Trace Evidence Methods Manual

Contents

1 Purpose 5
2 Examination Guidelines 6
2.1.1 General Examination Approach 6
2.2 Documentation 9
2.3 Verifications 9
2.4 Safety Considerations 10
2.5 Instrumentation/Equipment 10
2.6 References 10
3 Microscopy 10
3.2 Instrumentation / Equipment 10
3.3 Procedure 10
3.3.1 Maintenance 10
3.3.2 Illumination 10
3.3.3 Use of Compensators 10
3.4 References 11
4 Fourier Transform Infrared Spectroscopy (FTIR) 12
4.2 Safety Considerations 12
4.3 Minimum Standard and Control 12
4.3.1 Month of use: 12
4.3.2 Day of use: 12
4.4 Equipment/Instrumentation 12
4.5 Analytical method 13
4.5.1 Bench 13
4.5.2 ATR 13
4.5.3 Microscope 13
4.5.4 Diamond Anvil Cell (DAC) 13
4.6 Sample preparation for transmission/ATR/microscope: 13
4.7 Spectral Evaluation and Comparison 14
4.8 Interpretation Criteria 14
4.9 References 14
5 Visible Microspectrophotometry (MSP) 16
5.2 Equipment/Instrumentation 16
5.3 Minimum Standard and Control 16
5.4 Sample Preparation Procedure 17
5.5 Analysis Method 17
5.6 Spectral Evaluation and Comparison 17
5.7 Interpretation Criteria 17
5.8 References 18
6 Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy (SEM/EDS) 19
6.2 Safety Considerations 19
6.3 Equipment/Instrumentation 19
6.4 Minimum Standards and Controls 20

6.5 Sample Preparation Procedure 20
6.6 Analysis Method 22
6.6.1 Imaging analysis....23
6.6.2 Bulk analysis 23
6.6.3 Individual component analysis....23
6.6.4 Analysis of a primarily organic matrix 23
6.6.5 Qualitative analysis....24
6.6.6 Quantitative analysis 25
6.7 Spectral Evaluation and Comparison 25
6.8 Interpretation Criteria 25
6.9 References....25
7 Physical Break Match/Tear Configuration Comparisons 27
7.2 Equipment/Instrumentation 27
7.3 Minimum Standards and Controls 27
7.4 Procedure 27
7.5 Interpretation Criteria 27
7.6 References....28
8 Impression Comparisons....29
8.1 Equipment/ Instrumentation 29
8.2 Minimum Standards and Controls 29
8.3 Analysis Method....30
8.3.1 Enhancement Procedures 30
8.3.2 Physical....31
8.3.3 Chemical 31
8.3.4 Potassium and Ammonium Thiocyanates....31
8.3.5 Leucocrystal Violet (LCV)....31
8.4 Lifts 31
8.4.1 Gelatin Lifters 31
8.4.2 Electrostatic Lifts 31
8.5 Test Impression Procedures....32
8.5.2 Vaseline and magnetic fingerprint powder:....32
8.5.3 Lightning lifts with fingerprint powder: 32
8.5.4 Identicator kit 32
8.5.5 Footwear Database 32
8.6 Interpretation Criteria 33
8.7 References....34
9 Fiber and Fabric Analysis....35
9.1 Introduction....35
9.2 Equipment and Instrumentation....35
9.3 Minimum standards and controls 35
9.4 Analysis Methods 36
9.4.2 Determination of refractive index relative to mounting medium 37
9.4.3 Estimating Birefringence with a Berek Compensator 37
9.4.4 Preparing Cross-Sections....38
9.4.5 Maceration process for the release of fiber ultimates from natural fiber samples: 40
9.4.6 Solubility testing....41

9.4.7 Infra-Red Spectroscopy (FTIR) Analysis 44
9.4.8 Microspectrophotometry (MSP) Analysis 45
9.4.9 Fabric Comparison 45
9.4.10 Fabric Damage 46
9.5 References Fiber Comparisons 47
10 Paint Analysis 49
10.1 Introduction 49
10.2 Equipment/Instrumentation 49
10.3 Minimum Standards and Controls 49
10.4 Analysis Methods 49
10.4.2 Chemical reactivity (Spot Tests) 49
10.4.3 Instrumental analysis techniques 51
10.5 Paint Data Query (PDQ) 51
10.6 Interpretation Criteria 51
10.7 References 52
11 Gunshot Primer Residue Analysis 53
11.1 Introduction 53
11.2 Equipment/Instrumentation 53
11.3 Minimum Standards and Controls 53
11.4 Sample Prep Procedures 54
11.4.2 Analytical progression 54
11.5 Testing Parameters 55
11.6 Data Collection 56
11.7 Interpretation Criteria 56
11.8 References 57
12 Glass Analysis 59
12.1 Introduction 59
12.2 Minimum Standards and Controls 59
12.2.2 Day of Use: Refractive Index Known Reference samples 59
12.2.3 Day of Use: Micro-XRF Energy Calibration 60
12.2.4 Day of Use: Micro-XRF Source Performance Check 60
12.2.5 Day of Use: Micro-XRF System Performance Check 60
12.2.6 Micro-XRF Run Stability 60
12.2.7 Micro-XRF Control Samples 61
12.3 Equipment/Instrumentation 61
12.4 Analysis Methods 61
12.4.2 Refractive Index analysis using Craic Technologies rIQ 62
12.4.3 Elemental analysis 63
12.4.4 Direction of Force 64
12.5 Interpretation Criteria 65
12.6 References 65
13 Vehicle Lamp Examination 68
13.1 Introduction 68
13.2 Safety Considerations 68
13.3 Equipment/Instrumentation 68
13.4 Procedure 68

13.4.2 Interpretation Criteria 69
13.5 References 69
14 Pressure Sensitive Tape Analysis 70
14.1 Introduction 70
14.2 Minimum Standards and Controls 70
14.3 Equipment/Instrumentation 70
14.4 Sample Preparation 70
14.5 Analysis Methods 70
14.5.2 Microscopic Examination 71
14.5.3 FTIR Analysis 71
14.5.4 SEM/EDS and XRF Analysis 71
14.6 Interpretation Criteria 71
14.7 References 72
15 Miscellaneous Examinations 73
15.1.1 Equipment/Instrumentation 73
15.1.2 Analysis Methods 73
15.2 Interpretation Criteria 73
15.2.1 References 74
16 Suggested Report Wording 75
16.1 Method of Testing 75
16.2 Findings 76
16.3.1 Qualifying Statements 79
16.5.1 Example 1 Physical Break Match/Tear Configuration Comparisons 82
16.5.2 Example 2 Impression Comparison 83
16.5.3 Example 3 Fiber and Fabric Analysis 85
16.5.4 Example 4 Paint Analysis 87
16.5.5 Example 5 GSR 89
16.5.5 Example 6 Glass Analysis 90
16.5.6 Example 7 Vehicle Lamp Examination 91
16.5.7 Example 8 Pressure Sensitive Tape Analysis 92
17 Appendix I: Trace Evidence Note Abbreviations 94

1 Purpose

The purpose of this manual is to provide the Trace Evidence Examiner with a set of standard, recognized methods for the examination of physical evidence. The methods and practices described apply to casework, proficiency tests and competency tests and may be applicable in other situations, as determined by laboratory management. Deviations from written methods and conventions are at times necessary and are permitted as circumstances dictate. Significant deviations from the methods provided in this manual must be approved by laboratory management and must be accurately reflected in the analyst's notes.

2 Examination Guidelines

2.1 Case Approach

The analyst should be familiar with the case information prior to beginning the analysis. The choice of which items to examine first and which tests to use is based on the answers provided in regards to some basic questions:

- What information of fact can be established by the evidence?
- How does this information fit into the investigation?
- Where was the evidence found?
- How unique is the evidence?

When the analyst has a good understanding of what the investigator believes occurred during the crime, he or she can begin to see where the pieces of physical evidence fit into the investigation.

Certain items may have greater potential value for information than others. The analyst may want to examine these items first. This concept also applies to items which may have potential value as "investigative information" (information which may be useful to the investigator if presented while the investigation is pursued).

It is important to know if evidence was found in some place which is already associated with the victim or suspect. For example, finding paint from a scene on a tool left at the scene that has no association to a suspect may not be significant. Finding paint from the scene on a tool in the vehicle of a suspect may have significance. If fibers like those comprising the victim's clothes and fibers like those comprising the suspect's clothes are found in a location foreign to both, the evidence suggests both may have been in that location.

The types of trace evidence most commonly encountered in the crime laboratory include fibers, paint, glass, gunshot primer residue, impressions, tape, vehicle lamps for on/off determination and potential physical break or tear configurations. This evidence is often used to associate a person with a place, a victim and a suspect or an object which provides a link between people, places or both. Identifying the composition of an unknown material is important for collection of an appropriate standard for comparison. The identification of the unknown is often difficult due to the limited quantity available. However, the analyst can develop the ability to recognize a vast assortment of materials he or she has personally seen, particularly if a large, diverse reference collection is available.

Due to the nature and/or quality of the evidence examined, class characteristics play an important role in trace evidence both for elimination and association purposes. When possible, individual characteristics present in evidence are also used to draw a conclusion.

The usefulness of a reference collection should not be underestimated. There are various sources of useful reference standards and it is recognized that, in-house standards and collections may aid the identification process.

2.1.1 General Examination Approach

Once the analytical approach has been determined, the analyst should begin the examination in the following manner:

1. Properly mark all evidence packaging in accordance with accepted laboratory practice.
2. Documentation of the examination process will commence at the opening of the case and continue throughout. This includes an adequate description of the item, which may be aided with photography

and / or sketches, as well as the items general condition when applicable. Examination documentation must meet all requirements as described in the Laboratory Quality Assurance Manual.

3. Remove packaging with care, remembering that materials of evidential value may be adhering to the item. Opening the evidence over clean catch paper will prevent the loss of these materials.
4. Mark the evidence itself for future identification in accordance with accepted laboratory practice taking care not to cause detriment to other possible types of evidence (e.g. latent prints, DNA/FB).
5. Perform the necessary examinations including collecting, preserving and properly marking any items isolated for possible future testing. Questioned items will be evaluated for appropriate comparison suitability before a comparison.

6. Before subsequent evidence is examined in the same area, insure there is no threat of contamination by making certain the area is clean. This will usually include changing the catch paper between each exhibit.

2.1.1.1 Items not dried before submission

If the analyst discovers that an item is received in a wet condition, steps must be taken to minimize any potential evidentiary damage that may occur. The item must be dried as soon as possible. Dry the items to prevent loss, cross transfer, contamination and/or deleterious change to the evidence. These include natural air drying, or placing items in an exhaust hood or another vented location with the exhaust fan running sufficient to remove odors and moisture, but prevent trace evidentiary loss. Wet items should not be heated, nor should a direct fan be used in an attempt to accelerate drying. Items may be spread out on a flat surface or hung.

Caution must be exercised to prevent the loss of evidential material during the drying process. When clothing or other items with potential trace evidence require removal from packaging, drying will take place on or over an adequately sized sheet of clean catch paper Care will be taken to collect any loose trace evidence from the catch paper when the dried item is collected for examination. If the item is to be examined at a later date, it should be re-packaged in, or with, that paper to prevent the inadvertent loss of trace evidence during handling.

2.1.1.2 Items infested with vermin

Fleas, lice, and insect larvae may be discovered while examining various objects. If such infestation occurs, the following steps should be taken:

The infestation may be eradicated prior to analysis if the procedure will not compromise the analysis to be performed. This may be accomplished in a variety of manners, including: exposing the infested item(s) to dry ice in a sealed environment; the use of appropriate insecticides; etc.

If eradication is not possible, proceed as follows:

1. Wear gloves and a lab coat or other appropriate garment as necessary.
2. Examine the item carefully in an isolated area, if possible.
3. Examine the object as quickly as possible, take samples of evidential material, and completely seal the object within a plastic bag using tape or a heat seals.

2.1.1.3 Surface debris

Trace evidence is often present on an evidence item in the form of surface debris. This debris may have been transferred by direct contact with another item or from the collection of random debris over time and often originates from more than one source. Surface debris may consist of hairs, fibers, paint, wood fragments, glass, plant material, soil, and many other materials. Surface debris, by nature, is often transient. Care must be exercised in order to avoid loss during the examination process. It is generally advisable to collect and preserve surface debris prior to subsequent examination. Exceptions do exist, however. Method of debris removal and preservation, as well as the best time for removal during the course of the examination must be evaluated in terms of its effect on other potential Trace Evidence or testing by laboratory sections.

Surface debris removal and preservation:

Any of the following removal methods are permissible:

- Visible materials may be removed by clean gloved hand or forceps
- Tape lifts
- Shaking or scraping over an adequately sized sheet of clean paper

Vacuum sweeping is not generally a recommended method of collection. However, in some instances it may prove useful.

Preservation

Isolated materials will be placed in paper bindles and sealed in envelopes, pill boxes, or other suitable containers. Tape lifts are normally affixed to clear acetate. Paper containing shakings/scrapings will be folded and packaged in suitable containers. Debris containers will be sealed and marked and treated as evidence, as per BCI laboratory protocol.

2.1.1.4 Examination of weapons

Weapons may consist of knives, guns, bottles, baseball bats, tools, and numerous other items. Weapons are submitted frequently for fibers, tape, fracture match and other trace evidence examinations. The trace examiner must be aware of the possibility that latent prints or biological evidence may be present on the weapon. Caution must be exercised and interaction with other sections and the submitter may be required to determine analytical approach.

Firearms must be handled in accordance with established policy to insure the safety of the examiner.

When describing microscopic evidence located on various types of weapons, every effort should be made to use the correct nomenclature for the parts of the weapon.

2.1.1.5 Examination of clothing

Clothing is often submitted to the laboratory for examination. In most cases, these items will be dried before submission. Process items of according to the procedures previously outlined involving the removal of surface debris, as appropriate. Examine any cuffs or folds and turn pockets inside out and collect debris, as appropriate. Exercise caution when placing a hand into a pocket, since an unexpected sharp object could cause serious injury, and/or infection.

2.2 Documentation

In addition to the technical record requirements detailed in the Laboratory Quality Assurance manual, the following case documentation practices should be used whenever appropriate:

Photographs of evidence may also be taken to document the physical condition of evidence.
Photographs may be used to document comparison results which include individualizing characteristics that support the examiner's conclusion. A scale or ruler will be incorporated in photographs when the standardization of item size is relevant. Annotation of photographs should include at minimum the case number, item number, date, examiner initials and, if applicable, the magnification at which the photograph was taken. If the photograph serves as justification for a conclusion, the conclusion should be annotated on the photograph. The photographs will be included in the case record.

Evidence in digital format is stored utilizing a secure software program (e.g., LIMS) or secure hardware. Alternately, it may be printed or burned to an optical disc and then packaged, sealed and labeled appropriately for retention or to be returned to the submitting department. Burned discs will be tracked as sub-items in LIMS and barcoded.

Data produced during an examination may be rejected. If data is rejected, the case record will include the reason the data was rejected, the date the data was rejected and the person rejecting the data.

Case notes should include a description of the evidence analyzed, the method of sample preparation, the analytical instrumentation used, and its operating parameters, whenever applicable.

Case notes should include a copy of all of the instrumental data that was used to reach a conclusion.

2.3 Verifications

Conclusions established through comparison of impression (excluding negative footwear database searches) require verification by a second qualified examiner prior to final report release. Positive identifications established through comparison of fracture/tear evidence require verification by a second qualified examiner prior to final report release. Verifying examination is performed without any expectation of results by the confirming examiner.

Verification may be performed through direct evidence examination or examination of sufficiently registered images, copies, etc. Verification shall be recorded by the verifier in the original examiner's case notes and will include the date of the verification, the verifying examiner's initials (or electronic equivalent), and the outcome of the verification. If the verification results in changes to the notes, the reason for the changes will be documented including the date of the changes and the individual making the changes.

Disagreement between the original examiner and the second examiner will follow the Discrepancy Policy as defined in the Laboratory Quality Assurance Manual.

2.4 Safety Considerations

Standard laboratory safety practices apply to all methods described in this manual (see the Laboratory Safety Manual).

2.5 Instrumentation/Equipment

Examination of trace evidence requires the use of a variety of high precision hand instruments. These include, but are not limited to:

- Scalpel blades and handles of various styles
- Fine and coarse forceps/tweezers
- Probes, needles and scissors
- Appropriate rulers
- Hand magnifier or eyepiece magnifying loop
- Digital or manual micrometers

These instruments should be of appropriate quality and kept in good condition in order to perform the fine manipulation that is required in the examination of trace evidence.

2.6 References

1. Scientific Working Group on Materials Analysis “Trace evidence quality assurance guidelines”, Forensic Science Communications (January 2000).
2. Scientific Working Group on Materials Analysis. Trace evidence recovery guidelines, ForensicScience Communications (October 1999).

3 Microscopy

3.1 Introduction

The microscope is an importation tool used for the characterization, identification or comparison of trace evidence. Polarized Light Microscopy is a specialized application method of microscopy.

3.2 Instrumentation / Equipment

- Stereomicroscope
- Compound microscope
- Comparison microscope
- Microscope compatible camera
- Both permanent and temporary mounting media
- Glass microscope slides
- Cover slips
- Optical filters
- Quarter-wave plate
- Full-wave plate
- Quartz wedge
- Berek compensator

3.3 Procedure

For comparisons using a microscope, the questioned sample will be evaluated for adequate quality prior to the comparison to be conducted.

3.3.1 Maintenance

In general, the optical pathway of the microscope should be free of dust, dirt, debris, etc. Simple cleaning of optical components may be affected with alcohol and lint-free wipes, compressed air, or other methods that would leave the optics free of contaminants or structural damage.

Microscopes should periodically receive professional cleaning and general maintenance. Any vendor-provided cleaning, maintenance, and repair will be documented in a microscope maintenance log.

3.3.2 Illumination

Microscopes should be optically *aligned* to provide appropriate and adequate illumination. Under most circumstances this will be Köehler illumination for transmitted light microscopes. Köehler illumination is a specific microscope alignment which optimizes intense light for specimen examination. Correct alignment ensures that the entire optical system, including the objectives, stage, condensers and light source, are all aligned so as to focus maximum light on the sample and reduce background light scattering.

3.3.3 Use of Compensators

The quarter-wave, full-wave, quartz wedge, and Berek compensators all work by aligning the slow direction of the compensator, indicated by an arrow on the device, with either the fast or slow direction of the specimen. By using these compensators and the appropriate charts, one can often determine the birefringence and optical sign of specimens.

3.4 References

1. Eastman Kodak, compiler, "Photography Through the Microscope", 8th ed.; Eastman Kodak: Rochester, NY, 1985; Publication P-2.
2. Saferstein, Richard, Editor, "Forensic Science Handbook", Prentice -Hall Inc., Englewood Cliffs, N.J., 1982.
3. Smith, R. F., "Microscopy and Photomicrography", 2nd ed.; CRC: Boca Raton, FL, 1994.
4. Thomson, D. J.; Bradbury, S., "An Introduction to Photomicrography"; Oxford University: New York, 1987.
5. McCrone, W. C., McCrone, L. B., Delly, J. G., Polarized Light Microscopy, Ann Arbor Science, Ann Arbor, MI, 1978.
6. McCrone, W. C., Delly, J. G. Particle Atlas Volumes I-IV Ann Arbor Science, Ann Arbor, MI, 1973.
7. McCrone, W. C., Delly, J. G., Palenik, S Particle Atlas Volume V Ann Arbor Science, Ann Arbor, MI, 1979.
8. Saferstein, R. Ed. Forensic Science Handbook, Vol. I Chapter 9, "Foundations of Forensic Microscopy" Prentice Hall 1982.
9. Saferstein, R. Ed. Forensic Science Handbook, Vol. II Chapter 4, "Microscopy and Microchemistry of Physical Evidence" Prentice Hall 1988.

4 Fourier Transform Infrared Spectroscopy (FTIR)

4.1 Introduction

FTIR analysis is non-destructive and can provide important information related to the chemical based on characteristic absorbance of infrared energy. This information can be used in the classification of the sample and as a means of comparing the chemical structure of two samples.

Common sample types include, but are not limited to:

- Paint
- Fiber
- Other polymer
- Powder
- Oil/grease

4.2 Safety Considerations

When using the FTIR microscope, use personal protective equipment to cover eyes and skin while working with liquid nitrogen. Do not look directly into the laser.

4.3 Minimum Standard and Control

The following quality checks are run according to the schedule prior to conducting analyses on case samples. The results of these quality checks will be recorded in a log. The instrument data should be retained in accordance to the laboratory retention schedule.

Routine maintenance and repair must be recorded in a specified log.

4.3.1 Month of use:

- Modern FTIR software often comes equipped with built in automated calibration and / or performance checks. This procedure will be run in accordance with manufacturer's instructions.
- To insure wavelength calibration a polystyrene standard will be run on the instrument under normal operating conditions and for all analytical methods (microscope / bench / bench ATR/etc.) that will be used. The spectra will be compared to a corresponding spectra taken earlier in the instruments life run under the same conditions using the same method.

4.3.2 Day of use:

- The energy through-put of the instrument will be checked. To be useful this needs to be done under the same parameters as previous checks. Example: If the FTIR microscope's energy is checked using a 100um aperture it should continue to be checked at that aperture in order to give a consistent frame of reference.
- It is normal to see the energy value drop with time. There is no specific number or amount that will specifically cause the instrument to be taken out of service. This check allows the examiner to view both long term trends and current instrumental performance.

4.4 Equipment/Instrumentation

- Perkin Elmer (PE) FTIR bench with ATR accessory and microscope accessory
- Thermo Nicolet FTIR microscope
- Liquid nitrogen, small thermos, funnel, PPE

- Microtome
- Diamond compression cell
- Potassium bromide plate barium fluoride windows, microscope slides and sample holders
- Polystyrene calibration standard
- Sample handling and preparation tools, such as scalpels, probes, roller and tweezers

4.5 Analytical method

The type, quantity and condition of sample will often determine which method should be used for analysis via FTIR.

Instrumental parameters must be set to provide sufficient intensity and resolution for spectra comparison or classification based on the operating requirements of each accessory. Both *the* background and sample must be run under the same instrument parameters. A new background should be run anytime there is a significant change to the analytical system or a significant time has passed since the last background spectrum was collected, or when water vapor and carbon dioxide peaks become significant.

4.5.1 Bench

This method requires that the sample be prepared as a KBr pellet, placed into a KBr plate or placed onto a sample holder such as a diamond compression cell. The sample must be prepared thin enough that the IR beam can penetrate it. This may require rolling or pressing the sample flat prior to testing.

4.5.2 ATR

This method requires minimal sample preparation. No pre-pressing of the sample is needed but the sample should cover as much of the sample aperture as possible. Only the surface of the sample is analyzed and, therefore, this technique will not analyze the entire thickness of the sample. Depending on the circumstances this could be an advantage or a disadvantage.

4.5.3 Microscope

The sample is pressed or rolled and then placed onto the surface of a KBr or other suitable window. It is possible to analyze the sample using either transmission or reflectance modes. The choice of method will determine what type of substrate is used.

4.5.4 Diamond Anvil Cell (DAC)

This method is particularly useful for plastics, rubbers, and foam type materials. It may also be useful, due to the inert nature of the DAC, for liquids including possible corrosive materials. There is minimal sample preparation for powders or pliable materials (foam and rubber material). These samples are placed into the cell and pressed between the anvil surfaces. Harder materials, such as plastics, should be cut thinly or scored prior to analysis. Samples should not be excessively compressed since it may cause crystalline samples to lose some crystallinity. This often results in broadening the absorption bands or coalescing.

4.6 Sample preparation for transmission/ATR/microscope:

A scalpel may be used to remove individual polymer films, such a paint layers in a multilayer system, or slice a thin polymer sample. If possible the outer surface of the sample should be removed in order to remove dirt before analysis. A microtome may also be used for sample preparation.

If necessary, the sample may be flattened on a glass slide or other clean hard surface with a roller. The roller should be cleaned between uses.

4.7 Spectral Evaluation and Comparison

Questioned spectra will be evaluated visually for suitability prior to being used for a comparison. A spectrum will be judged suitable when the peaks that will be used for comparison purposes are reliably discernable above the background.

There are a number of factors that should be considered when comparing sample spectra including the presence or absence of absorption bands, and their peak position (wavenumber), shape, width, relative intensity and the symmetry. Sample thickness may affect the peak width and resolution. Multiple sample replicates are generally necessary to evaluate reproducibility of these spectral characteristics.

Note: When comparing spectra, the presence of additional absorption bands in one of the spectra could be from true differences between the samples or from extraneous material in or on the sample (possibly from an adjacent layer). If extraneous material is suspected as the source of the difference, additional samples should be prepared. If a sample without the extraneous material cannot be prepared, then spectral subtraction may be an option.

4.8 Interpretation Criteria

The following possible findings ~~conclusions~~ can be reached after evaluating and comparing spectra:

1) Matching- The spectra being compared correspond in the position, shape and relative intensities or respective absorbance bands ~~and other criteria~~ and no significant, unexplainable differences are noted.

2) Inconclusive- the spectra being compared exhibit both similarities and differences and the significance of the differences cannot be completely assessed due to the constraints such as sample size and/or condition.

3) Different- The spectra being compared exhibit unexplainable differences with regard to the positive, shape, and/or relative intensities of corresponding absorbance bands. These differences are attributed to differences in chemical composition between samples.

4) Inconclusive- The spectra being compared exhibit both similarities and differences and the significance of the differences cannot be completely assessed due to the constraint~~ s~~uch as sample size and/or condition.

4.9 References

1. SWGMAT. “Guideline for Using Fourier Transform Infrared Spectroscopy in Forensic Tape Examinations.” Web. 03 Feb. 2012.
2. Bartick, E. G.; Tungol, M. W. In Forensic Science Handbook; Saferstein, R.,Ed.; Prentice Hall Vol. III, Englewood Cliffs, N.J., 1993, Chapter 4.
3. Bartick, E.G., and Tungol, M.W. “Infrared Microspectroscopy and Its Forensic Applications”, Saferstein, R., Ed, Forensic Science Handbook, Vol. III, Englewood Cliffs, N.J.: reagents/Prentice Hall, 1993, pp. 196-252.

4. Norman, E.W.W., Cameron, R., Cartwright, L.J., Cartwright, N.S., Clark, W.H., and MacDougall, D.A., "The Classification of Automotive Paint Primers Using Infrared Spectroscopy - A Collaborative Study", Canadian Society of Forensic Science Journal, Vol. 16, 1983, pp. 163-173.
5. Robertson, J., Ed. Forensic Examination of Fibers; Ellis Horwood: New York, 1992.
6. Suzuki, E.M., "Infrared Spectra of U.S. Automobile Original Topcoats (1974-1989): I. Differentiation and Identification Based on Acrylonitrile and Ferrocyanide C N Stretching Absorptions", Journal of Forensic Sciences, Vol. 41, No. 3, 1996, pp. 376-392.
7. Tillman, W.L., "Automotive Paint Systems Identification", Proceedings of the International Symposium on Forensic Aspects of Trace Evidence 1991 June; Washington DC: U.S. Government Printing Office: pp. 123-152.

5 Visible Microspectrophotometry (MSP)

5.1 Introduction

Visible microspectrophotometry is defined as the spectral analysis of the wavelengths of visible light which are absorbed by a sample. The spectral results are directly related to the color of the sample.

This is a non-destructive technique and requires very little sample preparation.

Common sample types include, but are not limited to:

- Paint
- Fiber
- Ink
- Other colored material

5.2 Equipment/Instrumentation

- Craic 508PV Microspectrophotometer
- Stereomicroscope
- Light sources:
    - Halogen – Transmission spectra in the visible to near IR range
    - Xenon – Transmission and fluorescence spectra in the UV, visible and near IR range
    - Mercury – Fluorescence spectra in the UV, visible and near IR range

5.3 Minimum Standard and Control

The following quality checks are run according to schedule prior to conducting analyses on case samples.

1. Complete the MSP Validation. The instrument data must be retained in accordance to the laboratory retention schedule. Ensure that the microscope is aligned in Koehler illumination. Adjust as needed.

2. At the time the unit is setup for testing, conduct a Performance Check/Validation of the unit using the NIST standard filters.

3. Perform the daily performance checks prior to collecting data. These are conducted using the same NIST traceable standards which were used to validate the instrument.

5.3.1 Day of Use

Neutral density filters are used to calibrate the photometric accuracy of a microspectrophotometer. The holmium filter spectrum has peaks in both the visible and the UV regions, and can calibrate the wavelength scale of the MSP from 418 nm to 637nm. The didymium filter spectrum has peaks in the visible and near IR wavelength regions and is used to check the precision of the instrument measurements regarding the wavelengths from 441nm to 879nm.

Wavelength and Photometric Checks (Transmission check only) are completed using the NIST traceable standards.

5.4 Sample Preparation Procedure

Samples that need to be sectioned, such as paint, should be prepared using a microtome to ensure that the recovered and standard samples are of the same thickness. Twists or other sample variations should be avoided. Samples can be rolled or pressed flat and then tested.

Mount the samples to be tested on a clean glass microscope slide and apply a cover slip. For samples that will be directly compared, ensure that the coverslips, mounting medium and other supplies used for preparation are from a single source on samples that will be compared to each other in order to avoid introducing variability to the samples.

Note: If data is to be collected solely in the UV range (~200nm), quartz slides and coverslips and glycerin mounting medium should be used.

5.5 Analysis Method

Documentation of specific instrumental operating parameters must be included in the case notes as appropriate.

Testing parameters may be changed from those listed, as needed:

- Spectral range of the instrument is 350-950 nm. Under the Tools Menu, select Set Parameters or choose the Gear icon, then select Mode to change the testing range based on the type of analysis being run. Available Modes include: Full 350-950 nm and Fluorescence (Fl+) 350-820 nm. Additional Modes can be created and saved as needed.
- Select an aperture size that is slightly smaller than the sample itself so the sample edges and background are avoided. Use the same aperture size for both the reference scans and the sample.
- Scans: 25: The instrument will automatically choose the best integration time based on the number of scans and the sample characteristics
- Resolution Factor: 5
- Mode Average/Mode Resolution = 25/2

Sample data for comparison should be collected on a single day and all oriented in the same position within the aperture. Natural fibers generally exhibit sufficient variability to require additional data collection. Pleochroic fibers should be oriented in the same direction and may introduce additional variation to the data.

5.6 Spectral Evaluation and Comparison

Questioned spectra will be evaluated visually for suitability prior to being used for a comparison. A spectrum will be judged suitable when the peaks that will be used for comparison purposes are reliably discernable above the background.

There are a number of factors that should be considered when comparing sample spectra including the presence or absence of absorption bands, and their peak position (wavenumber), shape, width, relative intensity and the symmetry. Sample thickness may affect the peak width and resolution. Since the spectrum is a result of light being transmitted through the sample, absorption will be noted in the area of the UV spectrum that corresponds to the visible color of the sample.

The examiner should collect several spectra from each sample and obtain a mean spectrum for comparison.

5.7 Interpretation Criteria

The following possible findings can be reached after evaluating and comparing spectra:

1) Matching- The spectra being compared correspond in the position, shape and relative intensities of respective absorbance bands and no significant, unexplainable differences are noted.

2) Inconclusive - A sample exhibits featureless spectra (e.g. undyed samples or dyes that have wide weak absorption bands and flat transmission maxima) or spectra being compared exhibit both similarities and differences and the significance of the differences cannot be completely assessed due to the constraints ~~su~~ch as sample size and/or condition.

3) Different- The spectra being compared exhibit unexplainable differences with regard to the position, shape, and/or relative intensities of corresponding absorbance bands. These differences are attributed to differences in absorbance between samples.

5.8 References

1. SEE Applications Reports, by Dr. Paul Martin:
    - Advances in Ultraviolet-Visible-Near Infrared Range Microspectroscopy, 2001UV
2. Microspectral Analysis of Nylon Fibers, 1999:
    - Effects of Diffuse and Specular Light on Reflectance Microspectroscopy, 1998
    - Effects of Diffuse and Specular Light on Reflectance MSP, 1998
    - Microspectral Characteristics of Coated and Uncoated Yarns, 1997
    - Microspectral Characteristics of Inks on Paper, 1997
    - UV-Vis Microspectral Analysis of Black Inks, 2000
    - UV-Vis Microspectral Analysis of Blue Inks, 2000
    - Microspectral Analysis of Microfibers, 1997
    - Microscopic Spectra of Color Paint Chips, 1997

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

6.1 Introduction

The scanning electron microscope (SEM) is one of the most versatile instruments available for the examination of the microstructural characteristics of solid objects. It has imaging capabilities which provide high resolution, large depth of field and a three-dimensional image at both very high and very low magnifications. Further, these image characteristics are acquired non-destructively and often with little sample preparation.

A bi-product of the electron beam/sample interaction provided by the SEM is the generation of several complimentary useful signals, including x-rays. X-rays exhibit energies that are specific to the elements from which they originate. By combining the SEM with an energy dispersive x-ray spectrometer (EDS), the generated x-rays can be detected and characterized. The SEM/EDS combination provides structural, qualitative and, in some cases, quantitative inorganic compositional information about the sample in question.

SEM/EDS forensic applications typically include particle analysis, unknown characterization, paint analysis, explosive analysis, tape analysis and screening for other analytical methods (i.e., bullet jackets, cartridge cases, bullet lead, solder and others).

6.2 Safety Considerations

Personal protective equipment should be worn to cover eyes and skin when working with liquid nitrogen.

6.3 Equipment/Instrumentation

- Scalpel
- Tweezers (variety as needed)
- Aluminum and carbon sample planchets
- Carbon rods
- Evaporation source
- Carbon tape
- Reagent grade isopropyl alcohol
- Micro-scissors
- Embedding material
- Embedding molds
- Beem capsules
- Multi-sample holder
- Silicon carbide grinding papers, including 60 grit, 240 grit, and 600 grit
- Diamond polishing compound, including 6 m and 1 m particle size
- Nylon polishing cloth
- Extender for diamond paste
- 0.05 m alumina
- Buehler “Microcloth”, or equivalent
- Polisher
- Ultrasonic cleaner
- Detergent
- Distilled water
- Soft cloths

- Hemostatic forceps (various sizes)
- Jeweler saw
- Wood Applicator sticks
- Micropipettes drawn from disposable Pasteur pipettes
- Fine tip marking pens
- Single edge razor blades
- Vise or clamps
- Mounting putty
- Scanning electron microscope equipped with an energy dispersive x-ray spectrometer
- Sputter coater
- Vacuum evaporator
- Stereo microscope
- Microtome
- Metallurgical polisher

6.4 Minimum Standards and Controls

The SEM/EDS will undergo the following quality checks:

Full instrument calibration check must be successfully completed and recorded within the 30 days preceding case analysis.

Quantization optimization must be performed at analysis parameters, prior to each case analysis. Completion of this action must be recorded in the exam documentation.

6.5 Sample Preparation Procedure

Evaluate the analytical goals~~,~~ considering: sample size, overall sample hardness, hardness of individual components of sample, area of interest, allowed destructiveness of the sample.

Determine whether the sample should be embedded, and if so, what embedding material is appropriate.

Determine whether sample requires reduced temperature for desired cutting characteristics, as may be necessary for soft plastic samples.

If specimen and embedding material trimming is required, clamp the sample in the specimen holder support for manual trimming. Manually trim the specimen, removing large amounts of material with a suitable trimming tool:

1. Clamp specimen holder in specimen arm of microtome.
2. Select appropriate knife angle, illumination, cutting window limits, cutting speed, and cutting thickness.
3. Advance the knife to the block face.
4. Select trimming portion of knife.
5. Align block to knife.
6. Rough cut sample to yield appropriate cross section.
7. Select final cutting portion of knife.
8. Remove final sections. If sections are desired, they are transferred to a glass slide or grid for final analysis. If faced block is required, block is removed from holder and processed for analysis.

9. If using a multi-sample holder, the height of each sample is adjusted in order that the planes of the surface and the holder intercept the sample at an appropriate height.

Sample embedment procedure:

1. Place sample in suitable mold or beam capsule.
2. Position to reveal structure(s) of interest when cross sectioned.
3. If necessary, small or buoyant samples may be attached with a thin adhesive layer.
4. Place a specimen label with the sample.
5. Prepare embedding medium according to specific medium instructions.
6. Under a stereo microscope, with a pointed end of a wood applicator stick, place a drop of embedding medium adjacent to the specimen.
7. Draw the medium to the edge of the specimen. (Capillary movement of the medium around the sample prevents air bubble formation.)
8. Fill the remainder of the mold with embedding medium.
9. Allow mold to cure. Small molds may be cured at 40°C for one hour, larger at room temperature. (Room temperature curing time varies according to mold size and environmental conditions.)
10. Remove embedment blocks from molds or Beem capsules.

- **Cross Sectioning**
- **Free hand cutting**-Clamp sample in vise. Under stereo microscope, trim excess embedding medium with jewelers saw. Using single edge razor blade, make thin final shavings.
- **Microtome** - May be used to expose the internal structure of inhomogeneous materials for analysis with the SEM analytical system. The process may be used to produce ultrathin, thin, or thick sections, or may be used to produce flat bulk samples. Many samples will require embedment prior to microtomy (see “Embedment” above).
- **Ultramicrotome-** May be used with a glass knife for cutting ultrathin or thin sections of small samples of medium hardness, or facing unembedded materials of medium hardness. An ultramicrotome may be used with a diamond knife for cutting ultrathin and thin sections of small samples of extreme hardness, or facing extremely hard unembedded materials.
- **Histomicrotome with a tungsten carbide knife** -May be used for cutting thick sections of medium hard materials, or facing blocks of medium hard materials.
- **Polishing** - Polishing may be used to expose the internal structure of inhomogeneous materials in order to analyze each component with the SEM analytical system. Additionally, polishing is frequently necessary for the preparation of a flat, scratch free surface for quantitative analysis.
- **Grinding** –Water is applied to a sheet of 60 grit sand paper backed by glass. The multi-sample holder is placed with blocks protruding from the face, should be lightly sanded in a "figure 8" pattern. Holder is cleaned by brief ultrasonic agitation in water with a few drops of detergent, rinsed with water, and dried.
    - **60 grit silicon carbide paper**- Sanding is continued until scratches from the 60 grit paper appear uniformly on the face of the holder. Scratches should be made parallel, and continuous across the face of the holder and individual blocks. Tighten each block securely. If height adjustment was made, repeat the above polishing step.
    - **240 grit silicon carbide paper** - Sanding is continued with 240 grit sandpaper, using the sanding technique described above. Final sanding marks are made at a 900 orientation to the last sanding step. Sanding at this stage is continued only to the point of removal of the scratches left by the last step.

- **600 grit silicon carbide paper-** Sanding is continued with 600 grit sandpaper, using the sanding technique described above. Final sanding marks are made at a 900 orientation to the last sanding step. Sanding at this stage is continued only to the point of removal of the scratches left by the last step. Observe samples with LM at low magnification.

- **Nylon polishing cloth**-Clamp to the wheel of the polisher/grinder, and charged with either a 6 m or 1m diamond paste. A small amount of extender (diluent) is sprayed on to the wheel. The holder is pressed to the wheel with moderate pressure. The holder is moved in a clockwise direction, rotating the holder occasionally. Polishing is continued until the 600 grit sandpaper scratches are removed. Holder is cleaned by brief ultrasonic agitation in water with a few drops of detergent, rinsed with water, dried, and observed by LM. The selection of a final polishing step will depend on the analysis required; therefore steps 6 and 7 may not be required.
- **Micro cloth Polish**- Attach cloth to the wheel of a polisher/grinder and charged with 0.05m alumina. A small amount of water is sprayed on to the wheel. The holder is pressed to the wheel with moderate pressure. The holder is moved in a clockwise direction, rotating the holder occasionally. Polishing is continued until the 1 m diamond abrasive scratches are removed. Holder is cleaned by brief ultrasonic agitation in water with a few drops of detergent, rinsed with water, dried, and observed by LM.
- **Coating** - Some non-conductive samples require treatment to enhance surface conductivity, in order that x-ray analysis may be performed optimally. Insufficient conductivity may result in poor imaging of the sample and beam deflection from the intended analysis area.

6.6 Analysis Method

The following suggested instrument operating conditions are meant as general guidelines or starting conditions. Actual requirements may vary as the analyst determines specific analytical needs.

- A beam voltage of 20 - 25 KeV.
- A display range of 0 - 20 KeV.
- Pulse processor time constant at a mid-range value.
- Beam current adjusted to yield an x-ray detector dead time of at least 30% (newer detector models may be able to handle higher dead times).
- Counting time between 100 and 200 seconds for minor peak discrimination.
- Counting time between 10 and 20 seconds for major elements present.
- Beam/sample/x-ray detector geometry should be optimized for x-ray collection efficiency.

Generally, changes in the suggested starting operating conditions are required under the following circumstances:

- Beam voltage is increased when higher energy line excitation is required.
- Beam voltage is decreased when greater spatial resolution is required.
- Pulse processor time constant is lengthened when greater spectral resolution is required.
- Pulse processor time constant is shortened when a greater count rate is required, (for trace element analysis or construction of elemental distribution maps).
- Detector to sample distance can be reduced or increased to increase or decrease x-ray collection efficiency.
- Spectral energy display scale is expanded when sufficient detail is not evident.
- Beam current is increased when the X-ray count rate is too low. Decreasing the condenser lens current and/or increasing the final aperture size may increase beam current.
- Beam current is decreased when the X-ray count rate is too high. Increasing the condenser lens current and/or decreasing the final aperture size may decrease beam current.

6.6.1 Imaging analysis

1. Utilizing the secondary electron (SE) signal detector, optimize instrument operating conditions as dictated by the sample to be examined.
2. Beginning at low magnification, focus and proceed to higher magnifications, as needed.
3. A backscattered electron image is useful for defining structures based on the average atomic number of the matrix. Structures containing elements with higher atomic numbers will generally appear brighter than those with lower atomic numbers. This is often useful for evaluating homogeneity and layer structure.
4. Photographically document or print visual image(s). SEM micrographs should include a measuring scale or magnification scale or both. The micrograph should also display which signal (backscattered electron or secondary electron) was used to produce the image.

6.6.2 Bulk analysis

- Observe a backscattered electron image of the sample to evaluate the homogeneity of the sample.
- In order to compare the average composition of structures, the spectrum used for comparison should come from an area of the structure sufficient to produce representative composition.
- The representative nature of a spectrum can be determined by the critical comparison of spectra from adjacent areas. If no differences are evident, the sampled area is homogeneous at that magnification.
- A representative bulk analysis can be achieved by rastering the beam across as large an area as possible. Analyzing a single large area or summing the spectra from several smaller areas may achieve this.
- When comparing samples, all data and micrographs should be collected in the same manner with the same conditions.

6.6.3 Individual component analysis

- Additional evaluation of composition may be achieved by the spot (nonrastered) analysis of specific particles within layers. Generally, these particles appear bright in the backscattered electron image. Such an analysis may improve the detection limit beyond that achievable by a bulk analysis, as well as serve to associate elements detected by a bulk analysis. For example, the bulk analysis of a tape adhesive may reveal the presence of Al, Si, Mg, and O. Specific particle analysis may associate the elements Si, Mg, and O as being present in one type of particle, and Al, Si, and O in a second type. These associated elemental compositions would then indicate these particles could be talc and kaolinite, respectively.
- Because the beam interaction volume may be considerably larger than an individual particle, inclusion of other matrix components may be expected in the spectrum from an individual particle. Lower beam voltages may be used to confine more of the interaction volume to the particle. It should be noted, however, that the use of lower beam voltages may result in the loss of characteristic lines that may be found at higher energies.

6.6.4 Analysis of a primarily organic matrix

- Analysis of a substance that is primarily organic (e.g., duct tape backing, clear electrical tape adhesive) may be useful. Within such a matrix, the interaction volume is significantly larger than that of a substance that is primarily inorganic. This is a result of a lower average atomic number of the matrix. In order to reduce the interaction volume, the beam voltage may be reduced; however, the voltage should be sufficient to produce X-rays from all lines of analytical interest. Charging may also be an issue

with such samples. Therefore, precautions may be taken to prevent this from occurring (e.g., sample coating or operation at low vacuum).

- Because an organic matrix may contain small amounts of some elements, the counting time should be extended.

6.6.5 Qualitative analysis

- Once an X-ray spectrum is collected, a qualitative analysis is performed in order to determine the elements present. The process is straightforward for the peaks of elements present in major amounts and those not overlapping. Misidentifications or omissions of minor components are possible unless a systematic approach to elemental identification is used which includes consideration of X-ray line families, spectral artifacts, escape peaks, sum peaks, and overlaps.
- Reference lines, or energies, may be obtained from several sources; including energy slide rules, published tables, and computer-generated KLM reference lines that may be superimposed on the spectrum. Additionally, manufacturers often provide an automatic element identification application. These aids often are used in complementary fashion.
- Identification begins with high-energy peaks and major peaks. High-energy peaks are generally less likely to overlap than lower energy peaks. If a major peak is present, generally a complete family of peaks can also be identified. Each line within the family is labeled with elemental symbols. Spectral artifacts, including sum peaks and escape peaks associated with major peaks, should be evaluated and labeled.
- As spectral interpretation alternates between the identification of major and minor peaks, the vertical (counts) scale should be adjusted to reveal required detail. In addition to the higher energy peaks, the presence of any lower energy families and their expected relative intensities should be noted. Individual asymmetric peaks and inconsistent peak ratios within a family may indicate a peak overlap. Superimposing and scaling KLM reference lines on the spectrum or referencing the actual spectrum of an elemental standard aids elemental identification. The analyst should be familiar with the characteristic pattern and relative intensities of peaks of various atomic numbers. The identification of major elements is usually straightforward.
- Following the identification of major elements, lower intensity peaks and overlapped peaks are identified. The limited number of characteristic peaks present for minor elements can limit their identification.
- The presence of an element can be considered unequivocal only when a distinctive, unique set of lines is produced or when a single peak occurs at an energy where it cannot be mistaken for another element or spectral artifact. Unequivocal identification may not be possible if an element is present in low concentration or if lines required for confirmation are overlapped with the lines of other elements.
- Spectra should be displayed on a scale that clearly demonstrates the peaks identified. In order to display peaks from elements with significant differences in concentration, the peaks from the elements in low concentration may be viewed by displaying the spectra separately on different display scales.
- If an automatic identification application is used, the analyst should confirm the resulting element identifications.
- There may be an overlap of peaks in the energy dispersive X-ray spectroscopy spectrum of materials containing several elements. Some commonly occurring overlaps encountered in energy dispersive X-ray spectroscopy are as follows: Ti K-β/V K-α, V K-β/Cr K-α, Cr K-β/Mn K-α, Mn K-β/Fe K-α, Fe K-β/Co K-α, Pb M-α/S K- α/Mo L-α, Ba L-α/Ti K-α, K K-β/Ca K-α, Zn L-α/Na K-α, P K-α/Zr L-α, and Al K-α/Br L-α.
- In order to resolve these overlaps, several methods may be employed.
    - The live time count can be increased.

- The processing time of the pulse processor may be increased to improve spectral resolution.
- Mathematical spectral subtraction (deconvolution) methods supplied by the energy dispersive X-ray spectrometer manufacturer can be employed.
- An alternative method of elemental analysis or X-ray diffraction may be used.

6.6.6 Quantitative analysis

Determines how much of a particular element is present in the analyzed volume of a sample.

1. Sample must be homogeneous, flat, polished and larger than the beam/sample interaction volume.
2. Obtain and record spectra.
3. Compare recorded spectra with those of standards of known composition.
4. Correct for background and instrumental effects.
5. Apply matrix corrections and calculate composition of analyzed volume.

6.7 Spectral Evaluation and Comparison

Questioned spectra will be evaluated visually for suitability prior to being used for a comparison. A spectrum will be judged suitable when the peaks that will be used for comparison purposes are reliably discernable above the background.

Comparisons are facilitated by direct spectral comparison. Spectral details are generally evaluated in terms of background shape and peak composition and ratios.

- Differences in background shape may result from dissimilar sample geometry.
- Differences in the composition of major peaks may indicate that the spectra are not representative of the bulk composition of a heterogeneous sample.
- If there are no differences in major peak ratios, differences in minor/trace components may result from the presence of extraneous materials. If the sample was a fragment or unable to be cleaned, a small amount of foreign material may have been present during the analysis. Consequently, some of the minor elemental peaks in the spectrum may have been produced from elements in the extraneous material.
  Differences in carbon intensity may result from a contribution of carbon from the mount if the sample is very small. Furthermore, the presence of carbon, oxygen, and nitrogen in the tape matrix limits the usefulness of these elements in direct spectral comparison; therefore, they are typically not evaluated.

6.8 Interpretation Criteria

The following possible findings can be reached after evaluating and comparing spectra:

1) Matching- The spectra being compared correspond in the position, shape, and relative intensities of respective elemental peaks and no significant unexplainable differences are noted.

2) Inconclusive - spectra being compared exhibit both similarities and differences and the significance of the differences cannot be completely assessed due to the constraints such as sample size, condition or other factors.

3) Different-The spectra being compared exhibit unexplainable differences with regard to the position, shape, and/or relative intensities of corresponding elemental peaks. These differences are attributed to differences in elemental composition between the samples.

6.9 References

1. SWGMAT. “Guideline for Using Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy in Forensic Tape Examinations.” Web. 03 Feb. 2012.

<http://swgmat.org/Tape%20SEM%20guideline.pdf>

2. Friel, J. J., "X-Ray and Image Analysis in Electron Microscopy", Princeton Gamma Tech, Princeton, N. J., 1995.
3. Goldstein, J. I., et al., "Scanning Electron Microscopy and X-Ray Microanalysis", second edition, Plenum Press, New York, New York, 1992.
4. Lifshin, E., "Advances in X-Ray Analysis", Vol. 19, Kendall/Hunt, Dubuque, Iowa, 1975.
5. Malis TF, Steele D, Ultramicrotomy for Materials Science, published in "Workshop on Specimen Preparation for TEM of Materials II", ed. R. Anderson, Vol 199, Materials Research Symposium Proceedings (Materials Research Society, Pittsburgh, 1990).
6. McCrone, W. A. and Delly, J., The Particle Atlas, Vol. I, Ann Arbor Science Publishers, Ann Arbor, Michigan, 1973.

7 Physical Break Match/Tear Configuration Comparisons

7.1 Introduction

Physical breaks or tears to objects can result in damaged edges which may bear sufficient random characteristics to determine that the pieces were at one time a single item.

An examination for a physical break (fracture) matches or tear configurations should precede any potentially destructive chemical or physical analysis. However, the examiner should realize that further analysis via chemical or instrumental methods may be required if no clearly defined break/tear configuration is identified.

Samples commonly tested include any items which can be broken or torn.

7.2 Equipment/Instrumentation

- Stereomicroscope
- Hand magnifier or loop
- Transmitted light microscope
- Comparison microscope
- Light box

7.3 Minimum Standards and Controls

All physical break matches between a question specimen and a known specimen must be verified by another qualified analyst.

7.4 Procedure

Questioned evidence will be evaluated visually for suitable detail prior to being used for a comparison. Due to the nature of the examinations, the known and questioned items will come into contact with each other. Therefore, all items should be marked or otherwise documented in such a way as to clearly distinguish them during the examination.

1. Examine the broken or torn edges of each item including surface markings that may cross the break /tear.
2. Compare the geometric alignment of the fractured edges of the questioned items to the corresponding edges of the known items. Damaged fibers within a fabric sample, such as a torn garment, may exhibit a distinctive end characteristic (cut, torn or stretched ends) for identification. Examination of these samples should be done via stereomicroscope or greater magnification if necessary.

7.5 Interpretation Criteria

The following- possible-findings can be reached after evaluating and comparing physical break match/tear characteristics:

Source Identification- A significant quantity and/or quality of edge characteristics fit together between pieces revealing a matching tear/break configuration confirming that at one time they were a single piece. Other characteristics including striations, color, texture and /or shape may also be considered

Inconclusive- The of the tear/ break configuration(s) is not distinct enough to say with certainty that there was a matching or non-matching break/tear configuration but the general configuration between the pieces is consistent.

Non-matching tear / break configuration – A significant quantity and/or quality of edge characteristics that do not fit together between pieces. Other characteristics including striations, color, texture and/or shape may also be considered.

7.6 References

1. Hearle, J. W. S., Lomas, B. and Cook, W. D. Atlas of Fibre Fracture and Damage to Textiles, The Textile Institute, CRC Press 1998.

8 Impression Comparisons

Impression comparisons involve comparing class and individual characteristics of questioned impressions to known footwear to determine if they correspond. Class characteristic are characteristics shared by more than one item. Individual characteristics are most often caused accidentally or randomly and. The size, shape, and relative position of individual characteristics are important. Individual / accidental characteristics may also be referred to as "randomly acquired characteristics" or RAC for short. Accidental characteristic, individual characteristic, and RAC should be considered interchangeable.

Common samples include, but are not limited to:

- Digital images of impression evidence
- Photographs of scene impressions
- Lifts of recovered scene impressions
- Items submitted with visible impressions
- Known footwear
- Known tire tread exemplars or photographs
- Fabric

8.1 Equipment/ Instrumentation

- Ruler
- Caliper
- Magnifying glass or latent print loop
- Supplies for enhancement technique(s)
- Supplies for preparing test impressions
- Stereomicroscope
- Footwear database
- Flatbed scanner
- Digital imaging software (e.g. Photoshop)
- Digital camera

8.2 Minimum Standards and Controls

Known test impressions should be prepared for comparison in cases when the questioned impression cannot readily be eliminated through visual comparison with the known footwear. Test impressions for comparisons should be produced in a manner similar to the question impression(s), if possible. General test impressions that simply document the tread design of the outsole are sufficient for elimination.

All impression evidence source identification, support for same source, and source exclusion conclusions must be verified by another qualified analyst.

Addition of evidence footwear into the reference database should be documented in the case notes, but does not need to be referenced in the laboratory report.

If the impression is entered into the database, a report of the questioned impression record and known record(s), if applicable, should be printed and attached to the Laboratory report.

If the initial search of an impression fails to reveal a matching tread design in the database, additional searches may be conducted. All subsequent searches of an impression will be documented in a log. Additional

searches resulting in a tread match will be reported to the customer. Additional searches resulting in no make/model candidate determination must be documented, but do not require supplemental laboratory report issue.

With regards to examination quality digital images, the settings for converting RAW images to TIFF images and the enhancement of TIFF images will be tracked in the images' metadata unless otherwise specifically noted in case documentation.

8.3 Analysis Method

It may be prudent to avoid removal of debris from the tread of known footwear that could be contributing to an individual characteristic that may be present in the questioned impression.

Questioned impressions are first evaluated for suitability based on the amount and quality of detail present. If it is determined that the impression is of such poor quality that it could not be used for comparisons then no further examination needs to be completed. The impression is reported as unsuitable for comparison purposes.

Questioned impressions are then compared to the known shoes and any photographs, test impressions or transparencies of the known shoes. For footwear impressions an evaluation of the correspondence of class characteristics, wear, and identifying characteristics is carried out. To the degree that the known shoes or test impressions created from the known shoes and questioned impressions correspond; those two items can be associated as having a common source.

8.3.1 Enhancement Procedures

Enhancement methods (physical, chemical and / or photographic) may be employed to increase color and contrast in order to improve detail visibility.

The method(s) chosen for enhancement will depend upon the medium that the impression is registered in and the substrate the impression has been deposited onto. All impressions should be photographed in their original condition before attempting any type of enhancement. The following is only a partial list of enhancement techniques that are available for use. The book Footwear Impression Evidence by William Bodziak describes other procedures.

When applicable an area of the substrate void of impressions should be tested first with the chosen enhancement method to ensure there are not adverse effects.

8.3.2 Physical

Regular or magnetic fingerprint powders may work well for impressions that were deposited wet on clean non-porous surfaces or impressions composed of grease or other residue.

The powdered impression can then be photographed and lifted with gelatin lifting materials or wide clear adhesive tape.

8.3.3 Chemical

Chemical enhancement techniques react with trace components in the impression to increase the contrast and visibility between the impression and the substrate. Chemical enhancement reagents need to be tested prior to use. This test should be conducted with an appropriate material based on the composition of the questioned impressions.

8.3.4 Potassium and Ammonium Thiocyanates

Potassium and ammonium thiocyanate react with iron in the residue of impressions to create a reddish-brown color. It is best suited for use on wet residue or soil impressions on all surfaces. The reagent is sprayed over the impression using the finest mist possible to avoid over spraying. The amount of spraying should be controlled to get maximum reaction without causing the impression to run or bleed. The impression should be photographed shortly after spraying is completed.

8.3.5 Leucocrystal Violet (LCV)

Leucocrystal violet is used to enhance and develop impressions deposited in blood by turning the impression a dark violet color.

8.4 Lifts

Some lifting techniques produce a mirrored image of the impression relative to the orientation in which the actual impression was registered.

8.4.1 Gelatin Lifters

Gelatin lifters consist of a gelatinous layer and a clear protective covering. Commercially available gelatin lifters can be used on both porous and nonporous surfaces to lift both original impressions and impressions that have been dusted with powder. Gelatin lifters are available transparent, white or black. In order to easily realign the cover with the lifter after use, it is helpful to cut a small crooked piece off of one corner before removing the cover in preparation for use. Lifters should be allowed to rest for a few moments after removal of their protective covering and before application in order to “relax” back into their normal shape. The lifter should be applied carefully, avoiding air bubbles and without stretching or distortion caused by applying excess pressure. After application to the impression, firm but gentle pressure should be applied to the lifter with a roller to ensure good contact. If the impression was wet in origin the lifter should remain on the impression for ~10 minutes to improve transfer of the impression.

8.4.2 Electrostatic Lifts

Electrostatic lifters can be purchased and are an excellent method for lifting fine particulate impressions such as dust. These impressions are very fragile and the lifter should be photographed as soon as possible. Side lighting may be very useful in photographing such lifts. These lifts should be photographed and the digital image treated as evidence in cases where the impression is not durable.

8.5 Test Impression Procedures

8.5.1 Fingerprint ink and paper:

1. Spread a small amount of fingerprint ink over a piece of glass with an ink roller.
2. Press the object against the inked glass.
3. Press the object against white bond paper supported by sheets of newspaper or butcher paper.

Note: Fingerprint ink may cause very minute characteristics to become filled in and not be observed clearly.

8.5.2 Vaseline and magnetic fingerprint powder:

1. A small amount of Vaseline is rubbed into the gloved palm of the hand and then against the object.
2. A test impression is obtained by pressing the object against a receiving surface.
3. The receiving medium is then dusted with a Magna brush developing a clear image.

8.5.3 Lightning lifts with fingerprint powder:

1. Dust the outsole with the desired color of fingerprint powder. Tap off any excess powder. Apply the Lightning lifter and smooth flat over the surface of the shoe tread with a clean cloth or paper towel.
2. Remove the lifter from the outsole and apply it to the transparent acetate provided using a fingerprint roller. Turn the lifter over so as to correct the orientation of the lift and mark it with the appropriate case information.

8.5.4 Identicator kit

This is a commercial product and produces a high contrast black image on white, chemically treated paper. Simply press and roll the shoe tread onto the ink pad and then apply the outsole to the treated paper surface. Very little residue is left on the item.

8.5.5 Footwear Database

8.5.5.1 SoleMate™ Database

The SoleMate® database is purchased from the manufacturer but the system also includes an option for entry of case-related footwear and other known shoes into a second BCI-created outsole database. The purchased records may include the manufacturer, model name, the market release date, images and/or test impressions of the tread, images of the footwear uppers and a set of pattern feature codes which correspond to the tread elements on that specific shoe tread.

8.5.5.2 Database entries

If the impression is suitable both for comparison purposes and entry into the database, and have not been associated with any known footwear, they will be entered into the database and searched unless investigative circumstances indicate that the search is not necessary.

Questioned impression(s) from non-violent crimes, should be entered into the OHBCI CRIME database and searched against the footwear reference databases for possible make/model determination. If a make/model candidate is not identified in the original database search, the impression will not be searched again unless

specifically requested by the submitting agency. Records may be deleted from the OHBCI CRIME database as necessary.

Questioned impression(s) from violent crimes, should be entered into the OHBCI VIOLENT CRIME database and searched against the footwear reference databases for possible make/model determination. If a make/model candidate is not identified in the original database search, the search may be repeated following each of the next three SoleMate® database updates, or until a make/model candidate(s) is identified, whichever occurs first.

In order to potentially link violent crimes, questioned to questioned and known to questioned impression searches may be performed against or within the OHBCI VIOLENT CRIME database. These searches may be performed under the following conditions:

- The search request must originate from the customer or a BCI Crime Scene Agent.
- The request must specify, if possible, the extent of the database subject to search (e.g. specific case(s) retained in the database, geographical area for which cases may logically be related, similar offense types, offense time frame, etc.). This request information must be recorded in the case record.
- Search candidates identified for additional comparative examination should be from the customer specified search criteria.
- Results of the search will be documented in the case record and reported to the requesting customer.
- If search results identify possible candidate impressions originating from other customers (i.e. not the requesting customer) those customers will also be notified via report. Notification details will be recorded in the case record.
- No conclusive impression comparison results may be issued without direct examination of the evidence in question.

8.5.5.3 Adding known shoes to the database

Known shoes may be searched against the footwear reference databases for determining whether they are currently in the database inventory. If the known shoes are already represented in the databases, no further action is required. If the known shoes are not found in the databases, then a record should be created in the OHBCI REFERENCE database.

Note: The Reference Library consists of both the BCI-created Local Reference Database and the purchased SoleMate®® Database. The Reference Library should be chosen when searching for a matching shoe tread so that both the SoleMate®® and BCI local reference database will be searched simultaneously.

8.6 Interpretation Criteria

The following possible conclusions can be reached after evaluating and comparing impressions:

Source Identification
The questioned impression was found to be the same as the known with respect to tread size, tread design, wear characteristics (as applicable), and a sufficient quantity / quality of randomly acquired characteristics (RAC).

Support for Same Source
The questioned impression was found to be the same as the known with respect to tread size, tread design, and/or wear characteristics with no significant unexplained differences; without a sufficient quantity / quality of randomly acquired characteristics (RAC) for a source identification conclusion.

Inconclusive
The questioned impression was found to exhibit both differences and similarities to the known with respect to tread size, tread design, and/or wear characteristics; to the extent that no conclusion could be reached regarding an association or elimination. The significance of the differences cannot be completely assessed due to the constraints of sample size and/or condition.

Source Exclusion
The questioned impression was found to be different from the known with respect to tread size, tread design, and/or wear characteristics. The questioned impression could not have been made by this known.

8.7 References

1. Bodziak, William J., Footwear Impression Evidence, 2nd Edition, Elsevier: New York,
2. Brundage, David, Footwear Identification Workshop, MI, 1998.
3. Cassidy, Michael J., Footwear Identification, Public Relations Branch of the Royal Canadian Mounted Police, Ottawa, Ontario, Canada, 1990.
4. McDonald P., Tire Imprint Evidence, CRC Press, Inc., 1993.
5. Montgomery County Coroner's Office. Miami Valley Regional Crime Laboratory. Trace Evidence Impression Evidence. "Use of the SICAR System." March 01, 2012
6. Foster + Freeman Ltd. SICAR 6 User Manual. 2011. Print

9 Fiber and Fabric Analysis

9.1 Introduction

Fibers and the fabrics from which they originated may comprise some of the most important pieces of evidence found during the crime scene investigation. Large numbers of fibers can be shed and transferred in accordance with Locard's exchange principle.

Fibers can be divided into two categories, natural and synthetic. Forensic fabric and fiber analyses require an understanding of these fiber types and their manufacturing processes as well as modes of fiber transfer, and fiber collection, identification and comparison techniques. An understanding of the textile industry in regard to fabric types and their manufacturing processes is also needed.

Often, white (undyed) and "indigo" blue cotton fibers are encountered in evidence, however, they generally have no evidential value due to their prevalence. Knowledge of the specific case scenario will assist the examiner in knowing when these fibers are of forensic importance.

Destructive testing should only be conducted if the analyst deems them necessary and only after other non-destructive testing is completed.

Comparison of fibers is a painstaking process. The examiner can approach the fiber comparison by setting out to show that the samples are not similar. The failure to detect any significant differences throughout the examination results in the conclusion that the fibers could have the same origin. All noted features / characteristics must be consistent in order for a support for same source conclusion. Both the synthetic and natural fiber comparison forms list appropriate characteristics for examination of the different classes of fibers. Use of the appropriate form may help ensure that a complete examination has been conducted for each sample and the analyst's findings have been documented.

9.2 Equipment and Instrumentation

- Fine-tip tweezers, scalpels, scissors
- Clean paper
- Microscope slides, coverslips
- Mounting medium with a known refractive index (about 1.49 - 1.54 is recommended)
- Embroidery thread, sample threader, new razor blades, cross-section plates
- Xylene substitute (or similar appropriate solvent)
- Refractive index liquids
- Microchemical solubility reagents, spot plates
- Ruler, calipers
- Microscopes: Stereo, compound, PLM, comparison PLM
- FTIR
- MSP

9.3 Minimum standards and controls

A lab reference collection of synthetic and natural fibers, including human and animal hairs, fabric samples, ropes and cordage, and hairs.

All solvents used in solubility testing shall be checked on fiber standards from the laboratory reference collection before being applied to case samples.

Recovered fibers examined by FTIR shall be compared to either the fiber standards from that case, a standard from the reference collection or an FTIR library spectrum.

9.4 Analysis Methods

9.4.1 Microscopic Comparisons

The comparison of fabrics and fibers requires a combination of indirect and side-by-side comparison processes. The examiner should prepare the questioned and known samples in the same manner. Comparison should be conducted using the same methodology for both sample sources to determine if there are any unexplained (significant) differences.

Natural and synthetic fiber comparisons call for the examination of different characteristics due to the wide variety of generic classes and subclasses in each fiber group.

Synthetic fibers are examined for physical characteristics including color, tint, diameter, cross-sectional shape; manufacturing characteristics such as delustering and inclusions; and microscopic characteristics including relative refractive index, birefringence, sign of elongation and fluorescence.

Natural fibers are examined in both technical and ultimate forms. The technical fibers are examined for physical characteristics including color, texture, stiffness, cross-section and presence of crystals. A test for the degree of lignification is also conducted using phloroglucinol reagent. The fiber ultimates are released via a digestive procedure and are then examined for physical characteristics including average length, presence of dislocations and nodes, lumen shape and cell diameter. The Herzog test is conducted using a PLM.

Using a stereo microscope, examine the questioned sample. Note the presence of fine fibers, trilobal or other coarse fibers. For fabric, examine all fabric yarns (both directions). Fibers submitted on tape lifts can be removed using hexanes and tweezers under the stereo scope and mounted. Document the color, tint, or other physical characteristics of interest. Examine the fiber standard in the same way.

As appropriate, prepare a slide of each sample to include a representative sample of the fiber population. Examine the mounted questioned fibers with a polarized light microscope. Observe and note appropriate microscopic characteristics of each fiber. Compare the properties of both samples.

If the known and questioned specimens are similar, continue with the characterization of the samples using a compound comparison microscope. The comparison microscope should be utilized to confirm that two fibers are consistent or the results are to be reported as preliminary.

Confirm the color balance of the comparison microscope using two microscope slides each bearing samples of fibers which are known to originate from the same source. Rotate the stage under crossed-polars. Confirm that the fibers on both stages exhibit the same color and microscopic characteristics.

Continue with comparison of case samples by placing the questioned sample slide on one stage and the known sample slide on the other stage. Observe the color and microscopic properties of the fibers. Apply crossed polars and applicable compensators, each time with stage rotation. Compare the characteristics of the fibers at each step and document the findings.

Comparison of natural fibers is best approached by the process of elimination. There are many resources that provide guidance regarding what specific microscopic characteristics permit the association or elimination of various natural fibers from each other.

9.4.2 Determination of refractive index relative to mounting medium

1. Orient the polarizer of a polarizing microscope in the East-West direction.
2. Mount the fiber in a mounting medium of known refractive index.
3. Orient the fiber in the East-West direction to determine the relative refractive index of the fiber in the parallel direction (n parallel).
4. Move the objective away from the fiber (or lower the stage) and observe the Becke line (the halo of light surrounding the fiber).
5. The Becke line appears to move into the material of the higher refractive index.
6. If the Becke line moves into the fiber, then the refractive index of the fiber is greater than that of the mounting medium.
7. If the Becke line moves into the mounting medium, then the refractive index of the fiber is lower than the mounting medium.
8. Turn the fiber to a North-South orientation and repeat the process to determine the relative refractive index of the fiber in the perpendicular direction (n perpendicular).

9.4.3 Estimating Birefringence with a Berek Compensator

A Berek compensator is a tool that is used to estimate the retardation of a fiber. This retardation value, along with the diameter, can then be used to calculate the fiber's birefringence. The value obtained for birefringence is also an estimate but is accurate enough to allow classification of a fiber having a positive sign of elongation.

Each compensator is calibrated at four different wavelengths. These calibration charts are specific to that particular compensator. For measurements made without the use an interference filter, the E-line chart data should be used for calculation of retardation values.

Procedure:

1. Align the fiber of interest in a northeast to southwest orientation and through the center cross hairs if possible. Measure the fiber diameter (the same as thickness for a round fiber) and record the result. Do not reposition the fiber further.
2. Set the microscope at crossed polars. Insert the Berek compensator into the light path and set it at 30.
3. Confirm that a black "X" shape is visible, though out of focus, in the background.
4. While looking through the oculars, turn the spindle in one direction until the center of the fiber appears black where it intersects with the center of the cross hairs. Make a note of the value on the compensator to two decimal places.
5. Now turn the spindle in the opposite direction, past the starting point of 30, until the center of the fiber again appears black where it intersects with the center of the cross hairs. Make a note of this value to two decimal places.
6. Subtract the smaller value from the larger one and divide this difference by two. This value is the tilting angle. On the calibration chart for the e-line (wavelength of 546.1 nm), locate the integer value of the tilting angle along the left-hand column and the fraction across the top. The retardation is the value where these two columns intersect. Record this value from the chart as 'r'.
7. Calculate the birefringence: (B) = $\frac{\text{retardation (r)}}{}$

diameter x 10^3

8. Refer to the Michele-Levy chart or other data to determine the composition of the fiber based on the birefringence.

9.4.4 Preparing Cross-Sections

A fiber cross-section provides diagnostic information for identifying natural fibers and discriminating and diagnostic information for synthetic fibers. Cross-sectioning can be carried out by optical and physical techniques. Two of many acceptable methods for producing physical cross sections of fibers (or hairs) are detailed here.

It is recommended that each analyst practice various cross –sectioning methods in order to determine which technique works best for their skill set and samples before applying any such technique to recovered case samples.

The following method of cross sectioning was developed within the BCI laboratory.
Equipment:

- Plastic micro-pipets with extended fine tip (such as Samco Scientific 231)
- 28-gauge wire, brass if available
- Microscope slides
- Tweezers
- New scalpel
- Hot plate
- Tongs

Procedure:

1. Cut both sections A and B off of the transfer pipet (see photo) to create two pieces where the larger diameter piece “B” will hold the smaller diameter piece “A”. (Image 1)
2. Take a piece of wire and fold one end back along its length. Push the folded wire through the small diameter piece of section A. (Image 2)
3. Thread the desired number of fiber through the eye of the brass wire with it protruding through and out from section A. When sufficient fibers have been added to the wire loop, slowly pull the wire and fibers back into the center of section A. Do not pull the wire all the way through and out the other end of section A.
4. Cut section A just above brass wire to create a tube containing your fiber samples. (Image 3)
5. Insert section A with fibers inside the center of section B. Place the combined piece between two glass slides.
6. Place the stacked glass slides onto a hot plate set at low to moderate heat, example a setting of 3.5 out of 10 was used successfully. Apply moderate pressure to the top slide, pressing the tube sections as they melt. Continue pressing down with the tongs as the temperature of the glass slides increases. Use caution: the glass will become too hot to touch by hand.
7. Once the plastic pieces holding the fiber samples have been sufficiently melted, remove the glass slides from hot plate with the tongs. Allow to cool. (Image 4)
8. Once plastic has cooled, twist the slides apart. Use a new, sharp razor blade or scalpel to slice thin cross-sections of the melted plastic sample.
9. Create a permanent mount of the cross-sections and view with PLM.

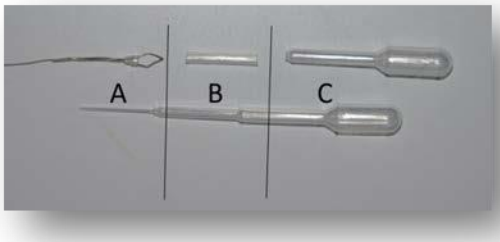


Image 1

Image 2

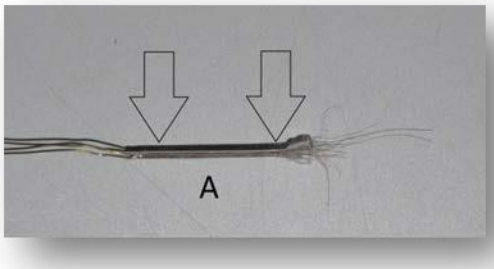
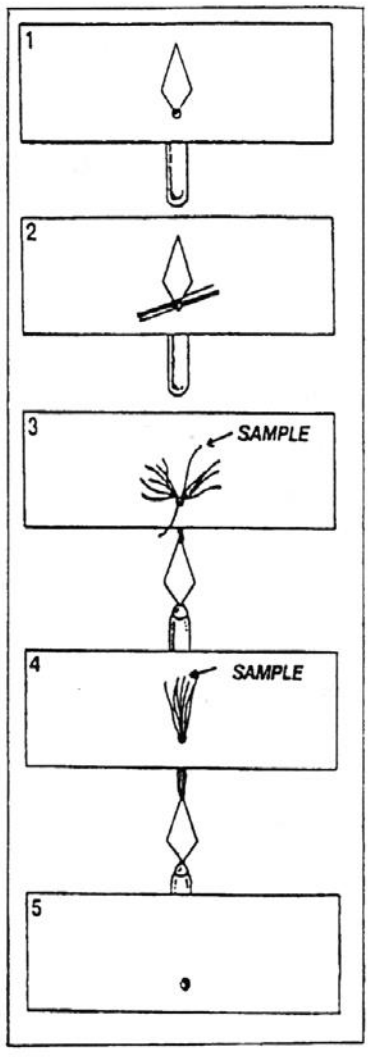


Image 3

er**Image 4**

CROSS-SECTIONING HAIRS AND FIBERS



Reference: Jolliff Cross-Section Kit, Industrial Analytical Laboratories, Fort Wayne, Indiana 46825

Procedure:

A. Push threader through hole in black cross-section plate.

B. Cut 4 2-inch lengths filler yarn of a color contrasting with the sample. Place these in the needle threader loop.

C. Pull the needle threader and filler yarn about 1/2 inch through the hole, leaving a tuft of filler yarn above the slide surface. Spread out the tuft and place a sample of the fiber to be examined across the tuft bundle.

D. Pull sample and filler yarn through the sample hole until the sample has partially passed through.

E. Cut filler and sample fiber above and below the slide with scissors. Cut filler and sample with a razor blade on both sides of the slide with a smooth even stroke.

F. A square around the cross-section can be cut out and mounted on a slide for permanent reference.

Additional cross-section slides available from Insulfab Plastics, Inc., 150 Union Avenue, East Rutherford, New Jersey 07073.

Note: Threader can be made by using a guitar string approximately 0.25 or 0.28 mm and a wall anchor.

9.4.5 Maceration process for the release of fiber ultimates from natural fiber samples:

1. Prepare a small beaker with water on a hot plate in a fume hood.
2. Add 2-3 boiling chips.
3. Heat the water to a gentle boil.
4. This is the vessel which will hold your individual sample tubes.

5. For each sample, pull out a technical fiber to macerate and place it into a test tube/scintillation vial. Pulling is important. Do not cut your samples. Unroll a twisted yarn and remove one fiber.
   For a sample with limited quantity, it may be best to pull out just one single technical fiber. Then if you only have one technical fiber, anything you observe about that sample must be generally applicable to the fiber as a whole. If ample sample is available, take multiple technical fibers and place them each in their own tube for individual digestion and analysis.

6. Prepare fresh maceration fluid each time. Mix equal parts 30% hydrogen peroxide and glacial acetic acid. Make enough to have 1 ml of maceration fluid for each single technical fiber sample.

7. Pipette enough maceration fluid into each tube to cover the tops of the fiber samples. Samples must be completely immersed in fluid.

Conduct the remainder of the process in a fume hood. Monitor the samples to ensure they do not evaporate to dryness as this may result in the formation of explosive peroxides.

8. Place the test tubes in the beaker of hot water and keep the water warm until the digestion process is complete. This is about 4-8 hours. Fibers will bubble with air and turn white in less than one hour. It is not necessary to continue to boil the water but it should be keep warm to hot. A good estimate for the hot plate setting is about 150-170°C.

9. When the fibers have broken down into a white slurry, carefully pour off the maceration fluid using a glass rod or similar to prevent the sample from falling out of the tube.

10. Add water and cover with either parafilm or a gloved fingertip. Shake vigorously by hand for 30-60 seconds or Vortex for 30 seconds to finish breaking up the ultimates.
11. Centrifuge, if possible, for approximately 3 minutes at 4,000 rpm and pour off the water again.

12. Wash, decant and centrifuge the fibers a second time using water.

13. Wash the samples with alcohol (70% ethanol, for example).

14. Use tweezers to remove the macerated sample and allow it to air dry in a clean vial before sealing. This sample is ready for further testing.

9.4.6 Solubility testing

The solubility of synthetic fibers in specific liquids and the fibers optical properties allows an examiner to identify the generic class of the fiber. Solubility tests are destructive and seldom allow an examiner to identify the sub-generic class of a fiber. For these two reasons, other non-destructive test should be run if the sample is of limited size.

Chemical Safety Considerations:

Chemical	NFPA Rating
acetic acid, glacial	3-2-1
acetone	1-3-0
acetonitrile	2-3-0
chloroform	2-0-0
cyclohexanone	1-3-0
dimethyl formamide (DMF)	1-2-0
formic acid	3-2-0
hexafluoroisopropanol	3-0-0

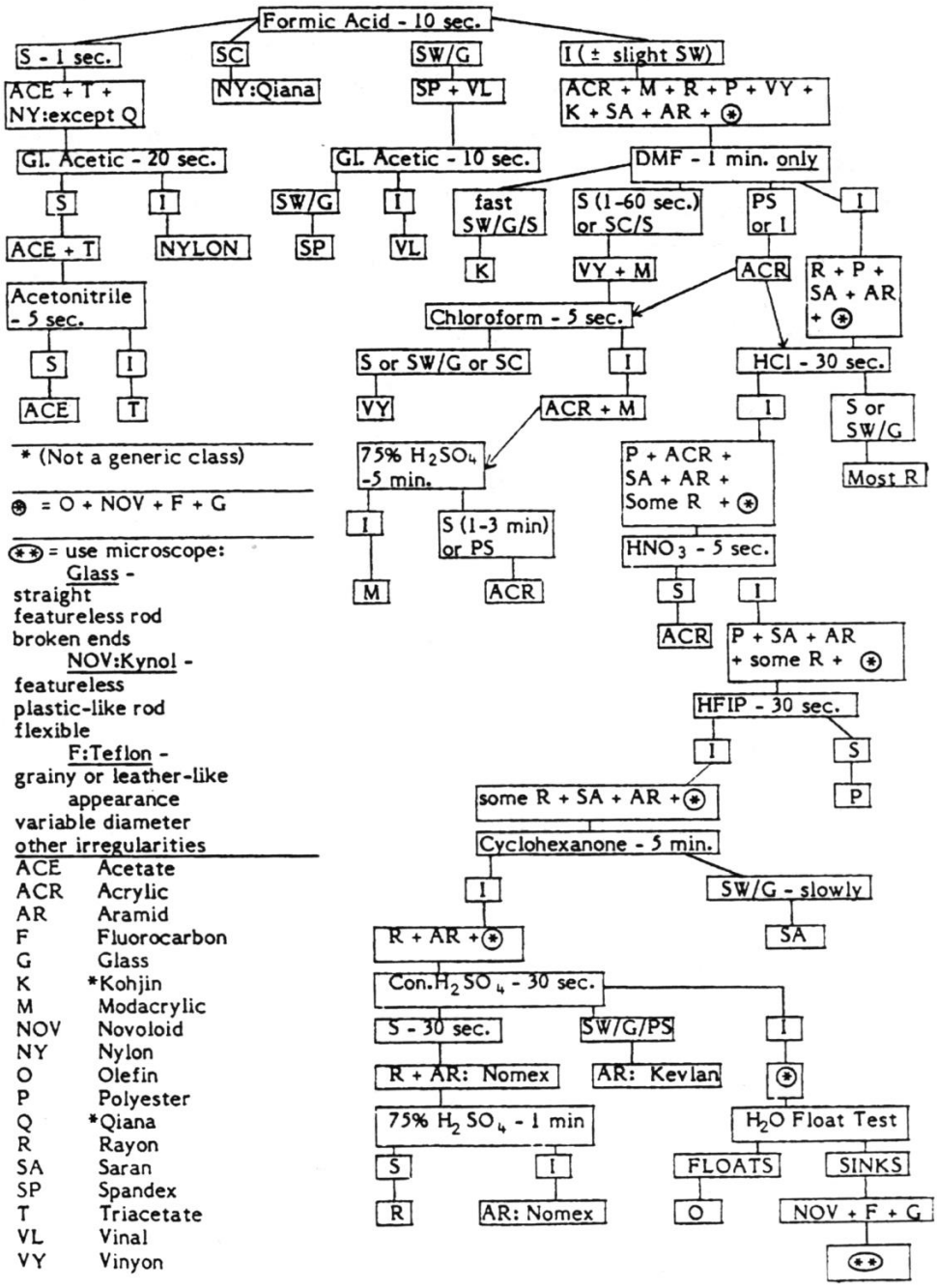
hydrochloric acid, conc.	3-0-0
nitric acid, conc.	3-0-0
sulfuric acid, conc. and 75%	3-0-2
water	0-0-0

Starting with the first solvent and working down the list, conduct solubility tests in the following manner:

1. Place a small piece of the fiber in a spot plate well of contrasting background
2. Place under the stereomicroscope
3. Focus on the fiber
4. Place a drop the selected reagent in the spot plate well with the fiber sample
5. Observe whether the fiber is soluble, insoluble, swells, gels, or shrinks
6. Compare the results with the selected chart and proceed to the next step
7. Continue until the fiber is identified.

Key to Solubility Reactions:

S = Soluble (fades, splinters, or breaks apart and goes into solution)
I = Insoluble (no reaction)
PS = Partly Soluble (not all portions of a fiber/all like fibers are soluble within 5 minutes)
SW = Swells
G = Gels (Plasticizes)
SC = Spinal Column (a fast SW/G which resembles a series of bones as in a backbone)



* (Not a generic class)

⊛ = O + NOV + F + G

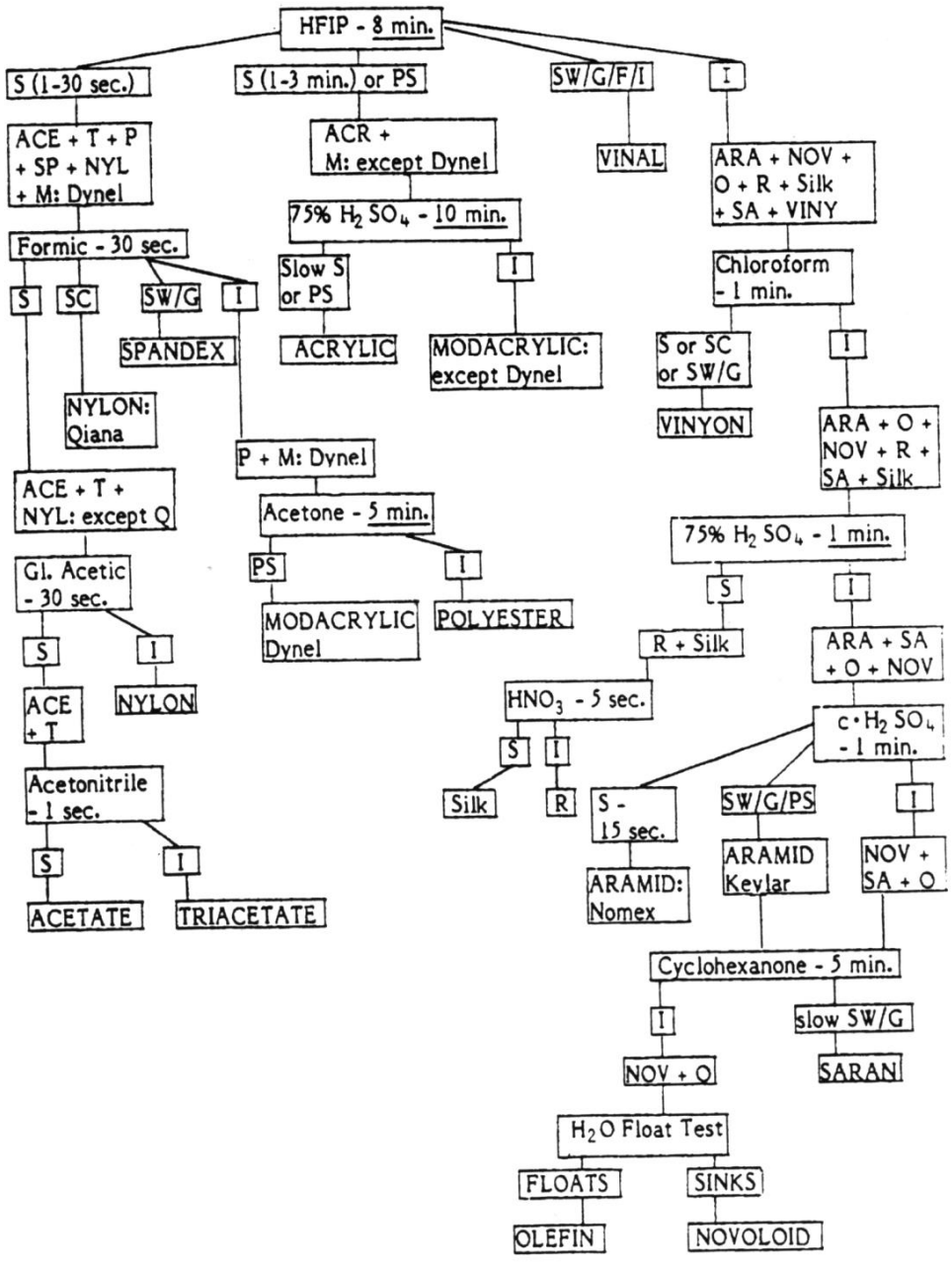
⊛⊛ = use microscope:

Glass -
straight
featureless rod
broken ends

NOV:Kynol -
featureless
plastic-like rod
flexible

F:Teflon -
grainy or leather-like appearance
variable diameter
other irregularities

ACE	Acetate
ACR	Acrylic
AR	Aramid
F	Fluorocarbon
G	Glass
K	*Kohjin
M	Modacrylic
NOV	Novoloid
NY	Nylon
O	Olefin
P	Polyester
Q	*Qiana
R	Rayon
SA	Saran
SP	Spandex
T	Triacetate
VL	Vinal
VY	Vinyon



These generic fiber classes are included in this scheme:

ACETATE	NYLON	SPANDEX
ACRYLIC	OLEFIN	TRIACETATE
ARAMID	POLYESTER	VINAL
MODACRYLIC	RAYON	VINYON
NOVOLOID	SARAN	(Silk is also included)

9.4.7 Infra-Red Spectroscopy (FTIR) Analysis

The generic classification of synthetic fibers has historically been determined by the combination of optical properties as observed via polarized light microscopy, solubility testing and melting-point determinations. Infrared analysis of fibers can provide information about fiber composition to supplement and confirm that obtained by the aforementioned methods. The American Society for Testing and Materials (ASTM) lists IR spectroscopy as the preferred method of analysis for identifying synthetic fibers (ASTM D276-87).

Due to differences in the crystalline nature among generic fiber classes, not all preparation techniques will give the best results for each fiber type. Acrylic, acetate, and triacetate fibers tend to crush easily and should not be pressed. Spandex, plasticized synthetic fibers, and irregular shaped fibers need to be compressed in order to hold a flattened shape. A diamond cell or two salt plates in a compression cell can be used when analyzing these types of fibers.
It is a good idea to gently roll the fiber on a frosted glass slide to slightly texture the surface of the fiber. This prevents reflection from the fiber appearing as interference fringes within the spectra.

See "Methods Manual: Instrumentation - FTIR" for additional information

9.4.8 Microspectrophotometry (MSP) Analysis

Visible microspectrophotometry is a non-destructive technique used for objective color analysis and comparison of fibers and other materials. Analysis of fibers by MSP is non-destructive and requires little sample preparation. The area to be tested should be flat and free of twists or bends. The sample can be rolled or pressed flat prior to mounting if needed. Questioned and standard samples should be prepared using the same stock of supplies and tested on the same day to eliminate non-sample sources of variation.

See "Methods Manual: Instrumentation - MSP" for additional information

Interpretation Criteria

The following possible conclusions can be reached after evaluating and comparing fibers:

Support for Same Source- The questioned fibers were found to the same as the known fibers with respect to class characteristics including color, physical characteristics, optical properties, and/or chemical composition with no significant unexplained differences.

Inconclusive – The questioned fibers were found to exhibit both differences and similarities to the known fibers with respect to physical characteristics, optical properties and/or chemical composition to the extent that no conclusion could be reached regarding an association or elimination. The significance of the differences cannot be completely assessed due to the constraints of sample size and/or condition.

Source Exclusion – The questioned fibers were found to be different from the known fiber with respect to color, physical characteristics, optical properties and/or chemical composition.

9.4.9 Fabric Comparison

Fabric comparisons begin with the examination of physical characteristics including weave pattern, thread count, dye pattern, and thread twist direction. Further examination is then conducted on fabric yarns from both directions. Since yarns may differ in composition from one another within one fabric or within a single yarn, and fiber composition may also vary within a single yarn, a complete segment of each yarn within a fabric should be teased out and mounted to reveal the fiber distribution within the sample.

Procedure

1. Count the number of yarns/unit length in each direction. Note: There will usually be more warp yarns/unit length than filling yarns.
2. Remove the warp yarn.
3. Determine the twist ("S" or "Z").

4. Count the number of crests and troughs/unit length.
5. On a simple one-over, one-under weave, the number of crests and troughs in the warp yarn should equal the number of filling yarns in the same unit length.

Use caution when counting or measuring loose weaves or those not constructed in a one-over, one-under pattern. One wave of the warp yarn should equal the width of a filling yarn in a tight weave. In a loose weave, the width of the weave may be much larger than the width of the filling yarn. Considerations should be made to collect measurements with a calibrated eyepiece.

In some cases where a weave goes over two or more fibers, it may be necessary to take two or more measurements.

Count the number of fibers in the yarns and evaluate the construction of the yarn.

The warp and fill yarns may be composed of fiber blends and these components should be determined. Other observations, measurement or comparisons should be conducted as appropriate for the samples which are being compared.

Interpretation Criteria

The following possible conclusions can be reached after evaluating and comparing fabrics:

Source Identification -The questioned fabric was compared to an edge or an area missing from the standard fabric and constitutes a matching individual tear/break configuration (See Physical Break Match/Tear Configuration Comparisons section in this manual).

Support for Same Source- The questioned fabric was found to be the same as the known fabric with respect to class characteristics including color, physical characteristics, optical properties, and/or chemical composition with no significant unexplained differences.

Inconclusive- The questioned fabric was found to exhibit both differences and similarities to the known fabric with respect to physical characteristics, optical properties and/or chemical composition to the extent that no conclusion could be reached regarding an association or elimination. The significance of the differences cannot be completely assessed due to the constraints of sample size and/or condition.

Source Exclusion - The questioned fabric was found to be different from the known with respect to color, physical characteristics, optical properties and/or chemical composition.

9.4.10 Fabric Damage

The manner of damage to textile materials sometimes becomes relevant in forensic analysis. This analysis may include determinations such as:

1. Whether a fabric item was cut, torn, abraded or melted.
2. Whether a cut originated from an implement such as scissors or a blade.
3. A range of the approximate size of implement which produced the damage

Procedure:

Conduct tests to simulate the damage observed on the questioned fabric or a sample of fabric consisting of approximately the same composition. Use of the suspected implement or method of damage should also be employed where applicable.

1. On fresh, clean catch paper, examine each item with emphasis on documenting and examining areas of damage to the fabric and documenting appropriate observations.
2. Draw a diagram and/or photograph the location, orientation, size, shape and appearance of any damaged areas.
3. Examine the edges of each defect using a stereomicroscope in an effort to determine if the fibers have been:
    - Crimped by double bladed implement.
    - Cut by a single bladed implement.
    - Broken by a blunt edged implement.
    - Stretched - It should be noted that in some instances fibers may exhibit stretching before they are cut by a dull blade or broken by a blunt edged implement.
4. Examine the edges of the fibers in the damaged area using a compound microscope or a scanning electron microscope.
5. Examine the prepared damage simulation samples and compare the visual and microscopic characteristics to the evidence fabric.

Interpretation Criteria

The following possible conclusions can be reached after evaluating the fiber damage:

Tear identified - Individual fibers and yarn ends reveal 'bearding'; the fabric reveals curled edges.
Cut Identified - Individual fibers and yarn ends reveal smooth cut ends and edges, possibly including angular cuts similar to razor tips; scissor cut fibers will often appear crimped (pointed) from having been compressed by the opposing scissor blades.
Tool Specified- Comparison between individual fibers and yarn ends and damage simulation samples prepared with known exemplars reveal consistent fiber ends and edge ends.

No cut/tear identified - Either no damage is noted or damage caused by melting, abrasion or other source is noted. The characteristics observed should be clearly documented in the analyst's notes.

9.5 References

Fiber Comparisons

1. Bartick, E. G., Tungol, M. W. Forensic Science Handbook Vol. III; Saferstein, R., Ed.; Prentice Hall: Englewood Cliffs, N.J., 1993, Chapter 4
2. Catling, D., Grayson, J., Identification of Vegetable Fibers; Chapman and Hall: New York, 1982
3. Gaudette, B. D., Forensic Science Handbook Vol. II; Saferstein, R. Ed., Prentice Hall: Englewood Cliffs, N.J., 1988, Chapter 5
4. Robertson, J., Ed. Forensic Examination of Fibers; Ellis Horwood: New York, 1992
5. Textile Institute. Identification of Textile Materials, 7th ed.; Textile Institute: Manchester, England, 1975
6. Forensic Fiber Examination Guidelines, Forensic Science Communications Vol 1, No.1 April 1999
7. Nielsen, M. R.; Common Natural Fibers, Bureau of Criminal Apprehension: October 1998
8. Stoeffler, S. F., Journal of Forensic Sciences; Volume 41 Issue 2, page 297; March 1996
9. Hicks, J. W., Microscopy of Hair- A Practical Guide and Manual, Federal Bureau of Investigation, Jan 1977
10. Forensic Examination of Fibres (Robertson & Grieve) Chapter 7 - Microscopical Examination of Fibers by S. Palenik, 2nd edition, p. 158)

Fabric and Yarn Comparisons

1. Forensic Fiber Examination Guidelines, Forensic Science Communications Vol 1, No.1 April 1999
2. Bodziak, William J., Footwear Impression Evidence, Elsevier: New York, 1990

3. Forensic Fiber Examination Guidelines, Forensic Science Communications Vol 1, No.1 April 1999

Fabric Damage

1. Hearle, J. W. S., Lomas, B. and Cook, W. D. “Atlas of Fibre Fracture and Damage to Textiles”, The Textile Institute, CRC Press 1998
2. Forensic Human Hair Examination Guidelines, Forensic Science Communications Vol 1, No.1 April 1999

10 Paint Analysis

10.1 Introduction

Paint evidence can be examined and compared visually, microscopically, chemically, and instrumentally. Characteristics including color, tint, texture, relative thickness, layer structure, organic, and inorganic composition may be used to compare samples. The order and type of examination will be dictated by the samples size and condition.

10.2 Equipment/Instrumentation

- Hotplate
- Spot plate
- Stereomicroscope
- Comparison microscope
- Polarized light microscope
- FTIR
- SEM/EDS
- XRF
- Sonicator

10.3 Minimum Standards and Controls

Each time a prepared reagent is used, appropriate blanks and controls will be used and documented on a reagent form and in the case notes.

10.4 Analysis Methods

10.4.1 Microscopic Examination

Each item should be examined separately on a clean sheet of paper. If the samples are appropriate for a break match examination, proceed with that examination.

A PLM may be used for the identification and/or comparison of some paint components, including paint pigments. These samples are effectively examined in the form of permanently mounted individual paint layer peels. Comparison of the microscopical characteristics of the corresponding paint layers provides good discrimination based on color, pigment distribution and overall layer appearance. Advanced training and experience is required to identify pigments based solely on PLM examination.

10.4.2 Chemical reactivity (Spot Tests)

Chemical reactivity tests are destructive and may, therefore, be avoided during examination.

1. Prepare the surface by washing it with a laboratory grade detergent followed by rinsing it with distilled water and drying. The sample may also be cleaned by sonication by wiping gently with a moistened cotton swab.
2. Place known and unknown paint chips in a spot plate in adjacent wells to allow for their simultaneous observation. Label each sample well.
4. Six different chemical reagents can be used to examine paints. These reagents and the reactions expected with them are listed below. Apply reagent directly onto each chip.

- Use 1-2 drops. When necessary, samples are submerged with an item such as a glass rod or probe tip.

REAGENT	REAGENT PREP	PAINT	REACTION
Methyl Ethyl Ketone (MEK)	Reagent Grade	Acrylic Lacquer Acrylic Enamel Nitrocellulose	Soluble Insoluble Soluble
Diphenylamine (DPA)	Add 0.25g of diphenylamine to 80% H2SO4 or 0.3g of diphenylamine to 20 ml concentrated H2SO4 and 10ml glacial acetic acid.	Nitrocellulose	Turns dark blue and color bleeds out into reagent. Pigments may turn different colors.
Chloroform	Reagent Grade	Acrylic Lacquer Acrylic Enamel Nitrocellulose	Soluble Insoluble Insoluble
Acetone	Reagent Grade	Acrylic Lacquer Acrylic Enamel Nitrocellulose	Soluble Insoluble Soluble
Concentrated Sulfuric Acid	H2SO4, (18.0M)	Pigments	May turn different colors.
Le Rosen	10 drops of 37% Formaldehyde to 10 mL conc. H2SO4	Pigments-House Paints	May turn different colors.

5. The effect of each reagent on the individual layers of one chip is compared to that of the corresponding layers of the other chip.
6. Observe and record the following qualities which apply:
    - Soluble
    - Partially soluble
    - Pigment leach
    - Swell
    - Curl
    - Discolor
    - Soften
    - Layer separation
    - Gas bubble formation
    - Other reactions
    - No reaction
7. The reactions given by each corresponding layer must be identical.
8. Reactions can be observed immediately, one and three minutes after reagent is applied, *or as documented in the case notes.*

9. Proceed with other reagents as sample quantity permits. A new paint chip should be used with each chemical reagent.
10. Be sure to retain sufficient sample for possible further analysis.

10.4.3 Instrumental analysis techniques

10.4.3.1 Fourier Transform Infrared Spectroscopy (FTIR)
FTIR can provide important information related to the chemical composition of the paint being examined. This information can be used in the classification of the paint film or a means of finding significant similarities or differences in the composition of the unknown as compared to the standard.

This technique is non-destructive and facilitates small sample analysis.

Transfers resulting in smears on various substrates may be analyzed in situ using an attenuated total reflectance (ATR) accessory. In situ analyses may require analysis of the substrate as a control.

FTIR analysis of paint samples can be conducted using transmission or ATR methods. Evaluating the results of these comparisons is described in the FTIR section of the manual.

10.4.3.2 Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy (SEM/EDS) and XRF
The SEM / EDS and XRF allow the analyst to obtain elemental information about the sample by collecting and processing the X-rays from the sample for chemical element identification.

The SEM may also be used for visual examination and comparison of the samples.

Composition comparisons are generally performed on a qualitative, not quantitative, basis using direct spectral comparisons or peak ratios. Lack of homogeneity in paint limits the value of obtainable quantitative information.

10.5 Paint Data Query (PDQ)

10.5.1 Introduction
In cases involving an accident or death where there is no suspect vehicle, original automobile paint that has been recovered may be coded and searched in the PDQ database. The coding involves analysis of the sample using FTIR. A PDQ search may provide manufacturer (make, model, and year) information about the suspect vehicle. The paint must be original paint to be searched. The database is user contributed and it should be understood that not all make, models, and year ranges will be represented.

NOTE: As a participating user in the PDQ system, Ohio BCI is required to provide a specified number and type of automotive samples from known vehicles to augment the database. Laboratory management is responsible for ensuring the collection of those samples.

10.6 Interpretation Criteria

The following possible conclusions can be reached after evaluating and comparing paint:

Source Identification -The questioned paint chip was compared to an edge or an area missing from the known paint and constitutes a matching individual break configuration (See Physical Break Match/Tear Configuration Comparisons section in this manual).

Support for Same Source–The questioned paint was found to be the same as the known with respect to class characteristics including color, physical characteristics, chemical composition, and/or elemental composition with no significant unexplained differences.

Inconclusive- The questioned paint was found to exhibit both differences and similarities to the known paint with respect to physical characteristics, chemical composition and/or elemental composition; to the extent that no conclusion could be reached regarding an association or elimination. The significance of the differences cannot be completely assessed due to the constraints of sample size and/or condition.

Source Exclusion- The questioned paint was found to be different from the known paint with respect to color, physical characteristics, chemical composition, and/or elemental composition.

10.7 References

1. Cartwright, L. J.; Cartwright, N.S.; Norman, E.W.W.; Cameron, R.; MacDougall, D.A.; Clark, W.H.; "The Classification of Automotive Paint Primers Using the Munsell Color Coordinate System - A Collaborative Study."; Canadian Society of Forensic Science Journal: 1984, Vol. 17, pp 14-18.
2. Cassista, A.R.; Sandercock, P.M.L.; Comparison and Identification of Automotive Topcoats: Microchemical Spot Tests, Microspectrophotometry, Pyrolysis-gas Chromatography, and Diamond Anvil Cell FTIR.; Canadian Society of Forensic Science Journal: 1994, Vol. 27, pp 209-223.
3. Castle, D.A.; Pigment Analysis in the Forensic Examination of Paints II. Analysis of Motor Vehicle Paint Pigments by Chemical Tests.; Journal of Forensic Science Society: 1982 Vol. 22, No. 2, pp 179-186.
4. Forensic Paint Analysis Guidelines, Forensic Science Communications Vol 1, No.2 July 1999.
5. Gothard, J.A.; "Evaluation of Automobile Paint Flakes as Evidence."; Journal of Forensic Sciences: 1976, Vol. 21, No. 3, pp 636-641.
6. Hamer, P.S.; "Pigment Analysis of the Forensic Examination of Paints III. A Guide to Motor Vehicle Pain Examination by Transmitted Light Microscopy."; Journal of Forensic Science Society: 1982, Vol. 22, No. 22, pp 187-192.
7. Linde, H.G.; Stone, R. P.; Application of the LeRosen Test to Paint Analysis.
8. Journal of Forensic; Science: 1979, Vol. 24, No. 3, pp 650-655.
9. P.D.Q. Software User Manual, Code Book, Database Contents, Spectral Information manuals Updates provided via the RCMP PDQ Maintenance Team
10. Ryland S. G.; Kopec, R.J.; "The Evidential Value of Automobile Paint Chips."; Journal of Forensic Sciences: 1979, Vol. 24, No. 1, pp 140-147.

11 Gunshot Primer Residue Analysis

11.1 Introduction

When a weapon is fired, a number of events occur immediately after the primer is struck. Initially the primer composition is crushed, causing ignition of the mixture. Ignited primer moves forward and ignites the propellant. Burning propellant produces a large volume of highly pressurized gases which force the projectile(s) out of the cartridge case and down the weapon barrel. As this happens, gases vent out of the barrel, cylinder gap, ejection port or other unsealed areas. These gases then rapidly cool and deposit on any nearby surface. The products of firearm discharge are collectively referred to as gunshot residue (GSR). Collection of GSR and subsequent analytical identification of primer components lead, barium and antimony can be used to associate an individual with a discharged firearm.

11.2 Equipment/Instrumentation

- Circular aluminum disks with adhesive collection surface and pin mounts
- Sample tweezers
- Carbon rods
- Carbon tape
- Reagent grade alcohol
- Sample collection kit, such as the Tri-Tech brand (Ohio Model)
- Scanning electron microscope equipped with an energy dispersive x-ray spectrometer
- Carbon coater
- Individual aluminum GSR collection stubs in vial

11.3 Minimum Standards and Controls

11.3.1 Analytical QA/QC

Known gunshot residue samples (positive controls) are to be analyzed under the following conventions:

A synthetic GSR positive control will be analyzed once per month. Documentation of the monthly positive control runs must be retained in a designated log.

- The monthly positive control shall be considered to have “passed” if the automated run finds and correctly identifies at least 90% of the 1 micron and larger Pb-Ba-Sb particles present on the analysis area of the sample.

- The positive control shall be considered to have “failed” if the appropriate minimum threshold of Pb-Ba-Sb particles cannot be identified. If the positive control fails, the instrument in question will not be used for GSR evidence analysis until the problem can be identified and corrected.

A laboratory produced GSR positive control will be analyzed at the end of each batch of sample stubs, prior to analysis of the blank stub. The positive control sample should be stored to protect it from loss and degradation.

- The batch run positive control shall be considered to have “passed” upon the automatic identification and subsequent user confirmation of at least 3 Pb-Ba-Sb particles.

- A copy of the positive control sample analytical data must be retained as part of the case notes for each case included in a sample batch.
- If the positive control fails during a batch run, any negative case sample findings in that analytical batch shall be reanalyzed once the problem has been identified and corrected.

11.3.1.1 A negative control (blank sample stub) will be analyzed at the end of each batch of sample stubs. These samples will be of like design and subjected to the same preparation and analysis procedures as case samples.

- A negative control shall be considered to have "passed" if no Pb-Ba-Sb, Pb-Ba, Pb-Sb, or Ba-Sb particle is identified. These stubs may be discarded after the analysis has been completed.
- A copy of the negative control sample analytical data must be retained as part of the case notes for each case included in a sample batch.
- The negative control shall be considered to have "failed" upon the automatic identification and subsequent user confirmation of at least one Pb-Ba-Sb, Pb-Ba, Pb-Sb, or Ba-Sb particle.
- If the negative control fails during a batch run, any positive case sample findings in that analytical batch shall be considered inconclusive. Any negative case samples within that analytical batch shall remain negative.

11.3.1.2 Contamination control

- Sample preparation and analysis must be conducted in an environment that restricts potential gunshot residue contamination.
- All sample manipulation utensils must be cleaned with reagent grade alcohol between samples.
- Cleaning of the sample preparation area and analysis equipment must be performed prior to each run.

11.4 Sample Prep Procedures

11.4.1 In-house clothing/surface collection

1. Put on disposable gloves.
2. Remove the cap with attached aluminum stub from vial.
3. While holding cap, repeatedly press collecting surface on sample area until desired area has been sampled, or until collection surface loses its adhesive quality.
4. Return the cap and stub to the vial.
5. Repeat steps 2 through 4, as necessary.
6. Label and mark vials, as required.

11.4.2 Analytical progression

The intent of this test is to establish the presence or absence of particles commonly associated with gunshot primer residue and to associate that finding with an individual. This is generally best accomplished by the identification of Pb-Ba-Sb particles on samples collected from the individual's hands. The analysis of clothing or other items generally does not satisfy the criteria given above, and should be discouraged; however, they may be sampled and analyzed when case facts warrant it.
In order to provide valuable analytical information in a productive and timely manner, the following method of batching and analytical progression will be followed, unless circumstances dictate otherwise.

11.4.2.1 Batching

- Multiple GSR kits may be run with each batch in order to efficiently utilize the SEM sample stage.
- Multiple cases may be analyzed together to accomplish this.

11.4.2.2 Hand samples

- Analysis will begin with one sample from the left hand and one sample from the right hand.
- In the event that a submitted GSR kit contains multiple samples from each hand or samples from other body areas, a maximum of one sample from each hand will typically be analyzed and preference will be given to samples collected from the back of the hand over those collected from the palm of the hand.

11.4.2.3 Clothing and surface samples

- Clothing and/or surface samples associated with an individual or item may be analyzed if hand samples test negative or are unavailable.
- Sample progression will move from areas most likely to contain GSR toward areas less likely, based on the case facts.
- Analysis of samples associated with one individual or item can be halted when one sample is found to be positive or when a sufficient representation of an area or an item has been analyzed.

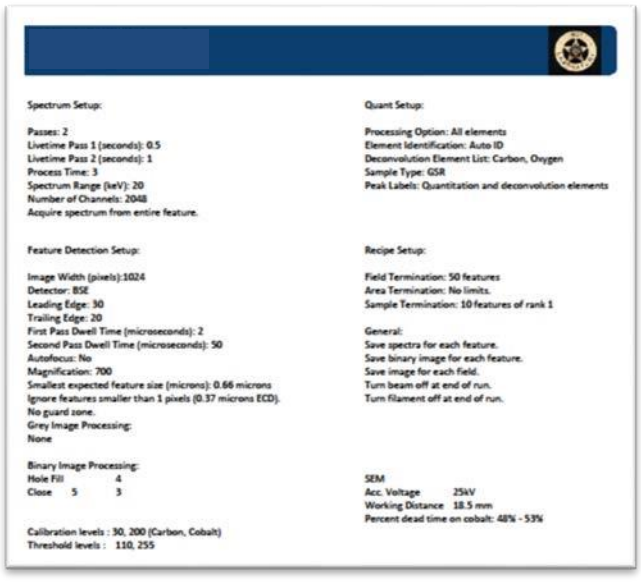
11.5 Testing Parameters

An instrument method with the following Recipe Setup should be used for SEM & EDS for GSR analysis:

Recipe Setup:

Field Termination: 50 features
Area Termination: No limits.
Sample Termination: 10 features of rank 1

One of the following methods should be used. The specific instrument and run parameter method name used will be documented in the case notes.



Spectrum Setup:

Passes: 2
Livetime Pass 1 (seconds): 0.5
Livetime Pass 2 (seconds): 1
Process Time: 3
Spectrum Range (keV): 20
Number of Channels: 2048
Acquire spectrum from entire feature.

Quant Setup:

Processing Option: All elements
Element Identification: Auto ID
Deconvolution Element List: Carbon, Oxygen
Sample Type: GSR
Peak Labels: Quantitation and deconvolution elements

Feature Detection Setup:

Image Width (pixels):1024
Detector: BSE
Leading Edge: 30
Trailing Edge: 20
First Pass Dwell Time (microseconds): 2
Second Pass Dwell Time (microseconds): 50
Autofocus: No
Magnification: 700
Smallest expected feature size (microns): 0.66 microns
Ignore features smaller than 1 pixels (0.37 microns ECD).
No guard zone.
Grey Image Processing:
None

Recipe Setup:

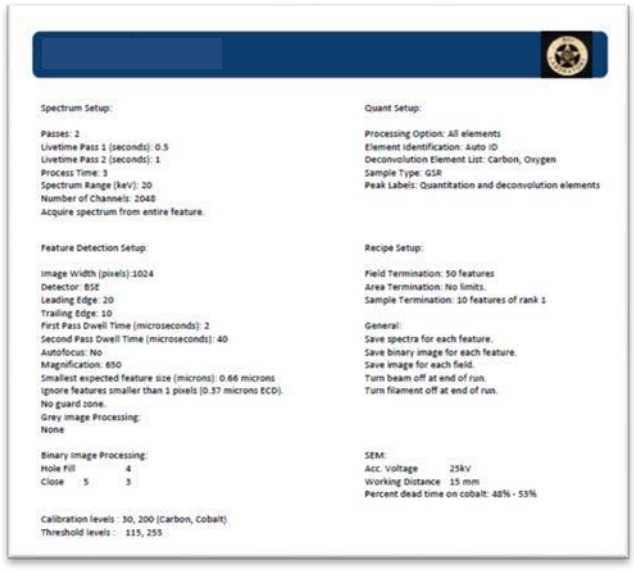
Field Termination: 50 features
Area Termination: No limits.
Sample Termination: 10 features of rank 1

General:
Save spectra for each feature.
Save binary image for each feature.
Save image for each field.
Turn beam off at end of run.
Turn filament off at end of run.

Binary Image Processing:
Hole Fill 4
Close 5 3

SEM
Acc. Voltage 25kV
Working Distance 18.5 mm
Percent dead time on cobalt: 48% - 53%

Calibration levels : 30, 200 (Carbon, Cobalt)
Threshold levels : 110, 255



Spectrum Setup:

Passes: 2
Livetime Pass 1 (seconds): 0.5
Livetime Pass 2 (seconds): 1
Process Time: 3
Spectrum Range (keV): 20
Number of Channels: 2048
Acquire spectrum from entire feature.

Quant Setup:

Processing Option: All elements
Element Identification: Auto ID
Deconvolution Element List: Carbon, Oxygen
Sample Type: GSR
Peak Labels: Quantitation and deconvolution elements

Feature Detection Setup:

Image Width (pixels):1024
Detector: BSE
Leading Edge: 20
Trailing Edge: 10
First Pass Dwell Time (microseconds): 2
Second Pass Dwell Time (microseconds): 40
Autofocus: No
Magnification: 650
Smallest expected feature size (microns): 0.66 microns
Ignore features smaller than 1 pixels (0.37 microns ECD).
No guard zone.
Grey Image Processing:
None

Recipe Setup:

Field Termination: 50 features
Area Termination: No limits.
Sample Termination: 10 features of rank 1

General:
Save spectra for each feature.
Save binary image for each feature.
Save image for each field.
Turn beam off at end of run.
Turn filament off at end of run.

Binary Image Processing:
Hole Fill 4
Close 5 3

SEM:
Acc. Voltage 25kV
Working Distance 15 mm
Percent dead time on cobalt: 48% - 53%

Calibration levels : 30, 200 (Carbon, Cobalt)
Threshold levels : 115, 255

11.6 Data Collection

Automated sample analysis may be terminated when any of the following criteria are met:

- Entire user described area has been analyzed.
- A total of ten (10) Pb-Ba-Sb particles have been identified on any combination of samples from an individual, single surface or item.
- A user specified time or total particle maximum has been reached.

Attended sample analysis may be terminated at any time, upon identification of characteristic gunshot residue particle(s).

Pb-Ba-Sb particles identified through automated analysis shall be relocated and confirmed. A detailed, quality image of each particle analyzed should be included in the case notes.

11.7 Interpretation Criteria

The following possible conclusions can be reached after evaluating GSR samples:

Positive (3-component particles) - characteristic of GSR
A positive finding for the presence of particles characteristic of gunshot primer residue shall occur with the automatic identification and subsequent user confirmation of at least one Pb-Ba-Sb particle exhibiting characteristic GSR morphology and supported by a sufficient number of automatically identified one or two-component GSR supporting particles (Pb, Ba, Sb, Pb-Ba, Pb-Sb and/or Ba-Sb) in the remaining particle population.

The particle population should NOT contain:

- major levels of iron (indicates particles consistent with brake lining)
- major levels of aluminum and magnesium in a single particle (indicates particles consistent with fireworks)
- major levels of copper and cobalt (indicates particles consistent with airbags)

Note: major amounts of sulfur, barium, magnesium, sodium, cobalt, manganese, zirconium, chromium, or titanium may be indications of non-firearms sources. Refer to the cited materials in this section for more information.

Elimination
A negative finding for the presence of particles characteristic or consistent of gunshot primer residue shall occur when the above conditions for a “positive” finding cannot be met on any analyzed sample associated with an individual or item.

Inconclusive

An inconclusive finding for the presence of particles characteristic of gunshot primer residue shall occur when the case sample findings are positive and the blank (negative control) run with that batch of samples is also deemed to be positive.

11.8 References

1. SWGGSR. "Guide for Primer Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray Spectrometry." November 29, 2011. Web. 03 Feb. 2012. <http://www.swggsr.org/documents.html>
2. ASTM E1588, "Standard Guide for the Analysis of Gunshot Residue by Scanning Electron Microscopy/Energy Dispersive Spectroscopy," 2005, ASTM: Philadelphia, PA.
3. Matricardi, V. R. and Kilty J. W., "Detection of Gunshot Residue Particles from the Hands of a Shooter," Journal of Forensic Sciences, Vol. 22, No. 4, October 1977, pp. 725-738.
4. Schwoeble, A.J., Exline, D.L., "Current Methods in Forensic Gunshot Residue Analysis", CRC Press LLC, 2000.
5. Tillman, J., "Automated Gunshot Residue Particle Search and Characterization," Journal of Forensic Sciences, Vol. 32, No. 1, January 1987, pp. 62-71.
6. Wallace, J. S. and McQuillan, J., "Discharge Residues from Cartridge Operated Industrial Tools," Journal of Forensic Sciences, Vol. 24, 1984, pp. 495-508.
7. Ward, D. C., "Gunshot Residue Collection for Scanning Electron Microscopy," Scanning Electron Microscopy, Vol. 3, 1982, pp. 1031-1036.
8. White, R. S. and Owens, A. D., "Automation of Gunshot Residue Detection and Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray Analysis (SEM/EDX)," Journal of Forensic Sciences, Vol. 32, No. 6, November 1987, pp. 1595-1603.
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 Corporation, El Segundo, CA, September 1977.
10. Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., "Particle Analysis for the Detection of Gunshot Residue I: Scanning Electron Microscopy/Energy Dispersive X-Ray Characterization of Hand Deposits from Firing," Journal of Forensic Sciences, Vol. 24, No. 2, April 1979, pp. 409-422.
11. Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., "Particle Analysis for the Detection of Gunshot Residue. II: Occupational and Environmental Particles," Journal of Forensic Sciences, Vol. 24, No. 2, April 1979, pp. 423-430.
12. Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., "Particle Analysis for the Detection of Gunshot Residue. III: Case Study," Journal of Forensic Sciences, Vol. 24, No. 4, October 1979, pp. 864-869.
13. Wrobel, H.Millar, J.J. and Kijek, M. "Identification of Ammunition From Gunshot Residues and Other Cartridge Related Materials—A Preliminary Model Using .22 Caliber Rimfire Ammunition." Journal of Forensic Sciences, 1998, pp. 324-328.
14. Mosher,P.V., et.al., "Gunshot Residue- Similar Particles Produced by Fireworks," Can Soc. Forens.Sci.J., 31 (20) 1998.
15. Berk, R.E. Automated SEM/EDS Analysis of Airbag Residue. I. Particle Identification. Journal of Forensic Sciences, Vol. 54, No. 1, January 2009, pp. 60-68.
16. Berk, R.E. Automated SEM/EDS Analysis of Airbag Residue. II: Airbag Residue as a Source of Percussion Primer Residue Particles. Journal of Forensic Sciences, Vol. 54 No. 1, January 2009, pp. 69-76.

17. Torre, C.A Source of Non-GSR Particles containing Lead, Barium and Antimony, Journal of Forensic Sciences, 2002, (47), pp. 474-504.
18. Wallace, James Smyth. “Chemical Analysis of Firearms, Ammunition, and Gunshot Residue”, CRC Press, 2008.

12 Glass Analysis

12.1 Introduction

Glass examination involves visual observations, physical tests and elemental analysis tests in order to conclude the identifying characteristics of the glass evidence. The results of the evidence glass can then be compared to known reference standards.

The refractive index (RI) determination is the most commonly measured physical property of glass because the required sample size is small. The refractive index is measured microscopically using silicone oil with a known refractive index.

The rIQ instrument can be utilized for a variety of glass samples and is most often used for the analysis of glass samples between the RI's of 1.48 and 1.55. The following are approximate n_d ranges for the RI of typical glass products:

- Flat 1.5081-1.5343
- Headlight (borosilicate) 1.4745-1.4827
- Container 1.5141-1.5276
- Tableware 1.4735-1.5600

The micro-XRF is a nondestructive elemental analysis technique that utilizes an X-ray source and capillary optics to excite the atoms of a sample. When excited by the X-ray beam, individual atoms emit X-ray photons of a characteristic energy. Spectral and elemental ratio comparisons of the glass specimens are conducted for source discrimination or association.

It is sometimes necessary to determine the direction of force applied to a window causing it to fracture. Determinations may be made from samples which have been damaged by a projectile or blunt force. This analysis may be conducted on samples which have damage in the form of either a hole (crater) in the pane caused by the projectile passing or fracturing from an applied force or projectile.

12.2 Minimum Standards and Controls

12.2.1 Annually/As needed: rIQ Calibration Curve

For rIQ, establish a new calibration curve on a yearly basis, anytime new immersion oil is used, and/or when indicated by the reference glass data.

12.2.2 Day of Use: Refractive Index Known Reference samples

The refractive index of two known reference glass samples (at least one of which was not used to create the calibration curve) should be collected each day casework is analyzed, before any casework data is obtained. This check assures that the rIQ instrument is set up and working correctly and that the immersion oil's refractive index properties have not changed.

The reference glass samples should have known refractive index values that are suitable to check two different points along the calibration curve. A minimum of three good quality edges per reference glass should be used.

The measured RI value of each reference glass should be compared to the stated RI for that glass (the refractive index values may need to be converted to standardized conditions for comparison). If measurements fall outside of 0.00010 of the stated RI, action may need to be taken. These actions may include preparing a new sample, using new immersion oil, checking the temperature of the hot stage, or creating a new calibration curve.

12.2.3 Day of Use: Micro-XRF Energy Calibration

The µ-XRF must be calibrated each day of use, prior to casework analysis.

Using the calibration function within the instrument's software and a calibration standard (typically aluminum and copper); calibrate the instrument at each Time Constant that will be used during casework analysis. The calibration data is automatically stored by the software.

12.2.4 Day of Use: Micro-XRF Source Performance Check

The source performance check must be performed each day of use, prior to casework analysis.

Check the performance of the X-ray source using a known element standard (for example, Cu). Maximum counts for the system should be obtained utilizing the following instrument settings: 50 kV, 50 µA, 12.8µs Amp Time. Monitor the count rate for approximately 10 counts during a collection, and record the maximum value observed. The maximum counts should not deviate from the previous reading by more than 10%.

12.2.5 Day of Use: Micro-XRF System Performance Check

The system performance check must be performed each day of use, prior to casework analysis.

For screening spectral comparisons: Demonstrate that the SNR of Ti in NIST SRM 1831 is at least 8 for the parameters to be used for casework.

For detailed spectral comparisons and statistical evaluations: Demonstrate that the SNR of Ti in NIST SRM 1831 is at least 10 for the parameters to be used for casework.

If the SNR of Ti in NIST SRM 1831 is under 8 for a set of screening parameters, or under 10 for a set of detailed comparison parameters, the typical parameters should be modified or instrument maintenance may be required. The spectrum and the channel intensity data must be stored (electronic or hard copies).

12.2.6 Micro-XRF Run Stability

If quantitative or semi-quantitative statistics will be evaluated, a reference sample (such as NIST SRM 1831) should be analyzed at the beginning and end of each multi-point run to verify the stability of the instrument throughout the run and to allow for inter-day comparison of data. The two reference sample spectra should demonstrate consistency by spectral overlay.

To allow for comparison of data from different runs/days, reference sample spectra for those days should demonstrate consistency by spectral overlay, and the run parameters should be similar.

12.2.7 Micro-XRF Control Samples

For thin samples (less than 1 mm), a control sample should be collected to assess the possibility of elemental contribution from other sources (mounting adhesive, substrate, etc.). This may be collected concurrent with casework samples in a multi-point run or may have been collected at a different time.

12.3 Equipment/Instrumentation

- Stereomicroscope
- Polarizing light microscope
- Glass standards
- Sonic cleaner
- rIQ (Refractive Index Quantification) instrument equipped as follows:
    - Phase contrast microscope with provision for long working distances
    - Filters (“D” RI 589, “F” RI 488 “C” 655)
    - Hotstage
    - Dedicated software controller unit
    - Monitor and computer with rIQ software
- Shortwave UV light
- XRF
- Mounting platform for glass fragments
- Kapton film
- Adhesive with low content of heavy elements
- Copper standard
- Copper/aluminum alloy standard

12.4 Analysis Methods

12.4.1 Visual Examination

Examine the evidence visually. If the sample is of an appropriate size and shape the possibility of a physical break match between the known and questioned sample should not be over looked.

Document observations to include color, glass type, shape, surface texture, inclusions and surface markings whenever possible.

To characterize a substance as glass, check the particles for characteristic fracture edges, isotropism, hardness and/or solubility as indicated below:

- Fracture - conchoidal fractured
- Amorphous - exhibits isotropic properties when viewed under crossed polars using a polarizing light microscope
- Hardness – does not indent or crush when compressed
- Solubility - insoluble in water or organic solvent

Manufactured glass surfaces should also be checked for ultra violet fluorescence at short and long wavelengths to determine if the glass is float glass. The float glass surface may exhibit colors that range from yellow, orange to blue.

12.4.1.1.1 Glass Thickness

A thickness measurement with a micrometer or caliper (preferably one with a precision of ±0.02 mm) can be performed if two parallel (flat) manufactured glass surfaces are available. Multiple samples from the known glass should be measured to determine the range of thickness. The measurement of the questioned glass sample must fall within the range of the known measurements ± the precision of the micrometer or caliper for the known glass to be included as a possible source for the questioned glass.

12.4.2 Refractive Index analysis using Craic Technologies rIQ

Questioned glass data will be evaluated for suitability prior to being used for a comparison.

Sample Preparation

A glass sample that has been selected for instrumental analysis should be cleaned to remove any surface contamination, if sample size permits.

Cleaning may include washing the sample in soap and water (with or without ultra-sonication) rinsing with deionized water, followed by rinsing in acetone, methanol or ethanol and then allowing to dry. For more difficult contaminants, soaking in various concentrations of nitric acid for 30 minutes or longer, followed by the above rinsing procedure removes most surface contamination without affecting the measured concentrations of elements inherent in the glass; however, the use of nitric acid may remove any surface coating that may be present.

Known standards

To ensure that the standard is properly sampled, a representative number of fragments are randomly picked out from the known sample. If one large piece of glass is submitted, include samples from opposite areas of the glass pane.

If sample size permits, at least 20 measurements should be taken of every standard for comparison that is tempered and at least 10 measurements should be taken of every standard for comparison that is not tempered.

To ensure that the complete RI range of the comparison standard is measured, each slide should be checked by either observing the slide at the match point temperature to find any glass still visible, or by raising the temperature 1˚C to determine whether any glass of slightly lower RI becomes visible. These may be indications of surface fragments.

Note: Surface fragments may have a different RI than the bulk glass RI. The float surface of flat glass typically has a lower RI than the bulk glass. The surface opposite the float surface typically has an RI higher than the bulk. Container glass may also exhibit surface anomalies; however, the RI's can either be higher or lower than the bulk RI.

Questioned samples

Each questioned glass fragment is treated as a separate sample, even if they were all recovered from the same item. Therefore, only one questioned fragment is mounted per slide.

The size/condition of the questioned glass fragment dictates the number of edge measurements collected.

If the questioned glass fragment is large enough, a portion of it should always be retained for elemental analysis and possible future reanalysis.

For a refractive index (RI) comparison to be considered a match the mean RI of the Questioned glass must fall within the range of the Known glass (+/- the measurement uncertainty of the instrument which is .00002 for the Craic rIQ instrument).

12.4.3 Elemental analysis

Elemental Analysis should be preceded by visual examination and thickness comparison (if full thickness fragments). Elemental analysis may be performed before or after refractive index analysis. The order of analysis will be influenced by the size and shape of the questioned fragment(s), and by which method may provide the fastest discrimination given the specific circumstances of the case. Consideration should be given to the fact that refractive index analysis is a semi-destructive technique, whereas elemental analysis is a non-destructive technique.

Fragments should be of sufficient size in order to be analyzed elementally on the micro-XRF.

Elemental spectra used for qualitative comparison of elemental composition will be evaluated for suitable quality prior to any comparison. Elemental peaks used for qualitative comparisons will have a signal to noise ratio of greater than equal to or greater than 3.

Elemental spectra used for semi-quantitative comparison of elemental ratios will be evaluated for suitable quality prior to any comparison. Elemental peaks used for semi-quantitative comparison of elemental ratios will have a signal to noise ratio equal to or greater of greater than 10.

Sample Preparation

Full-thickness float glass samples should be mounted float side down. Thinner fragments should be mounted to prevent elemental contribution from non-evidentiary sources (e.g. on X-ray permeable film (such as Kapton) using an adhesive that will not interfere with the sample spectra). For samples with coatings (including float side) on both sides, the fractured surface (bulk glass) may be analyzed. Because the precision of µ-XRF is somewhat dependent on geometry and thickness, the questioned and known fragments should have similar thicknesses and a relatively flat surface at each sampling point.

Analysis Method

Comparison of questioned and known glass via µ-XRF can be performed using spectral comparison and/or elemental ratio comparison. Spectral comparison may consist of screening runs or detailed comparison runs. Spectral comparison consists of comparison of the elements present and their relative peak heights between questioned and known glass. Elemental ratio comparison consists of comparisons of the ratios of the net intensity values of elements between questioned and known glass. Elemental ratio comparison may only be conducted on data from detailed comparison runs.

Elemental ratio data and/or questioned spectra will be evaluated for suitability prior to being used for a comparison.

Spectral comparisons – screening

Screening comparisons may allow glass samples from different sources to be discriminated using short sampling durations based on differences in major elemental content. This may also allow for selection of appropriate fragments for detailed comparisons when many questioned fragments are present. A minimum of 3 replicates from the known glass and a minimum of 2 replicates from each questioned fragment should be collected.
Recommended parameters: at least 100 LSec; 50 kV; 12.8 µs; ~35% Dead time.

Spectral comparisons – detailed comparison runs

When practical, a minimum of 9 replicates from the known glass and a minimum of 3 replicates from each questioned fragment should be collected.
Recommended parameters: at least 600 LSec; 50 kV; 12.8 µs; ~35% Dead time.

Elemental ratio comparison

Any element may be used in a ratio if it is quantifiable (SNR of 10 or more on at least one replicate) in the questioned or known sample. If an element is quantifiable (SNR 10 or more) in one set and not identified (SNR of less than 3) in the other set, this difference can be used to discriminate the glass sets. Possible ratios for evaluation include, but are not limited to: Ca/Mg, Ca/Ti, Ca/Fe, Sr/Zr, Fe/Zr, Ca/K, Na/Mg, Ti/Fe, and Sr/Fe.

- The elements present in the numerator and denominator of the ratio can be inverted if the denominator is a small number.
- Additional ratios may be chosen based on the elements present in the samples.
- Ratios of elements close in atomic number should be used to improve precision.

For an elemental analysis comparison to be considered a match the range of the Questioned glass elemental ratio(s) must overlap with the established range of the corresponding known glass elemental ratio(s).

12.4.4 Direction of Force

Direction of force examinations are conducted to determine from which side of a pane of glass a force was applied and caused a break. Often, this determination will substantiate a witness statement or clarify the chain of events at a crime scene.

All glass samples of reasonable size should be collected and the radial lines marked. It may be necessary to reconstruct a portion of the window to determine the radial and concentric fractures if they have not been marked as such by the investigating officer.

Edge examinations for direction

1. The long axis rib marks of the edge of a radial fracture and the rib marks in the edge of a concentric fracture are used to determine the direction of force.
    a. Radial fractures, the portion of the rib mark which is perpendicular to glass surface will terminate on the side opposite the origin of the force.
    b. Concentric fractures, the portion of the rib mark which is perpendicular to glass surface will terminate on the side of the origin of the force.

    - There is often secondary fracturing of pieces which have fallen to the ground after the incident.

- Caution should be used when evaluating samples which are close to the edge of the frame as they may be affected by the force of the frame.
- Direction of force does not apply to tempered, laminated or glass damaged by heat or explosion

12.5 Interpretation Criteria

Several possible conclusions can be reached after evaluating and comparing glass evidence:

Support for Same Source- The questioned glass was found to be the same as the known glass with respect to color, physical characteristics, refractive index, and/or elemental composition with no significant unexplained differences.

Inconclusive- The questioned glass was found to exhibit both differences and similarities to the known glass with respect to color, physical characteristics, refractive index, and/or elemental composition; to the extent that no conclusion could be reached regarding an association or elimination. The significance of the differences cannot be completely assessed due to the constraints of sample size and/or condition.

Source Exclusion- The questioned glass was found to be different from the known glass with respect to color, physical characteristics, refractive index, and/or elemental composition.

Direction of Force Determination- Identification of the projectile exit point and/or confirmation from radial/concentric fractures:

- The projectile exits via the expanding crater; therefore, the entrance is from the smaller opening.
- With radial fractures, the portion of the rib mark which is perpendicular to glass surface will terminate on the side opposite the origin of the force.
- With concentric fractures, the portion of the rib mark which is perpendicular to glass surface will terminate on the side of the origin of the force.

12.6 References

1. “Precision of GRIM Reference” Canadian Society of Forensic Science, Vol. 27, No. 3 (1994) pp. 203 -208
2. ASTM E 1967 - 98 (re-approved in 2003) Standard Test Method for the Automated Determination of Refractive Index of Glass Samples Using the Oil Immersion Method and a Phase Contrast Microscope.
3. Brewster, "The Retention of Glass Particles on Woven Fabrics", Journal of Forensic Sciences, Vol. 30, pp. 798.
4. Forensic Examination of Glass and Paint: Analysis and Interpretation, Caddy, B. Ed. Taylor& Francis 2001
5. Espinoza, E.O.; Thornton, J.I.; "Three Dimensional Presentation of Glass Density vs. Refractive Index Data."; Journal of Forensic Sciences: 1986, Vol. 31, No. 2, pp 687-690.
6. Hickman, D.A., "Linking Criminals to the Scene of the Crime with Glass Analysis", Analytical Chemistry, Vol. 56, No. 7, 1984, pp. 844-852.
7. Koons, R.D., Buscaglia, J., Bottell, M. Miller, E.T. Forensic Glass Comparisons in Forensic Science Handbook Vol. 1 2nd Ed., R. Safestein, Ed., Prentice-Hall, Englewood Cliffs, NJ, 2002.
8. Locke, J. “GRIM - A Semi-Automated Device for the Measuring the Refractive Index of Glass Particles” The Microscope. Vol. 35 (1987) pp. 151-176
9. McQuillan, J. and Edgar, K., “A Survey of the Distribution of Glass on Clothing”, Journal of Forensic Science Society, Vol. 32, No. 4, 1992, pp. 333-348.
10. Miller, E.T., "Forensic Glass Comparisons", in Forensic Science Handbook, R. Safestein, Ed., Prentice-Hall, Englewood Cliffs, NJ, 1982.

11. Newton, A. and Buckleton, J., “An investigation into the relationship between edge counts and the variability of the refractive index of glass Part I: Edge morphology,” Forensic Science International, Volume 177, Issue 1, pp. 24-31
12. Pearson, E.F., May, R.W. and Dabbs, M.D.G., "Glass and Paint Fragments Found in Men's Outer Clothing-Report of a Survey", Journal of Forensic Sciences, Vol. 16, No. 3, 1971, pp. 283-300.
13. Slater, D. P.; Fong, W.; "Density, Refractive Index and Dispersion in the Examination of Glass: Their Relative Worth as Proof."; Journal of Forensic Sciences: 1982, Vol. 32, pp 474-483.
14. Thornton, J.I.; Langhauser, C.; Kahane, D.; "Correlation of Glass Density and Refractive Index-Implications to Density Gradient Construction", Journal of Forensic Sciences: 1984, Vol. 29, pp 711-713.
15. Walsh, K. and Buckleton, J., "On the Problem of Assessing the Evidential Value of Glass Fragments Embedded in Footwear", Journal of Forensic Science Society, 26, 1986, pp. 55-60.
16. Bottrell, M.; “Forensic Glass Comparison: Background Information Used in Data Interpretation”, Forensic Science Communications, Vol. 11 (2009), Number 2.
17. Garvin, E.J. and Koons, R.D.; “Evaluation of Match Criteria Used For The Comparison of Refractive Index of Glass Fragments”; Journal of Forensic Sciences: 2011, Vol. 56, No. 2, pp 491-500.
18. ASTM E 2926 - 13 Standard Test Method for Forensic Comparison of Glass Using Micro X-ray Fluorescence (µ-XRF) Spectrometry.
19. Ernst, T., et al. Signal-to-Noise Ratios in Forensic Glass Analysis by Micro X-ray Fluorescence Spectrometry, X-Ray Spectrometry, 2014, Vol. 43, pp 13-21
20. Almirall, J., et al. “Significance of Elemental Analysis from Trace Evidence,” 2012, U.S. Department of Justice Award Number 2009-DN-BX-K252, Document Number 242325
21. Trejos, T., et al. “Cross-validation and evaluation of the performance of methods for the elemental analysis of forensic glass by µ-XRF, ICP-MS, and LA-ICP-MS,” Analytical and Bioanalytical Chemistry, DOI 10.1007/s00216-013-6978-y (early view)
22. Trejos, T., et al. “Forensic analysis of glass by µ-XRF, SN-ICP-MS, LA-ICP-MS and LA-ICP-OES: evaluation of the performance of different criteria for comparing elemental composition,” Journal of Analytical Atomic Spectrometry, 2013, Vol. 28, pp 1270-1282
23. Ryland, S. “Discrimination of Flat (Sheet) Glass Specimens Having Similar Refractive Indices Using Micro X-Ray Fluorescence Spectrometry,” JASTEE, 2011, Vol. 2, pp 2-12
24. Hicks, T., et al. “The classification and discrimination of glass fragments using non destructive energy dispersive X-ray µfluorescence, Forensic Science International, 137 (2003) pp 107-118.
25. Koons, R, et al. “The Forensic Significance of Glass Composition and Refractive Index Measurements,” Journal of Forensic Sciences, 1999, Vol.44, No.3, pp 496-503
26. Ryland, S. “Sheet or Container? - Forensic Glass Comparisons with an Emphasis on Source Classification,” Journal of Forensic Sciences, JFSCA, 1986, Vol. 31, No. 4, pp 1314-1329
27. Keeley, R. H. and Christofides, S., “Classification of small glass fragments by x-ray microanalysis with the SEM and a small XRF spectrometer,” Scanning Electron Microsc., 1979, I, pp 459-164
28. Hickman, D.A., “A classification scheme for glass,” Forensic Sci. Int., 1981, 17, pp 265-281
29. Hickman, D.A., “Elemental analysis and the discrimination of sheet glass samples,” Forensic Sci. Int., 1983, 23, pp 213-223,
30. Hickman, D.A., “Glass types identified by chemical analysis,” Forensic Sci. Int., 1987, 33, pp 23-46

31. Howden, C.R., Dudley, R. J., and Smalldon, K. W., "The Analysis of Small Glass Fragments Using Energy Dispersive X-ray Fluorescence Spectrometry," Journal of the Forensic Science Society, 1978, Vol. 18, pp. 99-112.
32. Curry, A. S. edit., "Methods of Forensic Science", Vol. IV, Interscience 1965
33. Kirk, Paul, "Crime Investigation", Interscience 1953
34. McJunkins, S. P. and Thornton, "Glass Fracture Analysis, A Review", Forensic Science Vol. 2, 1973 pp1-27.
35. O'Hara, C. and Osterberg, "James An Introduction to Criminalistics", Indiana (reprint from Macmillian Co. 1949), 1972.
36. Rhodes, E. F. and Thornton, J. I., "The Interpretation of Impact Fractures in Glassy Polymers", Journal of Forensic Science, Vol. 20, #2 Apr. 1975.
37. Ryland, Scott. "Discrimination of Flat (Sheet) Glass Specimens Having Similar Refractive Indices Using Micro X-Ray Fluorescence Spectrometry". JASTEE, Vol. 2, Issue 1. Ryland: Discrimination of Flat Glass p.1-11.
38. Tatiana Trejos, Robert Koons, Peter Weis, et. Al. "Forensic analysis of glass by u-XRF, SN-ICP-MS, LA-ICP-MA and LA-ICP-OES: evaluation of the performance of different criteria for comparing elemental composition". Journal of Anal. At. Spectrom., 2013.
39. T. Hicks, F. Monard Sermier, T. Goldmann, A. Brunelle, C. Champod, P. Margot. "The classification and discrimination of glass fragments using non destructive energy dispersive X-ray µfluroresсence". Forensic Science International 137 (2003) 107-118.

13 Vehicle Lamp Examination

13.1 Introduction

Many automobile lamps are incandescent electric lamps in which an electric current is sent across a thin tungsten wire. When the current is great enough, the wire will heat to an incandescent temperature and produce light. The following physical characteristics are the basis for any vehicle lamp condition determination:

- Tungsten oxidizes rapidly at incandescent temperatures; therefore, lamp filaments must be enclosed in an oxygen free glass bulb.
- Tungsten is ductile at incandescent temperatures and brittle when cold.
- Temperature of an incandescent tungsten filament is greater than the melting point of glass.

With proper evidence collection and preservation, and the knowledge of fundamental physical characteristics of tungsten filament lamps, the on/off condition of a lamp which sustained damage at the time of impact may be determined.

13.2 Safety Considerations

Protective eyewear and gloves should be worn.

13.3 Equipment/Instrumentation

- Stereomicroscope with digital camera
- Voltmeter
- Propane torch
- Hacksaw
- Screwdriver
- Wet towel
- Tape
- Diamond tip scribe

13.4 Procedure

13.4.1 Physical Characteristics Examination

Conduct both a visual and microscopic examination of the lamps submitted. When possible, observations should be photographed for documentation as part of the examination notes. The glass envelope, base, filaments and filament supports should be examined and the condition of each component noted. Use of an approved lamp examination form is recommended. If the filament connections appear intact, resistance across the circuit should be tested with a voltmeter to determine if the lamp is operational.

The following criteria indicate that a filament was incandescent:

- filament is blackened
- white oxide is noted on glass or supports
- fused glass is observed on a filament
- a filament support is burned or melted
- the glass is etched
- filament is stretched out or uncoiled

The following criteria indicate that a filament was not incandescent:

- separated filament exhibits melted, tapered or balled ends
- separated filament exhibits brittle, uneven ends

The above criteria are only some examples of criteria for determination of incandescence; refer to the cited materials in this section for more complete criteria.

13.4.2 Interpretation Criteria

Conclusions that can be reached after evaluating and comparing lamp filaments:

Incandescent- Physical characteristics observed on the lamp components indicate that the lamp was incandescent at the time of an impact

Not incandescent- Physical characteristics observed on the lamp components indicate that the lamp was previously burned out or otherwise not incandescent at the time of an impact

Inconclusive- The lamp failed to reveal sufficient damage to permit an on/off determination

13.5 References

1. Baker, J.S.; Aycock, T.L.; Lindquist, T.; “Lamp Examination for On or Off in Traffic Accidents.”; Northwestern University Traffic Institute: 1985.
2. Bradford; “Lamp Filaments (A Method for Determining Whether Lights Were On or Off).”; Unpublished manuscript: 1964.
3. Dolan; “Vehicle Lights and Their Use as Evidence.”; Journal of Forensic Sciences: 1971, Vol. 2, No. 2, pp. 69-82.
4. Powell, G.L.F.; “Interpretation of Vehicle Globe Failures: The Unit Condition.”; Journal of Forensic Sciences: 1977.
5. Woods; Bonnet; “Headlamps are Tools in Traffic Accident Investigation.”; Law and Order: June 1977.

14 Pressure Sensitive Tape Analysis

14.1 Introduction

The evaluation of tapes for class characteristics (such as design, construction and composition) can associate known and questioned tapes to a group but not to a single, individual source. A physical end match of two tape ends can determine that the pieces were at one time a single item.

Methods for the analysis of tape include examinations of physical characteristics, polarized light microscopy (PLM), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS).

14.2 Minimum Standards and Controls

Physical end matches between a question specimen and a known specimen must be verified by another qualified analyst.

14.3 Equipment/Instrumentation

- Hexane, xylene-substitute or other suitable solvent
- Tweezers, scalpels, probes
- Microscope mounting supplies
- Stereomicroscope
- Sonicator
- Polarized light microscope
- Comparison microscope
- SEM/EDS
- XRF
- FTIR

14.4 Sample Preparation

It may be desirable to obtain a sample cutting from the tape before a sample is analyzed by other laboratory disciplines. Samples for testing should not be cut from the ends of the tape if there is a possibility of a physical end match between specimens.

If tape is received in a tangled condition, an attempt should be made to separate it manually with a careful peel. More aggressive techniques such as gentle heat, liquid nitrogen, freezing, or solvents can be used if necessary. However, these techniques could affect the outcome of subsequent analyses and should, therefore, be applied only to the extent necessary.

14.5 Analysis Methods

14.5.1 Physical Examination

The physical characteristics are determined by the type of tape being examined but may include:

- Overall tape color
- Color of adhesive and backing
- Scrim description

- Surface texture
- Physical measurements

When conducting comparison examinations between two or more tape specimens, the free ends should be carefully examined for possible physical end matches. If the tape has a fabric reinforcement layer, solvent (e.g., hexane, chloroform, or xylene) may be used to remove a sufficient amount of adhesive to expose the fabric and ensure alignment of the yarns that have broken across the torn ends.

The analyst may elect to continue with a complete analytical analysis of these specimens depending upon the quality of the end match.

14.5.2 Microscopic Examination

There is variability in tape films, adhesives, and fibers that can be readily noted with transmitted and polarized light. Some tapes may exhibit microscopic variability that cannot be readily detected in other instrumental or macroscopic examinations.

The following microscopic details may be included in the comparison:

1. Determination of polypropylene film orientation
2. Determination of the extinction angle relative to the machine direction
3. Determination of the retardation
4. Thickness

14.5.3 FTIR Analysis

The following components, if present, may be characterized by IR spectroscopy.

- Backing
- Polymer film
- Plasticizers
- Fillers/Extenders
- Adhesive
- Release coating
- Fiber reinforcement

14.5.4 SEM/EDS and XRF Analysis

The geometry of each sample, including flatness and take-off angle, should be similar. Often, a backing can be pressed flat with clean glass in order to remove irregularities.

14.6 Interpretation Criteria

Several possible conclusions can be reached after evaluating and comparing pressure sensitive tape:

Source Identification- The questioned tape edge was compared to a known tape edge or an area missing from the known tape and constitutes a matching individual break configuration (See Physical Break Match/Tear Configuration Comparison section in this manual).

Support for Same Source–The questioned tape was found to be the same as the known tape with respect to physical characteristics, chemical composition, and/or elemental composition with no significant unexplained differences.

Inconclusive- The questioned tape was found to exhibit both differences and similarities to the known tape with respect to physical characteristics, chemical composition, and/or elemental composition to the extent that no conclusion could be reached regarding an association or elimination. The significance of the differences cannot be completely assessed due to the constraints of sample size and/or condition.

Source Exclusion- The questioned tape was found to be different from the known tape with respect to physical characteristics, chemical composition, and/or elemental composition.

14.7 References

1. SWGMAT. “Guideline for Forensic Examination of Pressure Sensitive Tapes.” Web. 03 Feb. 2012.
2. SWGMAT. “Guideline for Assessing Physical Characteristics in Forensic Tape Examinations.” Web. 03 Feb. 2012.
3. SWGMAT. “Guideline for Using Light Microscopy in Forensic Examinations of Tape Components.” Web. 03 Feb. 2012.
4. SWGMAT. “Guideline for Using Fourier Transform Infrared Spectroscopy in Forensic Tape Examinations.” Web. 03 Feb. 2012.
5. SWGMAT. “Guideline for Using Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy in Forensic Tape Examinations.” Web. 03 Feb. 2012.
6. Smith J. (1998). The forensic value of duct tape comparisons. Midwestern Association of Forensic Scientists Newsletter 27(1), 28-33.
7. Maynard P, et al.(2001). Adhesive tape analysis: establishing the evidential value of specific techniques. Journal of Forensic Science, 46(2): 280-7.
8. Bradley, M.J., Keagy, R.L., Lowe, P.C., Rickenbach, M.P., Wright, D.M., and LeBeau, M.A. (2006). A validation study for duct tape end matches. Journal of Forensic Science, 51 (3), 504–508.
9. Cockrell, R.P. (1982). Physical matching- fitting the pieces together. RCMP Gazette, 44(4), 17-21.
10. Thornton, J.I. (1986). Fractal surfaces and models of physical matches. Journal of Forensic Science 31(4), 1435-1438.
11. McCrone, W.C., McCrone L.B, and Delly J.G. (1978). Polarized light microscopy. Ann Arbor, MI: Ann Arbor Science Publishers.
12. Rappe, R.G. (1987). Measurement of the principal refractive indices of oriented polymer films. Microscope 35, 67-82.
13. Smith, J., Weaver, R.(2004). PLM examinations of clear polymer films. Microscope Vol 53:3/4, 112-118.
14. Kilbourn, J.H. and Marx, R. B. (1994). Polarized Light Microscopy of Extenders in Structural paints - Forensic Applications. Microscope vol 42:4, 167-175.
15. Merrill R.A, Bartick E.G. (2000). Analysis of pressure sensitive adhesive tape: I. evaluation of infrared ATR accessory advances. Journal of Forensic Sciences. 45(1), pp. 93-98.
16. Coates J., Reffner J.(1999). Visualization of micro-ATR infrared spectroscopy. Spectroscopy 14(4), 34-45.
17. Postek, M.T., Howard, K.S., Johnson, A.H., and McMichael, K.L. (1980). Scanning electron microscopy: a student’s handbook. Williston, VT: Ladd Research Industries, Inc.
18. Ward, D. C. A small sample mounting technique for scanning electron microscopy and X-ray analysis, Forensic Science Communications [Online]. (July 1999). Available: www.fbi.gov/hq/lab/fsc/backissu/july1999/ward.htm

15 Miscellaneous Examinations

Particles of safe insulation can be found on tools or clothing, in vehicles used to transport safes, and in locations where safes have been damaged. Known safe insulation should be collected from the door and walls of the safe in question. A safe insulation reference collection should be kept at each laboratory performing this type of examination. Due to bulk manufacturing, it is not possible to state that a certain building material came from a specific building to the exclusion of all others; however, the presence of a few particles of a single building material may have definite probative value when taken within context of other evidence gathered by the investigating officer.

15.1.1 Equipment/Instrumentation

- Stereomicroscope
- Comparison microscope
- Polarized light microscope
- SEM/EDS
- XRF
- FTIR
- Safe insulation reference material

15.1.2 Analysis Methods

Visual and microscopic examinations are conducted on the Items in order to locate possible questioned samples. Questioned samples are then compared to known samples with regard to their physical, optical, and chemical properties.

Typical safe insulation materials can include:

1. Vermiculite
2. Diatoms
3. Sawdust or wood chips
4. Fiberglass
5. Air bubbles
6. Matrix materials can include:
7. Portland cement
8. Natural cement (many particulate impurities)
9. Frothy cement (filled with air bubbles)
10. Gypsum

15.2 Interpretation Criteria

Several possible conclusions can be reached after evaluating and comparing questioned and known samples:

Source Identification- The questioned sample's edge was compared to a known's edge or an area missing from the known and constitutes a matching individual break configuration (See Physical Break Match/Tear Configuration Comparison section in this manual).

Support for Same Source - The questioned sample was found to be the same as the known sample with respect to physical characteristics, chemical composition, and/or elemental composition with no significant unexplained differences.

Inconclusive - The questioned sample was found to exhibit both differences and similarities to the known sample with respect to physical characteristics, chemical composition, and/or elemental composition to the extent that no

conclusion could be reached regarding an association or elimination. The significance of the differences cannot be completely assessed due to the constraints of sample size and/or condition.

Source Exclusion- The questioned sample was found to be different from the known sample with respect to physical characteristics, chemical composition, and/or elemental composition.

15.2.1 References

1. McCrone, W. C., and Delly, J. G., “The Particle Atlas”, Ann Arbor Science; Ann Arbor, MI, 1974.
2. McCrone, W. C., and Delly, J. G., “Polarized Microscopy”, Ann Arbor Science; Ann Arbor, MI, 1978.
3. Brady, George S.; Clanser, H. R., “Materials Handbook”, 12th ed.; McGraw-Hill: New York, 1986.
4. FBI Law Enforcement Bulletin, February 1973.
5. United States Gypsum “Gypsum Construction Handbook”, U. S. Gypsum: Chicago, IL, 1982.

16 Suggested Report Wording

Laboratory reports are generated in accordance with the accreditation requirements. Reports may include a Conclusions Table, a Remarks section, an Analytical Detail section or a narrative that ensures compliance to the Laboratory Quality Assurance manual policies.

When conclusions are made, the results must be clearly communicated. When no definitive conclusions can be reached, the reason(s) must be clearly communicated. Examples of situations where inconclusive results may be encountered include, but are not limited to:

- Insufficient sample size
- Insufficient detail/registration
- Similarities with minor unexplained differences

Comparative conclusions must be properly qualified. Significance of the conclusion will be expressed on the following basis:

1	Source Identification	The observations provide extremely strong support for the proposition that the evidence originated from the same source and the likelihood for the proposition that the evidence arose from a different source is so remote as to be considered a practical impossibility.
2	Support for Same Source	The observations provide more support for the proposition that the evidence originated from the same source rather than different sources; however, there is insufficient support for a Source Identification. The degree of support may range from limited to strong or similar descriptors of the degree of support. Any use of this conclusion shall include a statement of the factor(s) limiting a stronger conclusion.
3	Inconclusive	The observations do not provide a sufficient degree of support for one proposition over the other. Any use of this conclusion shall include a statement of the factor(s) limiting a stronger conclusion.
4	Support for Different Source	The observations provide more support for the proposition that the evidence originated from different sources rather than the same source; however, there is insufficient support for a Source Exclusion. The degree of support may range from limited to strong or similar descriptors of the degree of support. Any use of this conclusion shall include a statement of the factor(s) limiting a stronger conclusion.
5	Source Exclusion	The observations provide extremely strong support for the proposition that the evidence originated from a different source and the likelihood for the proposition that the evidence arose from the same source is so remote as to be considered a practical impossibility; or the evidence exhibits fundamentally different characteristics

16.1 Method of Testing

Method	Suggested Report Wording
Visual Examination	Visual examination was performed on Item #_____.
Microscopical Examination	Microscopical examination was performed on Item#_____.

SEM/EDS	Scanning electron microscopy/energy dispersive x-ray (SEM/EDS) spectroscopy analysis was performed on Item#_____ and revealed it to be composed of_____.
FTIR	Fourier transform infrared (FTIR) spectroscopy analysis was performed on Item #_____.

rIQ	Refractive index quantification (rIQ) analysis was performed on Item #_____.
Micro XRF	Micro X-ray fluorescence (micro XRF) spectrometry analysis was performed on Item # _____.

16.2 Findings

Finding	Suggested Report Wording
Pb-Ba-Sb identified – analysis terminated prior to all samples completed	Particles characteristic of gunshot primer residue were identified on one of the samples from _____. Analysis was not completed on the remaining sample(s).
Pb-Ba-Sb identified– all samples completed	Particles characteristic of gunshot primer residue were identified on the samples from_____.
Pb-Ba-Sb identified– one particle	A particle characteristic of gunshot primer residue was identified on the samples from_____.
No Pb-Ba-Sb found	Particles characteristic of gunshot residue were not identified on the samples from_____.
Footwear database Entry	An image of the questioned impression has been entered into the footwear database and may be compared to impressions and/or footwear submitted in the future.
No Footwear database Entry	Examination of Item # revealed one possible questioned footwear impression which is not suitable for entry into the footwear database due to poor tread registration and/or clarity.
Footwear database-- all tread components visible are consistent under the same orientation/position	Examination of Item #__ revealed the presence of__ partial footwear impressions which appear to be of the same tread design. A search of the Laboratory's footwear database revealed a corresponding tread design on known "_____" shoes, however other possibilities may exist. Copies are attached for your reference. If known footwear is submitted for comparison, please resubmit Item # at that time.
Footwear database results- multiple candidates returned	A search of the Laboratory's footwear database revealed numerous tread designs that could not be eliminated as potential sources. If known footwear is submitted for comparison, please resubmit Item #__ at that time.
Footwear database results- no matches	A search of the Laboratory's footwear database failed to reveal a corresponding known

	tread design. The footwear databases are regularly updated with the addition of new exemplars. Additional searches may be performed upon request.
Footwear database results- no matches, but unique tread design/logo	A search of the Laboratory's footwear database failed to reveal a corresponding known tread design. However, the tread elements/logo in the questioned impression is typically found on "Nike" brand shoes.
Footwear database – retention for violent crime	An image of the questioned impression has been entered into the footwear database and will be searched against each of the next three quarterly updates of the database, or until a corresponding known tread design is identified, whichever occurs first.
Source Identification- physical break match or tear/cut configuration	Microscopic/Visual comparisons revealed that Items_____exhibit matching physical break /tear (or cut) configurations. Based on the above findings, at one time the items formed a single object.
Source Identification- impressions match via class and individual characteristics	The questioned impression was found to be the same as the known left shoe/front passenger tire with respect to_____(tread size, tread design, wear characteristics and individual characteristics). These findings confirm that this questioned impression was made exclusively by the known left shoe/front passenger tire.
Support for Same Source – impressions match via class characteristics	The questioned impression was found to be the same as the known left shoe/front passenger tire with respect to_____(tread size, tread design, wear characteristics and some individual characteristics)-
Inconclusive – impressions	The questioned impression exhibited both differences and similarities in tread size and tread design. Based on these findings no conclusion could be reached regarding an association or elimination.
Source Exclusion – impressions	The questioned impression was found to be distinctly different from the known left shoe/front passenger tire with respect to (tread size, tread design, wear characteristics and / or some individual characteristics) and could not have come from the same source
Support for Same Source	The questioned _____was found to be the same as the known _____ in physical / microscopic / microchemical characteristics / chemical compositions / elemental composition and no significant unexplained differences are noted

	confirming that the questioned _____could have originated from the same source of the known _____but not exclusively

Inconclusive	The questioned _____ was found to exhibit both similarities and differences to the known _____ . The significance of the differences cannot be completely assessed due to the constraints of sample size and/or condition.
Source Exclusion– known and unknown exhibit significant differences	Analysis of the submitted known and unknown samples revealed distinct differences in physical structure, chemical composition or elemental composition. Based on the above findings, these samples could not have come from the same source.
Vehicle Lamp – on (incandescent)	The large filament (turn signal/brake light) is intact and moderately stretched/distorted. This filament is evenly blackened and has melted glass fragments fused to it. The support posts have a white deposit on them. Based on these findings, this filament was on (incandescent) at the time, or immediately before, the lamp was subjected to an impact and the glass envelope was broken.
Vehicle Lamp – off (cold)	The small filament (taillight) is intact with uniformly spaced coils. This filament is evenly covered with a white deposit and shows mild possible deformation. The support posts have a white deposit on them. Based on these findings, this filament was off (cold) at the time the lamp was subjected to an impact and the glass envelope was broken.
Vehicle Lamp - inconclusive	Examination of the right front lamp from vehicle #2 (Item #_) revealed the glass envelope is intact and clear; and the filaments are bright, intact and have a normal appearance. Based on these findings, this lamp is operational, however, it cannot be determined if this lamp was on or off at the time of impact.
Inconclusive- Vehicle Lamp On/Off determination	The lamp lacked sufficient characteristics to make an on/off determination.

16.3 Conclusion Table

Items- List all questioned items received. The known item is referenced within the “Findings” column.

Description- Provide an item description. For example: digital images, piece of glass, pry bar, Q paint chips, etc.

Findings- Provide the examination finding and the results of any comparisons to known items submitted (e.g. "Two partial questioned footwear impressions (Q1 & Q4)"; "Same Tread size and design as the known right shoe (Item #2)"

Conclusions- Provide the conclusion of all testing performed on evidence.

16.3.1 Qualifying Statements

Circumstance	Suggested Qualifying Statement
Source Identification	**Qualifying Statement:** The likelihood that the evidence arose from a different source is so remote as to be considered a practical impossibility.
Support for Same Source	**Strong Qualifying Statement:** This association is not exclusive; other manufactured items with the same characteristics may exist. While they could not be conclusively identified to the same source, the items were found to exhibit unusual matching characteristics that would not be expected to be found in the population of this evidence type.
	Normal Qualifying Statement: This association is not exclusive; other manufactured items with the same characteristics may exist.
	Weak Qualifying Statement: This association is not exclusive; other manufactured items with the same characteristics may exist. Due to significant limiting factors, this association has decreased evidential value.
Inconclusive	**Qualifying Statement:** The items exhibit both differences and similarities in observed and/or measured properties and/or chemical composition to the extent that no conclusion could be reached regarding an association or elimination.
	Lamp Qualifying Statement: The lamp lacked sufficient characteristics to make an on/off determination.
Source Exclusion	The evidence exhibits fundamentally different characteristics than the known reference and could not have come from the same source

Pb-Ba-Sb identified – hands	Particles classified as characteristic of gunshot primer residue have compositions rarely found in particles from any other source. A finding of "particles characteristic of gunshot residue were identified" on a person's hands means that individual either discharged a firearm, was in the vicinity of a firearm when it was discharged, or handled an item with gunshot primer residue on it.
Pb-Ba-Sb identified– item	A finding of "characteristic gunshot primer residue particles" on an item means that the item, at some time in its history, was in the vicinity of a firearm when it was discharged or came in contact with another item with gunshot residue on it. However, this does not eliminate the possibility of other non-firearm related sources of exposure.
Pb-Ba, Ba-Sb or Pb-Sb identified – hands or item	Lead barium, barium antimony and/or lead antimony particles are consistent with gunshot primer residue; however, particles with these compositions can be produced from non-firearm sources.
No Pb-Ba-Sb- hands	Particles classified as characteristic of gunshot primer residue have compositions rarely found in particles from any other source. A finding of "particles characteristic of gunshot residue were not identified" does not preclude the possibility of the individual having discharged a firearm, having been in the vicinity when a firearm was discharged, or having handled an item with gunshot primer residue on it.
No Pb-Ba-Sb- item	Particles classified as characteristic of gunshot primer residue have compositions rarely found in particles from any other source. A finding of "particles characteristic of gunshot residue were not identified" does not preclude the possibility having been in the vicinity when a firearm was discharged or having handled an item with gunshot primer residue on it.

16.4 Miscellaneous

Circumstance	**Suggested Report Wording**
Returned Evidence	The evidence is being returned to your department for retention.
Returned Evidence	All evidence (items) is/are being returned.
Evidence created during examination	_ was / were created during examination of Item # .

Photographs created	The impressions(s) in Item X were photographed and these images are retained at the laboratory. The remaining evidence (items) are being returned. -or- Digital images are being retained at BCI.
Item not examined	_____ was / were not examined.
Footwear database retention for violent crime	An image of the questioned impression has been entered into the footwear database and will be searched against each of the next three quarterly updates of the database, or until a corresponding known tread design is identified, whichever occurs first.
GSR evidence analyzed in Richfield/report written in Bowling Green	The GSR instrumental analysis of the above evidence was performed in the Richfield laboratory; the interpretation of the data was performed in the Bowling Green laboratory.

,

16.5 Report Content Examples

16.5.1 Example 1 Physical Break Match/Tear Configuration Comparisons

To:	ACME Police Department Det. Bunny Main Street Road	BCI Laboratory Number:	xx-xxxxx
		Analysis Date: July 14, 2019	Issue Date: July 15, 2019
		Agency Case Number:	xxxxx
Offense:	Homicide		
Subject(s):	John Doe		
Victim(s):	State of Ohio		

Submitted on May 24, 2019 by Steve:
1. Known tail light assembly
2. Questioned piece of red plastic

Item	Description	Finding	Conclusion
#2	Piece of red plastic	Matching individual break configuration to Item #1	Source Identification [1]

[1] The likelihood that the evidence arose from a different source is so remote as to be considered a practical impossibility.

Remarks
The evidence is being returned to your department. Digital images are being retained at BCI.

Analytical Detail
These findings were determined using visual examination techniques.

16.5.2 Example 2 Impression Comparison

To:	ACME Police Department Det. Bunny Main Street Road	BCI Laboratory Number:	xx-xxxxx
		Analysis Date: July 14, 2019	Issue Date: July 15, 2019
		Agency Case Number:	xxxxx
Offense:	Homicide		
Subject(s):	John Doe		
Victim(s):	State of Ohio		

Submitted on May 24, 2019 by Steve:

1. Known shoes from Yosemite Sam
2. Known shoes from Elmer Fudd
3. Cast of questioned impression
4. CD with digital images
5. CD with digital images
6. Cast of questioned impression
7. Cast of questioned impression
8. Cardboard box with questioned footwear impression

Item	Description	Finding	Conclusion
#3	Cast	One questioned footwear impression Same tread design, tread size, wear characteristics, and matching randomly acquired characteristics as Item #1	Source Identification [1]
#4	Digital Images	One questioned footwear impression Same tread design, tread size, wear characteristics, and some matching randomly acquired characteristics as Item #2	Support for same source [2, 3]
		One questioned footwear impression Same tread design, tread size, and wear characteristics as Item #2	Support for same source [2]
#5	Digital Images	One questioned footwear impression Same tread design to Item #2	Support for same source [2, 4]
#6	Cast	One questioned footwear impression Some matching and some non-matching tread design as Item #2	Inconclusive [5]
#7	Cast	One questioned footwear impression Different tread design from Items #1 & #2	Source Exclusion [6]
			Database association to “Nike” brand shoes
#8	Cardboard Box	One questioned footwear impression Different tread design from Items #1 & #2	Source Exclusion [6]

			No database match; however, the logo is typically found on "Nike" brand shoes

[1] The likelihood that the evidence arose from a different source is so remote as to be considered a practical impossibility.
[2] This association is not exclusive; other manufactured items with the same characteristics may exist.
[3] While they could not be conclusively identified to the same source, the items were found to exhibit unusual matching characteristics that would not be expected to be found in the population of this evidence type.
[4] Due to significant limiting factors, this association has decreased evidential value.
[5] The items exhibit both differences and similarities in observed and/or measured properties to the extent that no conclusion could be reached regarding an association or elimination.
[6] The evidence exhibits fundamentally different characteristics than the known reference and could not have come from the same source

Remarks
Due to the lack of a scale, no size comparison could be performed for Item #5.

Due to subsequent damage that was reported to Item #6, no conclusive determination could be made.

A search of the Laboratory's footwear database for Item #7 revealed a corresponding tread design on known "Nike" brand shoes. Do not limit the search for known footwear to only an exact match of these shoes, as other possibilities may exist.

A search of the Laboratory's footwear database for Item #8 failed to reveal a corresponding known tread design. The footwear database is regularly updated with the addition of new exemplars. Additional searches may be performed upon request.

The evidence is being returned to your department. Digital images are being retained at BCI.

Analytical Detail
These findings were determined using visual and overlay examination techniques.

Examination results of Item #6 could have been affected by evidence handling prior to submission.

16.5.3 Example 3 Fiber and Fabric Analysis

To:	ACME Police Department Det. Bunny Main Street Road	BCI Laboratory Number:	xx-xxxxx
		Analysis Date: July 14, 2019	Issue Date: July 15, 2019
		Agency Case Number:	xxxxx
Offense:	Homicide		
Subject(s):	John Doe		
Victim(s):	State of Ohio		

Submitted on May 24, 2019 by Steve:

1. Known flannel shirt from Yosemite Sam
2. Known plaid pants from Elmer Fudd
3. Known white t-shirt
4. Questioned piece of fabric
5. Questioned piece of fabric
6. Questioned fibers
7. Questioned fibers
8. Partially burned questioned fibers
9. Questioned fibers

Item	Description	Finding	Conclusion
#4	Questioned piece of fabric	Matching individual break configuration to Item #1	Source Identification [1]
#5	Questioned piece of fabric	Same color, weave, microscopical characteristics, optical properties, and chemical composition as Item #2 The questioned fabric also exhibits a similar size and shape to a damaged area on Item #2	Support for same source [2, 3]
#6	Questioned fibers	Same color, microscopical characteristics, optical properties, and chemical composition as Item #2	Support for same source [2]
		Different chemical composition from Items #1	Source Exclusion [6]
#7	Questioned fibers	Same color, microscopical characteristics, and optical properties, as Item #3	Support for same source [2, 4]
		Different chemical composition from Items #1 & #2	Source Exclusion [6]
#8	Burned questioned fibers	Similar color and chemical composition to Item #2	Inconclusive [5]
		Different chemical composition from Items #1 & #3	Source Exclusion [6]
#9	Questioned fibers	Different chemical composition from Items #1 #2 & #3	Source Exclusion [6]

[1] The likelihood that the evidence arose from a different source is so remote as to be considered a practical impossibility.
[2] This association is not exclusive; other manufactured items with the same characteristics may exist.
[3] While they could not be conclusively identified to the same source, the items were found to exhibit unusual matching characteristics that would not be expected to be found in the population of this evidence type.
[4] Due to significant limiting factors, this association has decreased evidential value.
[5] The items exhibit both differences and similarities in observed and/or measured properties to the extent that no conclusion could be reached regarding an association or elimination.
[6] The evidence exhibits fundamentally different characteristics than the known reference and could not have come from the same source

Remarks

Due to the prevalence of white cotton fibers in the environment the type of fibers found in Item #7 lack value for a comparison.

The fibers from Item #8 exhibit similarities and differences from the known fibers in Item #2. This may be due to the sample having been partially burned.

The evidence is being returned to your department. Digital images are being retained at BCI.

Analytical Detail

These findings were determined using visual examination techniques, microscopical examination techniques (stereo microscope, PLM, comparison microscopes) and instrumental analyses (FTIR).

16.5.4 Example 4 Paint Analysis

To:	ACME Police Department Det. Bunny Main Street Road	BCI Laboratory Number:	xx-xxxxx
		Analysis Date: July 14, 2019	Issue Date: July 15, 2019
		Agency Case Number:	xxxxx
Offense:	Homicide		
Subject(s):	John Doe		
Victim(s):	State of Ohio		

Submitted on May 24, 2019 by Steve:

1. Front car bumper of victim's car
   -Multilayer paint: red metallic basecoat, and clearcoat
2. Known Paint from front driver's side fender of victim's car
   -Multilayer paint: dark grey primer, light grey primer, red metallic basecoat, and clearcoat
3. Questioned paint chip
   -Multilayer paint: dark grey layer, light grey layer, red metallic layer, and clearcoat layer
4. Questioned paint chip
   -Multilayer paint: dark grey layer, light grey layer, red metallic layer, and clearcoat layer
5. Questioned paint chip
   -Multilayer paint: dark grey layer, light grey layer, red metallic layer, and clearcoat layer
6. Questioned paint chip
   -Clearcoat paint chip
7. Questioned paint smear from concrete divider
   -Red paint smear
8. Questioned paint chip
   -Green paint chip

Item	Description	Finding	Conclusion
#3	Questioned paint chip	Matching individual break configuration to damaged area on Item #1	Source Identification [1]
#4	Questioned paint chip	Same color, layer structure, microscopical characteristics, chemical composition, and elemental composition as Item #1 The questioned chip also exhibits a similar size and shape to a damaged area on Item #1	Support for same source [2, 3]
#5	Questioned paint chip	Same color, layer structure, microscopical characteristics, chemical composition, and elemental composition as Item #2	Support for same source [2]
#6	Questioned paint chip	Same color and microscopical characteristics, as known clearcoat Item #2	Support for same source [2, 4]
#7	Questioned paint smear	Similar color and chemical composition to red basecoat Item #2	Inconclusive [5]
#8	Questioned paint chip	Different chemical composition from Items #1 & #2	Source Exclusion [5]

[1] The likelihood that the evidence arose from a different source is so remote as to be considered a practical impossibility.
[2] This association is not exclusive; other manufactured items with the same characteristics may exist.

[3] While they could not be conclusively identified to the same source, the items were found to exhibit unusual matching characteristics that would not be expected to be found in the population of this evidence type.
[4] Due to significant limiting factors, this association has decreased evidential value.
[5] The items exhibit both differences and similarities in observed and/or measured properties and/or chemical composition to the extent that no conclusion could be reached regarding an association or elimination.
[6] The evidence exhibits fundamentally different characteristics than the known reference and could not have come from the same source

Remarks

Due to the size of the questioned paint chip (Item #6) no instrumental analysis was possible.

Due to the condition of the questioned paint chip (Item #7) it was not possible to isolate a sample free of foreign material for analysis.

The evidence is being returned to your department. Digital images are being retained at BCI.

Analytical Detail

These findings were determined using visual examination techniques, microscopical examination techniques (stereo microscope, PLM, comparison microscopes) and instrumental analyses (FTIR, micro-XRF).

16.5.5 Example 5 GSR

17 To: Police Department
Det.
Road

BCI Laboratory Number: xx-xxxxx

Analysis Date: July 14, 2019

Issue Date: July 15, 2019

Agency Case Number: xxxxx

Offense: Weapons under disability
Subject(s): John Doe
Victim(s): State of Ohio

Submitted on May 24, 2019 by Steve:

1. Brown paper Bag containing GSR kit from Yosemite Sam
2. Brown paper Bag containing GSR kit from Elmer Fudd
3. Brown paper Bag containing GSR kit from Daffy Duck
4. Brown paper Bag containing hat from Daffy Duck

Item	Description	Finding	Conclusion
#1	GSR Kit from Yosemite Sam	A particle characteristic of gunshot primer residue was identified on one of the samples.	Positive
#2	GSR Kit from Elmer Fudd	Particles characteristic of gunshot primer residue were identified on the samples.	Positive
#3	GSR Kit from Daffy Duck	Particles characteristic of gunshot primer residue were not identified on the samples.	Negative
#4	Hat from Daffy Duck	Particles characteristic of gunshot primer residue were identified on one of the samples. Analysis was not completed on the remaining sample(s).	Positive

Remarks

Particles classified as characteristic of gunshot primer residue have compositions rarely found in particles from any other source.

A finding of “positive” for particles characteristic of gunshot primer residue on a person's hands means that individual either discharged a firearm, was in the vicinity of a firearm when it was discharged, or handled an item with gunshot primer residue on it.

A finding of "positive” for particles characteristic of gunshot primer residue on an item means that the item, at some time in its history, was in the vicinity of a firearm when it was discharged or came in contact with another item with gunshot primer residue on it.

A finding of “negative” for particles characteristic of gunshot primer residue, does not preclude the possibility of any of the above stated events.

The GSR instrumental analysis of the above evidence was performed in the Richfield laboratory; the interpretation of the data was performed in the Bowling Green laboratory.

The evidence is being returned to your department for retention.

Analytical Detail

These findings were determined using scanning electron microscopy/energy dispersive x-ray spectroscopy analyses.

16.5.5 Example 6 Glass Analysis

To: ACME Police Department
Det. Bunny
Main Street Road

BCI Laboratory Number: xx-xxxxx

Analysis Date: July 14, 2019

Issue Date: July 15, 2019

Agency Case Number: xxxxx

Offense: Homicide
Subject(s): John Doe
Victim(s): State of Ohio

Submitted on May 24, 2019 by Steve:

1. Broken window from crime scene
   -*Known clear glass*
2. Questioned clear glass fragment
3. Questioned clear glass fragment
4. Questioned clear glass fragment
5. Questioned clear glass fragment
6. Questioned clear glass fragment
7. Questioned brown glass fragment

Item	Description	Finding	Conclusion
#2	Questioned glass fragment	Matching individual break configuration to Item #1	Source Identification [1]
#3	Questioned glass fragment	Same color, thickness, refractive index, and elemental composition as Item #1 The questioned chip also exhibits a similar size and shape to a damaged area on Item #1	Support for same source [2, 3]
#4	Questioned glass fragment	Same color, thickness, refractive index, and elemental composition as Item #1	Support for same source [2]
#5	Questioned glass fragment	Same color as Item #2	Support for same source [2, 4]
#7	Questioned glass fragment	Different color from Item #1	Source Exclusion [5]

[1] The likelihood that the evidence arose from a different source is so remote as to be considered a practical impossibility.
[2] This association is not exclusive; other manufactured items with the same characteristics may exist.
[3] While they could not be conclusively identified to the same source, the items were found to exhibit unusual matching characteristics that would not be expected to be found in the population of this evidence type.
[4] Due to significant limiting factors, this association has decreased evidential value.
[5] The evidence exhibits fundamentally different characteristics than the known reference and could not have come from the same source

Remarks

Due to the size of the questioned glass fragment (Item #5) no instrumental analysis was possible.

The evidence is being returned to your department. Digital images are being retained at BCI.

Analytical Detail

This document is uncontrolled if viewed outside the BCI document management system.

These findings were determined using visual examination techniques, microscopical examination techniques (stereo microscope, PLM) and instrumental analyses (rIQ, micro-XRF).

16.5.6 Example 7 Vehicle Lamp Examination

17 To: ACME Police Department
Det. Bunny
Main Street Road

BCI Laboratory Number: xx-xxxxx

Analysis Date: July 14, 2019

Issue Date: July 15, 2019

Agency Case Number: xxxxx

Offense: Homicide
Subject(s): John Doe
Victim(s): State of Ohio

Submitted on May 24, 2019 by Steve:

1. Brake light
   -Dual filament lamp
2. Reverse light
   -Single filament

Item	Description	Finding		Conclusion
#1	Dual filament lamp	Glass envelope: Broken	Small Filament: Intact with a normal appearance	Not Incandescent
			Large filament: Heavily tinted and distorted	Incandescent
#2	Single filament lamp	Glass Envelope: Intact Filament: Intact with a normal appearance		Inconclusive

Remarks
A dual filament lamp typically functions as both a running light (small filament) and a brake light (large filament).

The evidence is being returned to your department. Digital images are being retained at BCI.

Analytical Detail
These findings were determined using microscopical examination techniques (stereo microscope).

16.5.7 Example 8 Pressure Sensitive Tape Analysis

To: ACME Police Department
Det. Bunny
Main Street Road

BCI Laboratory Number: xx-xxxxx

Analysis Date: July 14, 2019

Issue Date: July 15, 2019

Agency Case Number: xxxxx

Offense: Homicide
Subject(s): John Doe
Victim(s): State of Ohio

Submitted on May 24, 2019 by Steve:

1. Roll of red electrical tape
2. Roll of black electrical tape
3. Piece of electrical tape
4. Piece of electrical tape
5. Piece of electrical tape
6. Black smear
7. Partially burned Piece of electrical tape

Item	Description	Finding	Conclusion
#3	Questioned piece of electrical tape	Matching individual break configuration to Item #1	Source Identification [1]
#4	Questioned piece of electrical tape	Same color, width, and chemical composition as Item #2 The questioned tape also exhibited some significant matching surface striations traversing the edge to Item #2	Support for same source [2, 3]
#5	Questioned piece of electrical tape	Same color, width, and chemical composition as Item #1	Support for same source [2]
		Different color from Item #2	Source Exclusion [6]
#6	Questioned black smear	Same color as Item #2	Support for same source [2, 4]
		Different color from Item #1	Source Exclusion [6]
#7	Questioned piece of burned material	Similar color, and chemical composition as Item #2	Inconclusive [5]
		Different color from Item #1	Source Exclusion [6]

[1] The likelihood that the evidence arose from a different source is so remote as to be considered a practical impossibility.
[2] This association is not exclusive; other manufactured items with the same characteristics may exist.
[3] While they could not be conclusively identified to the same source, the items were found to exhibit unusual matching characteristics that would not be expected to be found in the population of this evidence type.

[4] Due to significant limiting factors, this association has decreased evidential value.
[5] The items exhibit both differences and similarities in observed and/or measured properties and/or chemical composition to the extent that no conclusion could be reached regarding an association or elimination.
[6] The evidence exhibits fundamentally different characteristics than the known reference and could not have come from the same source

Remarks
Item #6 is too small for instrumental comparisons.

The material from Item #7 exhibits similarities and differences from the known tape in Item #2. This may be due to the sample having been partially burned.

The evidence is being returned to your department. Digital images are being retained at BCI.

Analytical Detail
These findings were determined using visual examination techniques, microscopical examination techniques (stereo microscope) and instrumental analyses (FTIR).

17 Appendix I: Trace Evidence Note Abbreviations

Brn = Brown	~ = Approximately	Q, Quest = Questioned
Blk=Black	+ = And	RB, RHB = Right Hand Back
BPB = Brown Paper Bag	Amt = Amount	RH = Right Hand
Cl = Clear	Char(s)=Characteristic(s)	RP, RHP = Right Hand Palm
Cont = Containing	Conc = Concentrated	S/Z=Stereo-Zoom Microscope
Contr = Container	Cont'd = continued	S=Suspect
DK = Dark	Diff =Different	Sim=Similar
Evid=Evidence	Dia=Diameter	Sm = Small
Env = Envelope	FM=Fracture Match	SO = Sheriff's Office
ET = Evidence Tape	Frag(s)=Fragment(s)	SS = Submission Sheet
HS = Heat Sealed	ID = Identification	Std = Standard
Man = Manila	Ind = Individually	Stkr = Sticker
ME = Manila Envelope	Indiv=Individual	V=Victim
MCE= Manila Coin Envelope	IS = Information Sheet	Unk = Unknown
Mkd = Marked	Insuff = Insufficient	W/ = With
Pa = Paper	K, Kn = Known	W/O = Without
PB = Paper Bag	LG = Large	XP(s)=Crossed Polars
Pkg = Package	LB, LHB = Left Hand Back	XS=Cross Section
Pkt = Packet	LH = Left Hand	ATR = Attenuated Total Reflectance
Pl = Plastic	LP, LHP = Left Hand Palm	MSP = Microspectrophotometer
Rec'd = Received	LT = Light	PA = Particle Atlas
RET = Red Evidence Tape	Med = Medium	PDQ = Paint Database Query
Sld = Sealed	Micro = Microscopy, Microscopic(al)	LCV = Leucocrystal Violet
SME = Small Manilla Envelope	Mod = Moderate	Xyl Sub = Xylene Substitute
Sub = Submitted	NA, N/A = Not Applicable	RT = Room Temperature
Un-sld = Unsealed	Neg., (-) = Negative	RAC= Random Acquired Characteristic(s)
WE = White Envelope	NOAEV = Nothing of Apparent Evidential Value	
Wht = White	NT = Not tested	
WPB = White Paper Bag	NWN = No Work Necessary	
	Orig = Original	
	Pos, (+) = Positive	
	Prep = Prepared	