.	inner diameter
ID	identification
J chem.	section J chemical
K	known
LM	left message
LVM	left voice mail
lt	left or light
m	metallic
mag	magnification
MC	metal can
MCDC	microcompression diamond cell
med	medium
MEN	manila envelope
ME	Medical Examiner
MEO	Medical Examiner's Office
Meth	methamphetamine
mic	microscopy
misc.	miscellaneous
morph	morphology
m/s	microscope slide(s)
mm	mounting medium
MT	empty
NCC	no color change
NCR	no color reaction
no CS	no controlled substances identified
NDF	no decorative flake
neg	negative
nm	nonmetallic
NNA	not necessary for analysis

NPF	no particles found
NR	no reaction
NSR	no significant reaction
o.d.	outer diameter
OOR	out of range (matchpoint out-of-range at given temp.)
o.p.	orange peel texture
PCS	Polar Check Sample
p	primer
PB	paper bag
PET	polyethylene terephthalate
pkging	packaging
pl	plastic
PLB	plastic bag
poly	polyester
pos	positive
ppt.	precipitate
PR	primer residue
Pseudo	ephedrine/pseudoephedrine
Q	questioned
QNS	quantity not sufficient for analysis
rec'd	received
RI	refractive index
r/o	rule out
R _x	reaction
RXN	reaction
RT +	retention times positive for
rt	right
S	softens
S&S	softens and swells
S/	sealed _ (e.g., S/EN is a sealed envelope)
S's	suspect's
Scope	microscope
Sec J	section J chemical
sig	significant
sl	slight
SM	solid material
SP	smokeless powder
STD	standard
sub	submission
syn	synthetic
T°	temperature
T _x	transferred to
Tab(s)	Tablet (s)
telo	telogen
tiss	tissue
TPS Cl	triphenylselenium chloride
TS, t/s	tape sealed
v.	very
var	variable
vert.	vertical
V's	victim's
VM	voice mail
VMX	voice mail
VSP	Virginia State Police
w/	with
WEN	white envelope
w/o	without

wk	weak
WPB	white paper bag
wrt	with respect to
x-polars	crossed polars
X	times (3X or X3 means 3 times)
XT	extract
x-section	cross-section
YEN	yellow envelope
z/l	ziplock
zplb	ziplock plastic bag

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