MICROANALYSIS UNIT

TEST METHODS

FOREWORD

The Microanalysis Unit of the Indiana State Police Laboratory Division has the responsibility for conducting forensic examinations and comparisons on a wide variety of trace physical evidence submitted by criminal justice agencies in the State of Indiana. The examinations include paint, plastics, glass, fibers, ignitable liquid residues, lamps, tapes, clandestine laboratory precursors, physical comparisons, and unknowns. These test methods are designed for the guidance of forensic scientists who support investigations of cases involving trace evidence. These test methods are to be used in conjunction with Laboratory Division Policies and the Quality Assurance Manual (QAM).

The Microanalysis Unit is staffed with trained and skilled forensic scientists who have academic backgrounds in chemistry and other physical science areas (see job description). Additionally these forensic scientists participate in extensive formalized training programs dealing specifically with the techniques for forensic examinations. These forensic scientists are under the direct control of the State Police Laboratory Division and are directly supervised by the Microanalysis Unit Supervisor. During the training program, the forensic scientist shall demonstrate competency through written tests, oral examinations, and sample analyses. The body of knowledge which comprises forensic science is a compilation of procedures adapted from other disciplines that encompass many of the physical and natural sciences. During the history of forensic science, a multitude of individuals have greatly contributed to the protocols, methods, and procedures that have become a routine part of analysis. All noted references contained in this document are a starting point and shall not be considered an all-inclusive list of available reference. This document supersedes all previous documents relating to trace examinations and is subject to change according to research, study, and laboratory policy.

This test method document is a general approach to the examination of trace physical evidence and the results as they relate to these examinations. While no two cases are identical, a general approach to their examination has been developed. The following are the test methods that shall be used for processing the “typical” case. Minor procedural deviations, other than those listed, may be employed with the approval of the Unit Supervisor.

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MICROANALYSIS UNIT
TEST METHODS

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1. Evidence Handling:

1.1. Scope: This test method is designed for the guidance of laboratory personnel who support investigations where trace evidence occurs as physical evidence in a variety of alleged criminal activities. All evidence submitted for analysis shall be handled, stored, and analyzed to protect the integrity of the evidence. Often a search is made on a myriad of different types of items for the presence of physical evidence, which have potential evidentiary value. The goal is to minimize the potential for cross contamination, destruction of evidence and to ensure the safety of the analyst. Specific procedures shall be used when multiple examination requests are involved on an item.

1.2. Precautions/Limitations: Forensic scientists shall take appropriate precautions to minimize contaminating, altering, or destroying the evidence for additional future examination. Specific procedures shall be used when multiple examination requests are involved on an item. There is always some risk to future examinations when an item of evidence is processed.

1.3. Related Information:

1.3.1. Appendix 1 Worksheets

1.3.2. Other Microanalysis Unit Test Methods

1.4. Instruments:

1.4.1. Stereomicroscope – a microscope which gives a three dimensional view capable of examining samples up to a magnification of approximately 70x. Stereomicroscopical observations (e.g., color, thickness, width, and reinforcement construction) provide initial and discriminating information for comparisons.

1.4.2. Heat Sealer – a device which allows for the heat sealing of plastic bags. Can be used in the sealing of plastic evidence bags.

1.5. Reagents/Materials: The varied nature of items dictates several types of containers be utilized. Paper sacks, paper envelopes, plastic bags, pill boxes, cardboard boxes, metal paint cans, and glass bottles are suitable for most items depending on the physical make-up of the sample. Permanent markers shall be used for marking the packaging.

1.6. Hazards/Safety:

1.6.1. General laboratory precautions for processing evidence include wearing of lab coats and gloves when contact with the item could occur.

1.6.2. Biohazards - Forensic scientist shall wear lab coat and gloves during the initial examination process (when the original item is open). Gloves shall be removed and hands washed prior to continuing with the examination. Pens and papers should not be handled with potentially contaminated gloved hands during the examination process. Pens that are handled with gloves shall be washed prior to handling with bare hands. If someone is to observe the examination of primary evidence they shall also be required to wear a lab coat and gloves. People may

be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination.

1.6.3. Sharps - Microscopy tools (i.e. scalpels, tweezers, etc…) and broken items of evidence pose a potential for punctures. Instrument injector needles can also be a hazard. Care shall be exercised to properly use and dispose of sharps.

1.6.4. Exposure to various materials and chemicals - Protective measures shall be used to comply with the provisions in the Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, Laboratory Safety Manual, and associated MSDS.

1.7. Reference Materials/Controls/Calibration Checks: N/A

1.8. Procedures/Instructions:

1.8.1. **Initial sample treatment:** Open the container, avoiding the breakage of existing seals when possible.

1.8.1.1. The identity of each item of evidence shall be verified by comparing the laboratory case number, item number, and evidence description between the Request for Laboratory Examination Form and the actual evidence.

1.8.1.2. The outer containers of evidence shall be marked with the initials of the forensic scientist, the laboratory case number, and item number.

1.8.1.2.1. If a forensic document examination is also being conducted the container shall not be marked until after the contents have been removed.

1.8.1.2.2. Whenever possible mark the actual specimen as well as all its containers.

1.8.1.2.2.1. Due to the nature of fire debris evidence, only the container shall be marked.

1.8.1.2.2.2. When marking specimens, care shall be taken not to interfere with forensically significant areas such as bloodstains, accidental characteristics, etc.

1.8.1.2.2.3. Permanent marking pens are the first choice when marking an item.

1.8.1.3. Verify that all evidence containers are properly sealed as per Laboratory Division guidelines and policies. (Evidence Policy #006)

1.8.1.4. Note if any suspected improper seals, cross contamination between items, or tampering has occurred. If so, the Laboratory Manager shall be immediately

notified and the situation documented in the analytical case notes.

1.8.1.5. Analysis notes and observations shall be initiated and continued throughout the analysis.

1.8.1.6. Verify the agency case number and item number marked on the evidence with the Request for Laboratory Examination form.

1.8.1.7. Verify that all the items of evidence are properly described on the Request for Laboratory Examination form. Compare each item of evidence to the descriptions on the form. Significant differences or conflicting information shall be correctly recorded on the form and initialed, and the correct information shall be updated in the Laboratory Information Management System (LIMS) case information. Incorrect information on the form shall be crossed out with a single line then corrected and initialed.

1.8.1.8. Store the evidence in a secure temporary storage area until release of the case back to the evidence clerk.

1.8.1.8.1. The following are approved secure temporary storage areas.

1.8.1.8.1.1. The Trace Evidence Storage Room Lockers (Room S311F).

1.8.1.8.1.2. The locking evidence cart garages (Room S311A).

1.8.1.8.1.3. The individually keyed examination rooms (Rooms S311B, S311G, and S311H).

1.8.1.8.2. Even though the trace rooms have limited access, measures shall be taken to ensure that evidence is not left out unattended. Evidence shall be secured when analysts are out of the laboratory (i.e. lunch, breaks). It is also understood that some items of evidence after they have been prepped and are in the process of being examined by instrumental methods do no lend themselves to always remain in an approved storage location during analysis. After items have been processed for analysis they shall be considered a work product and have slightly different security procedures during their analysis (e.g. auto-sampler vials, samples in vacuum).

1.8.1.8.3. If a forensic scientist wants to house evidence in a location other than the temporary evidence storage locations below, it shall be approved by the Microanalysis Unit Supervisor and when applicable the Laboratory Manager.

1.8.1.9. Every effort should be made to avoid handling evidence repeatedly. If necessary, the evidence may be closed and maintained in a secure temporary storage area until the analysis is complete.

1.8.2. Fingerprints

1.8.2.1. If an item is also submitted for fingerprint analysis, then the scientist shall take special precautions to preserve fingerprints during the examination process. If the analyst examines the item before fingerprint processing has occurred the following precautions shall be taken:

1.8.2.1.1. The analyst shall double glove (i.e. cloth gloves with latex gloves on the outside).

1.8.2.1.2. Handling of the items should be kept to a minimum.

1.8.2.2. For items of tape evidence, multiple examinations from different disciplines within the laboratory are often requested on the same piece of evidence. The order that the examinations will be conducted needs to be formalized either by the agency, the investigator, or the forensic scientists involved in the examinations requested. Although each type of examination is ostensibly independent of each other, any one can have a significant effect on another. Examinations should be conducted in order of increasing importance to a case. It is highly desirable that the disciplines be selected in an order that provides the greatest discrimination for all of the examinations. If another discipline is chosen before the tape examination, obtaining an unadulterated representative sample should be considered.

An effort should be made not to alter the condition of a questioned specimen (e.g., shape, position, layers, or relation of one piece of tape to another) before the preliminary examination. Tape evidence shall be examined in a manner that preserves other types of trace evidence. If tape is received in a tangled condition an attempt shall be made to separate it with a careful peel. More aggressive techniques such as solvents, freezing, liquid nitrogen, or heat may be used if necessary. However, these techniques could affect the outcome of subsequent analyses, and shall, therefore, be applied only to the extent necessary.

In some circumstances it may be desirable to obtain a sample cutting from the tape before a sample is analyzed for latent fingerprints. Samples for testing shall not be cut from the ends of the tape in case there is a possibility of physically matching a questioned specimen to a known specimen. Take the test sample away from the existing end(s) and mark the location. Physical matches shall always be considered if two pieces of tape are being compared. If the ends have been distorted, a physical match may be possible. A physical match shall be documented and include descriptive notes. Additionally, if the tape is fiber reinforced, describing the condition of corresponding threads and their relative positions in the separated area on the questioned and known pieces provides a detailed corroborative description. All physical matches shall be photographed for documentation. This photograph shall not be considered evidentiary in nature. If a physical match is not possible, comparison of the tape shall be conducted.

Necessary precautions shall be used to not add additional fingerprints, or obliterate existing fingerprints.

1.8.3. DNA: If an item is also submitted for DNA analysis, then the forensic scientist shall take special precautions to prevent contamination of the item with DNA containing materials. Even if DNA sampling occurs first, the scientist should still comply with precautions during examination for potential DNA analysis in the future.

1.8.3.1. During the examination the forensic scientist shall wear a disposable face mask and gloves until the item is completely examined.

1.8.3.2. Gloves shall be changed after each item.

1.8.3.3. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

1.8.3.4. At the conclusion of sampling of items with DNA analysis requested, the face mask and gloves shall be removed, disposed of and hands washed.

1.8.4. Toolmarks: If an item is also submitted for toolmarks, then the forensic scientist shall take precautions to prevent the loss or destruction of evidence during examination. The forensic scientist shall consult with a member of the Firearms Unit to determine the most efficient method for collecting trace evidence that will not interfere with toolmark analysis when necessary. If the forensic scientist examines an item before the toolmark examination has occurred the following precautions shall be taken:

1.8.4.1. When the analyst is collecting trace evidence (i.e. paint, building materials, etc.).

1.8.4.1.1. The analyst shall not use metal instruments to collect trace evidence or mark the item.

1.8.4.1.2. The analyst shall take precautions to protect the item from damage (i.e. scratches).

1.8.4.1.3. The tool shall not be altered unless absolutely necessary, and with the consultation of the toolmark analyst.

1.8.4.1.4. If debris is to be removed from the marking edge it shall be documented, photographed, and noted before removal.

1.8.4.2. If the toolmark forensic scientist examines the item before trace evidence processing has occurred, the following precautions shall be taken:

1.8.4.2.1. The toolmark forensic scientist shall consult with the trace evidence forensic scientist, and form the best course of action to be taken, or

have the trace forensic scientist present during the initial examination.

1.8.4.2.2. Handling of the item shall be cautious to not disturb any debris that might be trace evidence. If trace evidenced is found, then it shall be placed in an appropriate container for future analysis.

1.8.4.2.3. Keep the paper that the item is examined over; the paper may contain trace evidence that may be used for future analysis. Place the paper in the original packaging, with the item, when applicable.

1.8.4.2.4. Change gloves and paper in between items if they are packaged separately.

1.8.4.2.5. When analyzing an item that has a possible trace evidence transfer, no more than one item shall be examined at a time.

1.8.5. Footwear: If an item is also submitted for footwear examination, then the forensic scientist shall take special precautions to preserve the footwear impressions or the shoe during the examination process. If the forensic scientist examines the item before footwear processing has occurred, the following precautions shall be taken:

1.8.5.1. The forensic scientist shall consult with the footwear impression analyst, and form the best course of action to be taken, or have the footwear analyst present during the examination.

1.8.5.2. Handling of the item shall be held to a minimum to not disturb any debris that would aid in the identification of the impression.

1.8.5.3. The bottom of the footwear shall not be altered unless absolutely necessary, and with the consultation of the footwear analyst.

1.8.5.4. If debris is to be removed from the shoe or impression it shall be documented, photographed, and noted before removal. This photograph shall not be considered evidentiary in nature.

1.8.5.5. If the footwear analyst examines the item before trace evidence processing has occurred, the following precautions shall be taken:

1.8.5.5.1. The footwear forensic scientist shall consult with the trace evidence forensic scientist, and form the best course of action to be taken, or have the trace forensic scientist present during their examination.

1.8.5.5.2. Handling of the item shall be cautious to not disturb any debris that might be trace evidence. If trace evidence is found, then it shall be placed in an appropriate container for future analysis.

1.8.5.5.3. Keep the paper that the item is examined over. The paper may contain trace evidence that may be used for future analysis (e.g. glass,

fibers). Place the paper in the original packaging, with the item, when applicable.

1.8.5.5.4. Wear gloves when examining footwear and change gloves and paper in between items if they are packaged separately.

1.8.5.5.5. When analyzing an item that has a possible trace evidence transfer, no more than one item shall be examined at a time.

1.8.6. Firearms: If an item is also submitted for firearms analysis, then the forensic scientist shall take safety precautions as well as special precautions to prevent the loss or destruction of evidence during examination. If the forensic scientist examines an item before a firearms examination has occurred the following precautions shall be taken:

1.8.6.1. If the item is a firearm,

1.8.6.1.1. The forensic scientist shall ensure the firearm is unloaded and safe to examine.

1.8.6.1.2. The forensic scientist shall not use metal instruments to collect trace evidence from the chamber area or barrel.

1.8.6.2. If the item is a bullet or projectile,

1.8.6.2.1. The forensic scientist shall not use metal instruments to collect trace evidence or mark the item.

1.8.6.2.2. The forensic scientist shall take precautions to protect the item from damage (i.e. scratches).

1.8.6.3. If the item is a fabric or textile,

1.8.6.3.1. The forensic scientist shall consult with a member of the Firearms Unit to determine the most efficient method for collecting trace evidence that shall not interfere with distance determination analysis.

1.8.7. Forensic Documents: If an item is also submitted for a forensic document examination, then the forensic scientist shall take precautions to not impart any changes that would prevent a full examination. The following precautions shall be taken:

1.8.7.1. Do not impart any additional folds to documentary evidence. Evidence containers with documentary evidence shall not be folded or bent.

1.8.7.2. Do not impart any additional impressions to documentary evidence. Evidence containers shall be written on before evidence is placed inside. Evidential seals on containers shall be marked with very light pressure using

a writing instrument such as a fiber tipped evidence marker.

1.8.7.3. Do not impart additional impressions on documentary evidence by writing on paper that is in contact with the evidence. Forensic scientist shall be aware that impressions may be created on documents as far down as ten sheets of paper or more from a document being written on.

1.8.7.4. If destructive testing is going to take place on a document by another forensic discipline (i.e. chemical processing, cutting a portion of the evidence), an image of the original evidence shall be made first. This may be a photocopy, electronic image, traditional photography, etc. This photograph shall not be considered evidentiary in nature.

1.8.8. Trace recovery of microscopic evidence: A careful visual search and when applicable a collection procedure is conducted. Care shall be taken to not lose evidence from items. When warranted, these methods can be used in conjunction or separately. The notes shall reflect the techniques used for detection, collection, and preservation of the evidentiary items. Multiple examination rooms equipped for collection are available for collection of trace evidence. Each type of trace evidence and each case has different advantages and disadvantages for collection. Refer to the specific test method for each discipline.

1.8.8.1. Particle picking: The evidence of interest may be removed with a tool (i.e. forceps). If removing an item of evidence from a rigid location (i.e. glass from the sole of a shoe) note the location from which the evidence is located.

1.8.8.2. Scraping: The item is suspended above the examination surface and gently scraped with a spatula. Scraping in a downward direction allows surface hairs, fibers, and debris to fall onto the examination surface. The fibers and debris can be collected using an adhesive tape. Do not scrape heavily bloodstained articles that can create blood to become airborne. The item shall not be scraped too vigorously causing debris to fly all over the room.

1.8.8.3. Tape Lift: Clear plastic tape can be applied sticky side down to the surface of the item to be examined, pressed down, and then pulled away. Fibers and other surface debris will adhere to the adhesive on the tape. This is especially useful on large items, or dark-colored items on which fibers of interest may be difficult to see. A tape roller is practical for this application. The lifts are generally placed on a transparent backing which protects against contamination and permits the samples to be easily viewed.

1.8.8.4. Vacuuming: An approved vacuum sweeper may be used to collect hairs, fibers, dust, and debris on a particular item.

1.8.9. Cross transfer prevention:

1.8.9.1. Only specimens which have been securely and appropriately packaged shall be examined. Evidence not meeting this criterion shall be reported to the Unit Supervisor for review. If the possibility of cross contamination appears likely, no examination shall be performed and a Certificate of Analysis shall be issued to reflect that opinion.

1.8.9.2. The following steps shall be taken to minimize the possibility of cross transfers.

1.8.9.2.1. The examination surface shall be cleaned off and a clean piece of exam paper placed out on the examination surface.

1.8.9.2.2. The examination surface shall be cleaned off between each item.

1.8.9.2.3. When analyzing for possible trace evidence transfers no more than one item shall be examined at a time.

1.8.9.2.4. Pens and sampling tools/equipment shall be cleaned between examinations of another item.

1.8.9.2.5. Drafts around the examination area shall be minimized.

1.8.9.2.6. The examination of questioned and known items for trace evidence shall be conducted at different times to help prevent contamination. It is recommended that questioned items or items with the most probative value be examined first.

1.8.9.2.7. Any contact, condition, or situation which could cause contamination or otherwise compromise the trace evidence examination shall be documented.

1.8.10. RETURN OF TRACE EVIDENCE:

1.8.10.1. All evidence packages shall be properly sealed in a manner to prevent tampering and eliminate loss or contamination of the trace evidence through open edges.

1.8.10.2. No items of evidence shall be retained by the analyst without approval from the submitting agency and the Microanalysis Unit Supervisor.

1.8.10.3. If an item has been repackaged, the container shall be marked to reflect this information.

1.8.10.4. Small or loose trace evidence shall be secured. When possible the evidence collected from an item shall be marked, sealed, and returned with

the original item.

1.9. Records: All evidence descriptions shall be described in detail in the analysis notes or worksheet. Details shall be sufficient to enable the forensic scientist, or other qualified individual, to identify the evidence at a later date.

1.9.1. Notes shall be taken to record a physical description for the item of evidence. Describe how the item was received (packaging, how it is sealed, all the items that are contained, etc.). If discrepancies exist between the description of the evidence and the actual item, it shall be noted. The macroscopic description shall include the color, condition, label information, measurements, diagrams, pictures, and any remarkable details when applicable.

1.9.2. Any worksheets or separate notes shall become part of the case record.

1.9.3. The date of each test or observation shall be recorded in the case notes.

1.9.4. Photographs, if taken for the purposes of analysis, shall be included in the notes or uploaded to LIMS and attached as part of the analysis. Proper identifiers and scales shall be in the content of the photograph for identification when applicable.

1.10. Interpretations of Results: Proper evidence packaging is determined by an intact and sufficient seal that prevents loss, cross-contamination or deleterious change of the evidence in a container. Markings shall be on the container and the seal for the purposes of identification and security.

1.11. Report Writing: All evidence shall be described as being “sealed” in the report, unless it is the forensic scientist’s opinion that there is a question regarding the integrity of a seal. In the event that a seal may be insufficient to prevent loss, cross-contamination or deleterious change, the word “sealed” shall be removed from the evidence description on the Certificate of Analysis. If an item is deemed compromised, it shall be reported in the results.

1.12. References:

1.12.1. ASTM E1459 Standard Guide for Physical Evidence Labeling and Related Documentation

1.12.2. ASTM E1492 Standard Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Lab

1.12.3. SWGMAT Trace Recovery Guidelines [Online] (October 1999). Available: http://www.fbi.gov/hq/lab/fsc/backissu/oct1999/index.htm

2. Proficiency Testing

2.1. Scope: Each forensic scientist conducting analysis in the Microanalysis Unit shall participate in the Laboratory Division's proficiency testing program. Participation, evaluation, documentation, and any necessary corrective actions shall comply with proficiency testing procedures listed in the Laboratory Division's Quality Assurance Manual. Analysis of proficiency samples shall use the procedures in the Microanalysis Unit's Test Methods. The following are guidelines for compliance with the proficiency testing program. All Microanalysis Unit Forensic Scientists shall demonstrate the continuing capability by completing proficiency tests.

2.2. Precautions/Limitations:

A qualified forensic scientist will encounter evidence covering a wide variety of materials. The unit receives a myriad of different types of evidence usually falling into one of the general categories; lamps, fibers, glass, paint, ignitable liquid residues, clandestine laboratory samples, tape, physical matches and unknowns. However, not every case assigned to the unit will fall into one of these categories. Many of the infrequent types of evidence do not have an appropriate proficiency test available or applicable. Therefore only the trace categories that the laboratory has accreditation in shall be proficiency tested.

2.3. Related Information:

2.3.1. Appendix 1 Worksheets
2.3.2. Appendix 2 Abbreviations
2.3.3. Appendix 3 Definitions
2.3.4. Other Microanalysis Unit Test Methods
2.3.5. Quality Assurance Manual

2.4. Instruments: Each proficiency test in the Microanalysis Unit uses different instruments depending on the sub-discipline. The instruments are specified in each test method.

2.5. Reagents/Materials:

2.5.1. External Proficiency Samples and Internal Proficiency Samples

2.5.2. The materials and reagents for each sub-discipline proficiency test shall be listed in that specific sub-discipline test method.

2.6. Hazards/Safety: The proficiency in each sub-discipline category shall have hazards associated with the procedures in each of the test methods. The specific test method shall be referenced for each sub-discipline proficiencies safety and hazards concern.

2.7. Reference Materials/Controls/Calibration Checks:

2.7.1. External Proficiency Testing: This sample shall be obtained from an approved test provider annually.

2.7.2. Open Internal Proficiency Testing: A proficiency test known as such to the participant. This sample can be either a proficiency sample prepared and distributed within the respective laboratory or an inter-laboratory proficiency sample.

2.7.2.1. All internal proficiency samples shall be prepared using known reference materials.

2.7.3. Blind Proficiency Testing: A proficiency test not known as such to the participant. If blind proficiency testing is conducted, then the Microanalysis Unit Supervisor or the Chemistry Section Supervisor shall prepare the blind proficiency samples using known standards or old external proficiency tests.

2.7.4. Proficiency Re-analysis: The Microanalysis Unit Supervisor shall select case items for proficiency re-analysis from each forensic scientist's completed cases. Old external proficiency tests may also be used for a re-analysis.

2.8. Procedures/Instructions:

2.8.1. Microanalysis Unit External Proficiency Testing

2.8.1.1. The Indianapolis laboratory shall participate in one external proficiency test in each accredited sub-discipline category that is in the scope for trace analysis and that there is an approved proficiency test available. This will be an open proficiency testing situation.

2.8.2. Microanalysis Unit Internal Proficiency Testing

2.8.2.1. The Indianapolis laboratory shall participate in internal proficiency tests in each accredited sub-discipline that is in the scope for trace analysis and that there is not an approved proficiency test available. This will be an open proficiency testing situation.

2.8.3. Forensic Scientist Proficiency Samples: All Microanalysis Unit Forensic Scientists shall demonstrate maintenance of skills and ability in each type of sub-discipline category that they conduct casework in by completing proficiencies.

2.8.3.1. Each Trace analyst shall take a minimum one proficiency test every year.

2.8.3.2. Each analyst shall take a proficiency in each sub-discipline that they are conducting casework in at least once every accreditation cycle.

2.8.4. Proficiency Test Corrective Action: If corrective action is required after a proficiency test, the analyst shall be taken off casework in that sub-discipline until the corrective action has been resolved. The corrective action may include additional training and supervised casework.

2.9. Records:

2.9.1. External and Internal Proficiencies: All notes, documentation, and results shall be returned to the Microanalysis Unit Supervisor by assigned deadline. The Microanalysis Unit Supervisor shall ensure the completed necessary documentation has been submitted to the external vendor for evaluation.

2.9.2. Proficiency Re-Analysis: After the re-analysis is complete, copies of all documentation including conclusions for and the original analysis shall be forwarded to the Microanalysis Unit Supervisor. The original re-analysis documentation shall be stored in the original case record with the Certificate of Analysis and notes of the original analysis (A new Certificate of Analysis shall not be created for the re-analysis).

2.9.3. A Proficiency Test Report form shall be prepared upon evaluation of the test results by the Microanalysis Unit Supervisor responsible for administering the proficiency and given to the appropriate forensic scientist and disseminated as per the Laboratory Division's Quality Assurance Manual.

2.9.4. The forensic scientist shall maintain spectra, notes, and photographs in a similar manner as casework documentation.

2.10. Interpretations of Results: Satisfactory completion shall be determined by the Microanalysis Unit Supervisor.

2.10.1. External Proficiency: The Microanalysis Unit Supervisor shall review and evaluate the proficiency test by comparing to the manufacturer's report when it becomes available.

2.10.2. Open Internal Proficiency: The Microanalysis Unit Supervisor shall review and evaluate the analysis of the known material in the sample(s).

2.10.3. Proficiency Re-Analysis: The Microanalysis Unit Supervisor shall review and evaluate the re-analysis by comparing to the original analysis.

2.11. Report Writing: The proficiency shall be written up in a manner that is consistent with the sub-discipline protocol for that specific test method.

2.12. References: N/A

3. Casework Guidelines and Reviews

3.1. Scope:

3.1.1. All Microanalysis Unit forensic scientists shall demonstrate both technical knowledge and competency in each specific sub-discipline after the completion of their training program in each sub-discipline. After being approved and released to conduct trace analysis in each sub-discipline the analyst shall have a period of supervised casework which shall include a 100% technical review of their casework. The Unit Supervisor shall occasionally evaluate the evidence and review the raw instrumental data.

3.1.2. After being approved and released to conduct non-supervised trace analysis in each sub-discipline, all analysts shall have technical reviews of their casework.

3.2. Precautions/Limitations: A forensic scientist will encounter evidence covering a wide variety of materials. The unit receives a myriad of different types of evidence usually falling into one of the general categories; lamps, fibers, glass, paint, ignitable liquid residues, clandestine laboratory samples, tape, physical matches, and unknowns. Every case is different and therefore not every aspect of each case may is always be covered in the technical review form. Also, not every case assigned to the unit will fall into one of the major discipline categories. Many of the infrequent types of evidence do not have a designated peer technical review sheet. However, the cases shall still be peer technical reviewed.

3.3. Related Information:

3.3.1. Appendix 1 Worksheets
3.3.2. Appendix 2 Abbreviations
3.3.3. Appendix 3 Definitions
3.3.4. Other Microanalysis Unit Test Methods

3.4. Instruments: Casework in the Microanalysis Unit uses different instruments depending on the sub-discipline.

3.4.1. The instruments are specified in each test method.
3.4.2. When instrumental analyses are conducted, operating parameters shall be recorded in the case notes.

3.5. Reagents/Materials: N/A

3.6. Hazards/Safety: Casework in each sub-discipline shall have hazards associated with the procedures in each of the test methods. The specific test method shall be referenced for each sub-discipline safety and hazards concern.

3.7. Reference Materials/Controls/Calibration Checks: N/A

3.8. Procedures/Instructions:

3.8.1. The length of time that supervised casework technical review is conducted shall depend on the satisfactory completion of the casework with minimal suggestions being made for the analyst. The technical reviews shall be conducted by a member of the Microanalysis Unit with casework experience in that sub-discipline.

3.8.1.1. At minimum, 5 cases after the training module shall be reviewed.

3.8.2. All casework shall be administratively and technically reviewed prior to the release of the Certificate of Analysis. The technical review shall be conducted by someone with casework experience in that sub-discipline.

3.8.2.1. Technical Reviews**:** A technical review is an evaluation of examination records which form the basis for a scientific conclusion and the Certificate of Analysis after examination has been performed. This review consists of determining whether the appropriate examinations have been performed, the conclusions are consistent with the recorded data and are within the scope of the discipline or category of testing.

3.8.2.2. Administrative Reviews: Administrative Reviews are those reviews of administrative records and a cursory review of analytical information prior to the approval of the report. This is not intended to be a technical review, but may include Test Method compliance and technical matters. The administrative review takes place after a technical review.

3.8.3. Technical and Administrative reviews shall not be conducted by the author of the Certificate of Analysis

3.9. Records

3.9.1. Reviews shall be documented in the case record. An electronic copy of the form used for the technical review shall be appropriately named and saved in the respective case record in the LIMS system by the reviewer.

3.9.2. The analyst is responsible for preparing accurate, complete and organized examination records (case notes). The analyst shall review documentation constituting the case file (examination and administrative records) for compliance with laboratory policy and procedures and technical accuracy prior to submitting for administrative or technical review.

3.9.3. When the analyst submits the case for technical and administrative review, the hardcopy or electronic examination record and casework shall be considered completed. The Unit Supervisor shall also be notified if additional analysis is performed after the case is submitted for administrative or technical review.

3.10. Interpretations of Results:

3.10.1. Changes to notes**:** If the correction requires a change(s) to case record, the analyst shall make the corrections in the electronic version of the examination record and upload the corrected electronic version of the examination record to LIMS under the Case Images section of the Imaging Module with appropriate nomenclature. All pages of the original electronic examination record shall be retained. If possible, the corrected file should be sent back to the original reviewer.

3.10.2. Resolution of Conflicts of Opinion:

3.10.2.1. Once a formal verification and/or case review has begun, the same reviewer, if available, shall complete the process.

3.10.2.2. If the analyst and reviewer disagree, the analyst shall not seek a second reviewer. Both analyst and reviewer shall consult the Unit Supervisor to attempt to resolve the disagreement.

3.10.2.3. Unit Supervisor shall be notified of substantive variations of opinions. The analyst, the reviewer, and Unit Supervisor shall discuss the examination results, interpretation, opinions and conclusions. The technical disagreement, discussion and resolution shall be documented in the case notes.

3.10.2.4. If the difference of opinion cannot be resolved, Unit Supervisor(s) shall notify the Section Supervisor and Quality Assurance Manager to pursue resolution as per Laboratory Quality Assurance Procedures.

3.10.2.5. After consultation with Quality Assurance Manager, Section Supervisor, Unit Supervisor and other staff as necessary, the situation will be evaluated and a determination will be made if a corrective action is to be required.

3.11. Report Writing:

3.11.1. Changes to Reports: Prior to release: If the correction requires a change to the report prior to the approval and release of the original report, the analyst shall make the correction, and send the corrected file back to the original reviewer, if possible.

3.11.1.1. After release: If the correction requires a change(s) to the Certificate of Analysis after the original report has been released, the analyst shall make the necessary correction(s) and issue an Amended Report. The case reviewer shall perform an Administrative Review on the Amended Report and shall ensure all necessary corrections were made**.**

3.12. References: N/A

4. Tape Examinations

4.1. Scope:

This test method is for the analysis, examination, and comparison of evidentiary pressure sensitive tapes. The forensic examination of pressure sensitive tape encompasses the determination of physical construction and chemical composition of tape products. This test method provides an overview of techniques applied to the analysis of tape components. 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).

4.2. Precautions/Limitations:

4.2.1. The analysis and comparison of tape evidence in the forensic science laboratory can provide information due to the variability of tape products. However, some classes of tape exhibit more variability than others. In general, the more complex the product (e.g., duct tape), the more variable it is. Studies have shown differences between randomly selected rolls of tape. (References at the end of this section.) Because of the ever-changing tape markets, suppliers, and economics, it is not practical to establish the statistical probability that a given sample would have the same physical and chemical characteristics as a randomly selected tape.

4.2.2. Tape samples may be degraded by environmental exposure or subjected to physical damage. Tape may not be in its original condition due to weathering, stretching, chemical exposure, etc. The strength of an association between a damaged piece of tape and a more pristine sample might be weakened depending upon the degree of damage. In some cases, the damaged tape may be unsuitable for comparison purposes. These changes may limit the information obtained from the analyses. Tape does not always allow for the full range of examinations. The examinations and analyses that are performed shall be documented in the forensic scientist's notes.

4.2.3. If tape is received in a tangled condition an attempt shall 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 shall, therefore, be applied only to the extent necessary.

4.2.4. Although quantitative and semiquantitative instrumental methods are available for analysis, they are not appropriate for most tape analyses because of the typical condition of the tape.

4.2.5. The information available from a heterogeneous specimen may diminish as its size is reduced and its condition degrades. As sample size is reduced, it may no longer be representative of the original material.

4.3. Related Information:

4.3.1. Appendix 1 Worksheets
4.3.2. Appendix 2 Abbreviations
4.3.3. Appendix 3 Definitions
4.3.4. Test Method for Fibers
4.3.5. Test Method for Physical Edge Match

4.4. Instruments:

4.4.1. Stereomicroscope – a microscope which gives a "three dimensional view" capable of examining samples up to a magnification of approximately 70x. Stereomicroscopical observations (e.g., color, thickness, width, and reinforcement construction) provide initial and discriminating information for tape comparisons.

4.4.2. Polarized Light Microscope (PLM) - a microscope which can be used to evaluate the optical properties of tape samples. Characterization of inorganic materials and other tape additives are accomplished with the use of PLM. Optical properties of oriented polymers such as polypropylene (MOPP and BOPP) and polyester can also be determined. PLM is also used to evaluate and differentiate the reinforcement fibers of tapes (e.g., duct tape and strapping tape).

4.4.3. Fourier Transform Infrared (FTIR) Spectrometer– an analytical instrument capable of chemically analyzing the molecular vibrations of the chemical components of tape. Organic and some inorganic constituents may be evaluated with the use of infrared spectroscopy. These components include the backing polymer, adhesive elastomer, plasticizers, additives, and reinforcement fibers. The use of an ATR (attenuated total reflectance) accessory is particularly useful for surface analysis of a larger area of the adhesive and backing.

4.4.4. Scanning Electron Microscope with Energy Dispersive Spectroscopy (SEM/EDS) – an analytical instrument capable of chemically analyzing tape components and to magnify samples. It can be utilized for the characterization of the inorganic constituents of tape components. Additionally, SEM has imaging capabilities to evaluate surface topography of tape backings.

4.4.5. Calipers – an instrument capable of accurately and precisely measuring the thickness of different samples. It can be used to measure the thickness of different tapes and tape components.

4.4.6. Microtome – an instrument for cutting thin cross sections of specimens. It can be used to make cross sections of tape samples and tape components.

4.4.7. UV Box – an instrument designed to allow for the viewing of specimens in short and long wavelengths of light. It can be used to look at the reinforcement fibers in duct tape.

4.5. Reagents/Materials: Chemical solvents are used (i.e. xylene substitute, acetone, and pentane) in the separation and processing of the tape.

4.5.1. Microscope slides
4.5.2. Microscope cover slips
4.5.3. Mounting medium
4.5.4. Scalpel or razor blade
4.5.5. Low e^- microscope slide
4.5.6. SEM stubs
4.5.7. Roller knife.
4.5.8. Disposable gloves
4.5.9. Laboratory coat
4.5.10. Tweezers
4.5.11. Probes
4.5.12. Hair dryer
4.5.13. Liquid nitrogen

4.6. Hazards/Safety:

4.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as “Biohazard” shall be handled with gloved hands during the entire examination process. Items handled with gloves shall be disinfected prior to handling with bare hands.

4.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling occurs first, the forensic scientist shall still take precautions during examination for potential additional DNA analysis in the future. During the examination the forensic scientist shall also wear a disposable face mask. Gloves shall be changed after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

4.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to minimize additional fingerprints, or obliterating existing fingerprints.

4.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

4.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS’s shall be consulted.

4.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

4.7. Reference Materials/Controls/Calibration Checks:

4.7.1. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

4.7.2. Questioned and known samples shall be analyzed under similar conditions.

4.7.3. The calipers shall be performance checked as required in Appendix 13.

4.7.4. The SEM instrument shall be performance checked as required in Appendix 5.

4.7.5. The FTIR instrument shall be performance checked as required in Appendix 4.

4.8. Procedures/Instructions:

This section provides an overview of suggested flow of analytical techniques to be utilized for the analysis of tape. The selection of methods may vary depending upon sample size, condition, and/or examination request.

4.8.1. Initial Tape Evaluation and Handling

Each case shall be evaluated to select appropriate procedures in terms of sampling, collection, preservation, and order of examinations.

4.8.1.1. Different forensic disciplines may be called upon to examine the same item of evidence. The order in which the examinations shall be conducted needs to be resolved on a case-by-case basis. The order of examinations shall be selected and conducted so as to preserve the most transient evidence and provide the greatest discrimination and most valuable information. Forensic scientists may make the submitting agency aware of the effects that some processing and examinations may have upon other examination requests. If another discipline is chosen before the tape examination, obtaining an unadulterated representative sample shall be considered. Samples for testing shall not be cut from the ends of the tape if there is a possibility of a physical end match between specimens. A sample shall be obtained from an area that does not interfere with the existing end(s), and the location shall be marked.

4.8.1.2. When the amount of a tape specimen present for comparison purposes is adequate in size—as deemed by the forensic scientist —bulk or lot sampling is the sampling method of choice. Considerations involved with bulk sampling shall include where the sample is taken, how much sample is taken, and if the sample is considered representative of the whole. The examiner shall be able to explain how the samples were taken and why the sampling technique was used. Nondestructive methods shall be exhausted before

subjecting the sample to any destructive tests.

4.8.1.3. Techniques to untangle tape specimens shall be chosen with care to minimize alterations in the chemical or physical properties. Methods include mechanical separation using warm air, liquid nitrogen, or appropriate solvents. If tape is received in a tangled condition an attempt shall 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 shall, therefore, be applied only to the extent necessary.

4.8.1.4. Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics. Preliminary examination of tape construction shall include its general appearance macroscopically and under a stereomicroscope, including any adhering matter. For all pressure sensitive tapes, document and record any physical damage (e.g., worn, cut, torn, frayed). The following general macroscopic characteristics shall be observed and documented

4.8.1.4.1. General condition
4.8.1.4.2. Wad, flat pieces, or fragments
4.8.1.4.3. Dimensions (e.g., nominal width and length)
4.8.1.4.4. Number of pieces
4.8.1.4.5. Colors
4.8.1.4.6. Severed ends for possible physical matches

4.8.1.5. The item of evidence shall be preserved in a manner that does not interfere with future testing. All procedures shall be conducted in such a manner to ensure that no cross-contamination occurs. The item shall be photographed or described prior to conducting any analyses in order to provide documentation of original condition. Transient evidence (e.g., hair, fiber, paint) shall be preserved and documented.

4.8.2. Physical Edge Match Evaluation

Physical end matches can provide individualizing associations. When conducting comparison examinations between two or more tape specimens, the free ends shall be carefully examined for possible physical end matches. A physical end match is defined as free ends of separate pieces of tapes that physically fit together demonstrating that the two pieces were once one continuous piece. Even though this type of association is the most compelling type of association, the forensic scientist may continue with a complete analytical analysis of these specimens depending upon the quality of the end match.

4.8.2.1. Observe the tear or cut pattern from the backing and adhesive side of both specimens to determine if a physical association is plausible. For finer detail, a stereomicroscope shall be used to examine the ends.

4.8.2.2. If the backing is distorted or folded over and adhered to the adhesive layer, gently straighten it out to restore the torn/cut edge. This may be

accomplished with the careful use of forceps, gentle heat, or mild solvent.

4.8.2.3. Depending on the type of tape, manufacturing marks, creping on a paper backing, printing, or any other continuous surface features may be present across fractured edges and would provide additional points of comparison.

4.8.2.4. Determine if there are individualizing characteristics (e.g., a flaw or mark) that extends across the fracture. This would be an accidental or anomalous mark that initiates on one piece and terminates across the fracture edge on the other.

4.8.2.5. 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.

4.8.2.6. Any physical associations shall be documented with descriptive notes. Physical associations between specimens that link an individual to a crime scene or to another individual shall be imaged. The imaging method shall be dimensionally accurate and include a measuring scale.

4.8.2.7. Any/all associations between a question specimen and a known specimen shall be verified by another qualified forensic scientist.

4.8.3. Physical Characteristic Evaluation

Macroscopic and stereomicroscopical observations (e.g., color, thickness, width, and reinforcement construction) provide initial and discriminating information for tape comparisons. Structural details, such as design, construction, and composition, can provide information that may assist the forensic scientist in reaching a conclusion. Physical characterization of tape specimens is the initial step of a comprehensive forensic pressure sensitive tape analysis. The construction, composition, and color of tapes vary and, therefore, are useful characteristics for forensic examinations. Visual characteristics and physical measurements are the quickest, most discriminating and least invasive examinations. If no significant physical differences are found between samples, instrumental analyses are warranted.

4.8.3.1. General Characteristics The following characteristics shall be documented when applicable:

4.8.3.1.1. Color of adhesive and backing
4.8.3.1.2. Surface texture
4.8.3.1.3. Width measurement
4.8.3.1.4. Overall thickness
4.8.3.1.5. Backing thickness

Each of these characteristics can have a number of sub-elements, all of which can be characterized to complete the examination. Physical characteristics of a tape may change after removal from the original roll. The forensic scientist shall decide what is within an acceptable tolerance. Any

measuring devices shall be properly performance checked with applicable quality control.

4.8.3.2. Backing The type of backing shall be recorded (e.g., paper, polymer film). The backing shall be examined both macroscopically and by using a stereomicroscope for color, texture, and appearance under multiple illumination sources. For comparative examinations, a side-by-side color comparison of two or more backings is appropriate; otherwise, the Munsell or CIE color systems may be utilized.

4.8.3.3. Markings on the Backing Under the stereomicroscope the tape shall be examined for features such as calendaring marks, striations, dimples, and inclusions. The shapes and type of markings shall be documented.

4.8.3.4. Multiple Layer Backings Multiple layers may be present in tape backing and shall be examined to determine if multiple layers are present. There are a number of ways to cross-section tapes (e.g. hand sectioning, microtome). The multiple layers, if present, shall be characterized and then analyzed with appropriate analytical instrumentation.

4.8.3.5. Adhesive The adhesive shall be examined both macroscopically and by utilizing a stereomicroscope for color and appearance under multiple illumination sources. For comparative examinations, a side-by-side color comparison of two or more adhesives is appropriate; otherwise, the Munsell or CIE color systems may be utilized.

4.8.3.6. Reinforcement If reinforcement is present in a tape, it shall be characterized.

4.8.3.7. Duct Tape Reinforcement The three main features to examine in duct tape reinforcement are weave, yarn description, and scrim count.

4.8.3.7.1. Assess the weave of the scrim fabric under the stereomicroscope. This may require separating the adhesive from the scrim. The most frequently encountered weave patterns are weft-insertion and plain weave. Weft-insertion has chain-stitch warp yarns with texturized filaments in the fill direction. A plain weave has a one over/one under pattern.

4.8.3.7.2. The yarns can be present in the warp and fill directions. The warp and fill directions can be a combination of any of the following types of yarns:

4.8.3.7.2.1. Twisted yarns (Z- or S-twist).
4.8.3.7.2.2. Filament fibers bound by filament fiber.
4.8.3.7.2.3. Texturized filament fibers.
4.8.3.7.2.4. Straight filament fibers.
4.8.3.7.2.5. The fluorescence of the threads shall be examined using short- and/or long- wavelength illumination.

4.8.3.7.2.6. The number of filaments per bundle in the fill and warp directions may be counted. The scrim count is the warp count per inch and the fill count per inch and shall be recorded.

4.8.3.8. Strapping (Filament) Tape Reinforcement The fibers in filament tape most often consist of synthetic or glass fibers. The fibers are only in the warp direction. The number of bundles across the width of the tape may be counted, and the fluorescence of the threads shall be examined using short- and/or long- wavelength illumination.

4.8.3.8.1. Microscopical Evaluation
Tape samples shall be examined first under a stereomicroscope. Areas of the tapes that appear in their original state (i.e., not stretched out of shape and having a clean adhesive area) shall be selected for analysis. Fingerprint powders or chemicals shall be gently but thoroughly cleaned from the film backing. Characterization of inorganic materials and other tape additives are accomplished with the use of PLM. PLM is a useful adjunct to FTIR and elemental analysis. Optical properties of oriented polymers such as polypropylene (MOPP and BOPP) and polyester can also be determined. PLM is also used to evaluate and differentiate the reinforcement fibers of tapes (e.g., duct tape and strapping tape)

4.8.3.9. Fabric-Reinforced Tape (Duct tape, **Gaffer's tape, Strapping tape)**
All three layers of fabric-reinforced tape may be mounted separately for microscopy. The adhesive and reinforcement fibers shall have more discriminating microscopic features than the film backing. These methods describe the full examination; however, it is at the discretion of the examiner to choose those methods that are most suitable to the given case. Refer to the SWGMAT *Forensic Fiber Examination Guidelines*, "Microscopy of Textile Fibers" section 7.2 for mounting media recommendations.

4.8.3.9.1. Sample Preparation- Areas of the tape, free of contamination, are selected for analysis. This is best done under a stereomicroscope. Ends shall be avoided when cutting samples. The tape is initialed at the site of the cut. The film backing is separated from the adhesive and fabric. A suitable solvent (e.g., hexane) may be used if mechanical separation is not feasible. The clean film is mounted in the appropriate medium and cover-slipped.

4.8.3.9.2. Microscopical examinations of the adhesive are useful only in opaque adhesives. The adhesive is separated from backing by pinching with tweezers and cutting with a scalpel and then transferred to a microscope slide. Care shall be taken not to include fibers in this sample. Xylene or a similar solvent may be added to the adhesive sample on the slide to disperse the adhesive's rubber base. After drying, the sample is mounted in a suitable mounting medium and cover-slipped. Most minerals of PSAs can be evaluated in mounting

media having refractive indices of 1.66 and 1.55.

4.8.3.9.3. Fibers from the scrim fabric can be gently pulled and clipped from the adhesive for mounting. If necessary, the fibers can be rinsed of any adhering adhesive using hexane or other suitable solvent. The warp and fill yarns may be cotton/polyester blends. Therefore, the whole bundle shall be loosely mounted on a microscope slide in a mounting medium. Warp and fill yarn fibers are mounted separately.

4.8.3.9.4. Tape backings with some transparency may be cleaned of adhesive and mounted in a mounting medium. In duct tapes the gray color of polyethylene film backing is due to the presence of aluminum powder. Viewing mounted duct tape films under transmitted light on a comparison microscope may offer some comparative information about the density, size, and dispersion of the aluminum particles in tapes. Note that duct tape backings may be multilayered. A cross section of the duct tape backing shall be examined for physical characteristics and chemical composition. In clear and matte backings from strapping tapes and office tapes, additives are looked for and noted in plane polarized and cross polarized light. In addition, clear backings may be suitable for the methods described in the microscopy section of this test method.

4.8.3.9.5. The inorganic fillers of PSAs may be examined under transmitted plane and crossed polarized light. Mounting media with refractive indices of 1.66 and 1.55 are suitable for most mineral types that may be found in PSAs. The morphological and optical features of the different inorganic fillers can be noted. These particles are mixed with the elastomer and tackifying resin and include, but are not limited to, kaolinite, calcite, dolomite, rutile, zincite, or talc. Dispersion of adhesive samples first in xylene allows for a better assessment of these fillers. The identity of these minerals may be surmised from their optical properties along with the IR spectra and elemental composition.

4.8.3.9.6. Refer to the Fiber Test Methods for methods of determining the optical properties of the reinforcement fibers of the tape. Using these microscopic methods the following observations shall be made separately for the warp and fill fibers:

4.8.3.9.6.1. Fiber class - usually cotton or polyester.
4.8.3.9.6.2. Diameter - of each class of fibers.
4.8.3.9.6.3. Delusterant - either absent, light, medium, or heavy.
4.8.3.9.6.4. Shape - may be round, polygonal, tri-lobal, etc.
4.8.3.9.6.5. Blending - cotton may be blended with polyester.

4.8.3.10. Non-Reinforced Tape - Examinations of Oriented Films (Clear Polypropylene Packing Tape). The methods described in this section are recommended for clear packing tapes; however, they are applicable to other non-reinforced tapes with clear backings. Transparent 1/2” office

tapes and some strapping/filament tape may have oriented polymer backings. The variability in the polymer films in clear packing tape is imparted during the manufacturing process. Controlled heating, cooling, and stretching produce films with both amorphous and crystalline areas. Biaxially oriented polypropylene (BOPP) is stretched in two directions with crystalline bundles lining up along the two stretched directions. Monoaxially oriented polypropylene (MOPP) is stretched in one direction. The differences in these two types of packing tapes can be distinguished with polarized light microscopy. Within each of these subclasses of packing tapes, variances may also be noted in the extinction direction with respect to the machine direction of the tape. Differences in interference colors will reveal differences in tape film thickness. The polypropylene film of packing tapes behaves as an optically biaxial crystal. There are two perpendicular refractive indices in the plane of the film. One runs roughly in the cross direction and the other in the machine direction. The third refractive index runs normal to the plane of the film.

4.8.3.10.1. Sample Preparation - Select about an inch of tape that has both machine edges and appears to be in its original state (i.e., has not been damaged by heat, stretching, or contamination). Stick this piece directly onto a clean microscope slide, adhesive side down. An arrow noted on the mounted sample can help keep track of which direction is the machine direction. There is no need to separate the adhesive from the film for the microscopic examination of clear packing tape. Brown packing tapes with clear film backings and colored adhesive must have the adhesive removed. The cleaned film may be mounted in an appropriate medium for microscopic examinations.

4.8.3.10.2. Determination of Polypropylene Film Orientation – The surface of the clear packing tape sample is brought into focus in transmitted light at about a 100X magnification. The polars are crossed, and the extinction position is found. The stage is rotated just off extinction, and patterns are observed in the film. These patterns may be sharpened by refocusing and closing down the aperture diaphragm. A pattern of “X”s is seen in biaxially oriented tapes (BOPP) and shows the bi-directional stretching in the production process. The pattern seen in monoaxially oriented tapes (MOPP) shows the one direction of stretching. Its pattern may be hazy and show more than one interference color that streak in the one direction of the stretch. The angles of the crosshatches in the BOPP tape pattern described above may vary from one tape film to another but will be consistent throughout a roll of tape. These angles can be determined with an appropriate eyepiece reticule.

4.8.3.10.3. Determination of the Extinction Angle Relative to the Machine Direction - The machine direction of the tape relative to the extinction direction may vary from 0 to 15 degrees between different tapes. The surface of the tape is brought in focus in transmitted light. One of the

machine edges of the tape is aligned with the vertical line of the eyepiece graticule. The stage position is noted in degrees. The polars are crossed and the stage is rotated until the tape film is at its nearest full extinction. The stage position is again noted. The difference in degrees is the extinction angle relative to the machine edge. Tape samples from the same roll will show similar extinction angles.

4.8.3.10.4. Determination of the Retardation - The thickness of the polymer film in clear packing tapes can vary within manufacturers. When the birefringence of the films of the different packing tapes is the same, the variance in the interference color of the films will be a function of the thickness. Slight differences in thickness will show noticeably different interference colors. These interference colors depend only on the tape film thickness, not the total tape thickness (film + adhesive). The clear adhesive layer is isotropic and does not contribute to the interference colors. Using approximately 400X magnification, the surface of the tape is brought into focus and the polars are crossed. The interference color is noted with the stage rotated to maximum brightness (close to 45 degrees). The fast wave (lower refractive index) is found with one refractive index running roughly across the tape and the other running roughly lengthwise along the tape. The fast wave is aligned parallel to the slow wave of a quartz wedge or Berek compensator. The point of compensation is found and from this, the retardation can be calculated. The birefringence of the polypropylene tape film (BOPP) in the plane of the tape (the difference between the refractive indices of the machine and cross directions of the tape) has been reported in the range of 0.014 - 0.016. (Reference at the end of this section.)

4.8.3.10.5. Other Observations - Some clear tape films may have additives that may be visible in transmitted or polarized light and their presence is useful for comparison purposes between tapes. Some assessment of their optical properties shall be noted: size, distribution, relative interference colors, etc. Irregularities in the thickness of the tape film may be observed under crossed polars as multiple interference colors in any given field. Some tape films may not totally extinguish, or they may show undulose extinction (i.e., areas of lightness and darkness).

4.8.3.11. Microscopy of other Tape Classes - Any tape class that has inorganic fillers in the adhesive or backing, reinforcement fibers, or clear or semi-opaque film backings may lend itself to examinations described in these guidelines.

4.8.3.11.1. FTIR Evaluation
Organic and some inorganic constituents may be evaluated with the use of infrared spectroscopy. These components include the backing polymer, adhesive, elastomer, plasticizers, additives, and reinforcement fibers. The ATR (attenuated total reflectance)

accessory is particularly useful for surface analysis of the adhesive and backing. ATR is a rapid sampling method for the analysis of the tape backing and the adhesive as virtually no sample preparation is necessary. Tackifiers and/or plasticizers may be extracted from the adhesive or backing using a mild solvent such as hexane or acetone. They are subsequently analyzed in transmission by casting a thin film. For the infrared analysis of the fibers in the reinforcement component of tapes see Fiber Test Method.

4.8.3.11.1.1. Samples being compared shall be prepared and analyzed in the same manner. Sample and background scans shall be run under the same instrument conditions. A resolution of 4 cm^{-1} is optimum (one data point every 2 cm^{-1}). Higher resolution may be used.

4.8.3.11.1.2. Samples analyzed on the main bench

4.8.3.11.1.2.1. Samples prepared for analysis by transmission techniques shall be thin enough to allow infrared radiation to pass through without being over-absorbed by the sample. For transmission data that are viewed in absorbance, the sample shall be thin enough to produce a maximum absorbance of 1 absorbance unit. For transmission data viewed in % Transmittance, optimally, spectral peaks shall not fall below 10% T. This typically requires a sample thickness of approximately 5 – 10 micrometers.

4.8.3.11.1.2.2. ATR methods are used in the examination of the tape intact. Since ATR is a surface technique it is necessary to remove any extraneous material from the area to be examined. The bench ATR (single reflection) is useful for forensic casework size samples. These accessories utilize an internal reflection crystal to condense the beam onto a spot-sized sampling area. ATR is also useful in the analysis of duct tape backings for layer structure determination. The adhesive is removed, and the backing is analyzed on both sides. The compositions are then compared.

4.8.3.11.1.2.3. The use of a FTIR microscope accessory is preferred for very small samples. Spectra can be obtained from samples as small as 10-20 micrometers in diameter after flattening. The microscope attachment permits the analysis of multiple samples placed on an appropriate support material. The method affords the advantage of viewing the sample optically and choosing the most appropriate area for analysis.

4.8.3.11.1.2.3.1. Transmission measurements are commonly used because they generate spectra with fewer artifacts. However, transmission methods generally entail more sample preparation than reflection techniques. The tape sample shall be rendered thin enough not to over-absorb. Samples can be placed directly over a small aperture for analysis or placed on an appropriate salt plate. This typically requires a sample thickness of approximately 3-5 micrometers.

4.8.3.11.1.2.3.2. If samples are flattened directly on an infrared light reflecting surface (e.g., low e-glass or gold mirror), the reflection mode can be used to produce spectra mimicking double-pass transmission spectra. The technique is sometimes referred to as "transflection" or “reflection/absorption”. Samples need to be approximately half the thickness of an optimum transmission sample. The FTIR microscope can also be used in the specular reflection mode; however, it is not useful for tape unless the surface of the sample is highly reflective.

4.8.3.11.1.2.3.3. ATR objectives are available for infrared microscopes. Consistent pressure shall be applied to each sample to mitigate spectral variations. Intra-sample variations may result from sample heterogeneity; therefore, multiple samplings shall be considered.

4.8.3.11.1.2.4. Spectral comparisons of the tape component spectra can be accomplished by digital overlays with full scale expansion. Comparison of samples may be conducted with both spectra displayed in transmittance and/or absorbance. Certain information may be seen more readily in one format or the other.

4.8.3.11.1.2.4.1. There are a number of significant factors that shall be considered when comparing spectra including the presence or absence of absorption bands, and their position (wave number), shape and relative intensity. Additional sample replicates may be necessary to evaluate reproducibility of these spectral characteristics.

4.8.3.11.1.2.4.2. The presence of additional absorption bands could be from true differences between the samples or from extraneous material adhering to the tape. If extraneous material is suspected as the source of

the difference, the sample shall be cleaned or additional samples prepared. If the sample cannot be cleaned or resampled, then spectral subtraction may be an option.

4.8.3.11.1.2.4.3. For spectra to be considered indistinguishable, the position of the absorption bands shall have reasonable agreement with each other. A rule of thumb is that the positions of corresponding peaks in two or more spectra being compared shall be within a few wavenumbers of each other, depending on whether the peak is sharp or broad. For sharp absorption peaks one shall use tighter constraints and with broad peaks the variation shall be slightly greater.

4.8.3.11.1.2.4.4. For spectra to be considered indistinguishable, the shape of the absorption bands shall be consistent between comparison samples. The peak width and the symmetry of each peak shall be evaluated. Sample thickness may affect the peak width and resolution.

4.8.3.11.1.2.4.5. For spectra to be considered indistinguishable, the relative intensities of respective absorption bands shall be similar between comparison samples. The relative intensity may be affected by the heterogeneity of the sample.

4.8.3.11.1.2.5. Three possible conclusions can be reached after evaluating and comparing spectra: 1) the spectra are dissimilar, 2) the spectra are indistinguishable, or 3) inconclusive.

4.8.3.11.1.2.5.1. The spectra are dissimilar if there are one or more significant differences in the spectra. Significant differences are differences in which the spectral variation cannot be explained other than as differences between samples.

4.8.3.11.1.2.5.2. The spectra are indistinguishable when there are no significant differences in the spectra. Differences are not significant if the spectral variation can be explained

4.8.3.11.1.2.5.3. An inconclusive determination is one in which the significance of the differences cannot be completely assessed due to the constraints of sample size

and/or condition.

4.8.3.11.1.2.6. Tape is often comprised of a number of components that result in overlapping bands in the IR spectra; therefore, caution shall be exercised while evaluating the data. Not all of the components of tape can be elucidated by IR due to overlapping bands and/or relative concentration.

4.8.3.11.1.2.6.1. Tools that can assist in the characterization of the spectra include, but are not limited to, spectral libraries, flow charts, and reference materials. Most commercial spectral libraries consist of transmission (as opposed to reflection) spectra. It is desirable to use reference spectra that were obtained using the same sample preparation and collection technique.

4.8.3.11.1.2.6.2. The following components, if present, may be characterized by IR spectroscopy depending on the condition of the tape and on the concentration of the material.

4.8.3.11.1.2.6.2.1. Backing
4.8.3.11.1.2.6.2.2. Polymer Film
4.8.3.11.1.2.6.2.3. Plasticizers
4.8.3.11.1.2.6.2.4. Fillers/Extenders
4.8.3.11.1.2.6.2.5. Flame Retardants
4.8.3.11.1.2.6.2.6. Adhesive
4.8.3.11.1.2.6.2.7. Elastomer
4.8.3.11.1.2.6.2.8. Tackifiers
4.8.3.11.1.2.6.2.9. Fillers/Extenders
4.8.3.11.1.2.6.2.10. Release Coating
4.8.3.11.1.2.6.2.11. Fiber Reinforcement

4.8.3.11.1.2.6.3. For chemical identification of tape components, the positions of the absorption bands according to wavelength or wavenumber and their relative intensities shall be compared to those of known reference spectra. It is desirable to confirm the identification by other methods such as polarized light microscopy (PLM), and/or scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS).

4.8.3.11.1.2.7. Case notes shall include a description of the evidence analyzed by IR, the method of sample preparation, the analytical instrumentation used, and its operating parameters.

4.8.3.11.2. SEM/EDX Evaluation
An analytical technique utilized for the characterization of the inorganic constituents of tapes includes scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS). SEM/EDS provides elemental profiles of analyzed specimens. Additionally, SEM has imaging capabilities to evaluate surface topography of tape backings.

4.8.3.11.2.1. When samples are prepared for scanning electron microscopy, a map identifying sample location on an SEM mount can be constructed to assist in locating the sample when performing the analysis. This may be in the form of a sketch, a photomicrograph, or a captured video image and may include an index mark on the mount.

4.8.3.11.2.1.1. Samples shall first be examined with a stereomicroscope, noting size, color, structure, and any extraneous material adhering to the sample.

4.8.3.11.2.1.2. The choice of a specific method for sample preparation depends on the size, nature, and condition of the specimen, as well as the analytical objective. It may be necessary to use multiple preparation methods in order to analyze all sample characteristics.

4.8.3.11.2.1.3. In developing a strategy for analysis, the following shall be considered:

4.8.3.11.2.1.3.1. Determination of the presence of extraneous materials and a strategy for removal.

4.8.3.11.2.1.3.2. Method of attachment to a scanning electron microscopy mount.

4.8.3.11.2.1.3.3. Method(s) for producing a uniform geometry.

4.8.3.11.2.1.3.4. Determination of the presence of surface features of analytical interest.

4.8.3.11.2.1.4. If the analytical objective is to determine elemental composition, then any possible contribution from extraneous materials shall be eliminated or accounted for. For the accurate comparison of elemental composition and structure, samples shall be prepared in the same manner. It is not unusual for extraneous materials to be present on the surface of a specimen submitted for analysis, particularly on an adhesive component. Because the scanning electron microscopy method is a surface analysis, the presence of even a small amount of

this material can prevent an accurate determination and comparison of composition. Therefore, recognition and removal or abatement of this material shall be performed.

4.8.3.11.2.1.4.1. Depending on sample size and type, extraneous material may be physically removed with a brush, probe, or fine blade. Debris can also be removed from the backing surface with methanol and a cotton swab or lifted off the backing with office tape. Care shall be taken that the adhesive from the office tape does not adhere to the sample surface, which might interfere with any subsequent organic or inorganic analysis. If necessary, a fresh surface may be exposed by scraping or cutting with a fine scalpel blade.

4.8.3.11.2.1.4.2. When extraneous materials cannot be removed, these materials shall be avoided during analysis.

4.8.3.11.2.1.5. All samples to be analyzed in the scanning electron microscope shall be attached to an appropriate SEM mount. It may be useful to cool the tape below the glass transition temperature of the adhesive by submersing it in liquid nitrogen if the adhesive is going to be removed from the backing. A backing may be attached directly to a mount using the tape's own adhesive. Alternatively, if the tape's adhesive has been removed, the backing can then be attached to the mount with a mounting adhesive. This mounting adhesive may be applied as a liquid or as a double-sided tape.

4.8.3.11.2.1.6. The geometry of each sample, including flatness and take-off angle, shall be similar. Often, a backing can be pressed flat with clean glass in order to remove irregularities. The use of variable pressure on the instrument may also eliminate charging.

4.8.3.11.2.2. The SEM imaging of pressure-sensitive tape backings at moderate magnifications (75-250x) yields structural information complementary to that of traditional optical microscopical methods. It can be used to image very small striations, craters, and surface features on the backings of polymer-based tapes, such as black electrical tape and duct tape. It can be used to view the paper fibers in masking tapes, as well as show the cross-sectional structure of each of these tapes. A backscattered electron image is useful for defining structures based on the average atomic number of the matrix. SEM micrographs shall include a measuring scale or magnification

scale or both. The micrograph shall also display which signal (backscattered electron or secondary electron) was used to produce the image.

4.8.3.11.2.3. The following are suggested operating parameters for starting conditions. As the forensic scientist determines specific analytical needs, actual operating conditions may vary. A beam voltage of 20-30 KeV is an adequate compromise between the need for sufficient over-voltage necessary for efficient X-ray excitation and X-ray spatial resolution. Most of the X-ray lines produced may be displayed with an energy range of 0 to 20 KeV. The pulse processor time constant shall be set at a midrange value, which is a compromise between maximum count rate and maximum spectral resolution. The beam current shall be adjusted to yield an X-ray detector dead time of approximately 30 percent. A live time of 100-200 seconds is usually sufficient to provide reasonable counting statistics for minor peaks. Generally, changes in the suggested initial conditions are required under the following circumstances:

4.8.3.11.2.3.1. Beam voltage is increased when higher energy line excitation is required.

4.8.3.11.2.3.2. Beam voltage is decreased when greater spatial resolution is required.

4.8.3.11.2.3.3. Pulse processor time constant is lengthened when greater spectral resolution is required.

4.8.3.11.2.3.4. Pulse processor time constant is shortened when a greater count rate is required (e.g., for trace element analysis or construction of elemental distribution maps).

4.8.3.11.2.3.5. Detector to sample distance can be reduced to increase X-ray collection efficiency.

4.8.3.11.2.3.6. Spectral energy display scale is expanded when sufficient detail is not evident.

4.8.3.11.2.3.7. 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.

4.8.3.11.2.3.8. 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.

4.8.3.11.2.4. Once heterogeneity of the material is evaluated, a spectrum of the average (bulk) elemental composition of the sample is obtained. The raster shall include as much area of the sample 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 shall be collected in the same manner with the same conditions.

4.8.3.11.2.5. 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.

4.8.3.11.2.5.1. 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.

4.8.3.11.2.5.2. 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, shall be evaluated and labeled.

4.8.3.11.2.5.3. As spectral interpretation alternates between the identification of major and minor peaks, the vertical (counts) scale shall 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 shall 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 forensic scientist shall be familiar with the characteristic pattern and relative intensities of peaks of various atomic

numbers. The identification of major components is usually straightforward.

4.8.3.11.2.5.4. Following the identification of major components, lower intensity peaks and overlapped peaks are identified. The number of characteristic peaks present in a spectrum can limit minor element identification.

4.8.3.11.2.5.5. 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.

4.8.3.11.2.5.6. If identification is unequivocal, each individual peak is labeled with the corresponding elemental symbols (and X-ray line if the software permits). If the identification is probable but not absolute, the peak label shall so indicate.

4.8.3.11.2.5.7. Spectra shall 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.

4.8.3.11.2.5.8. If an automatic identification application is used, the forensic scientist shall confirm the resulting element identifications.

4.8.3.11.2.5.9. 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.

4.8.3.11.2.5.9.1. The live time count can be increased.

4.8.3.11.2.5.9.2. The processing time of the pulse processor may be increased to improve spectral resolution.

4.8.3.11.2.5.9.3. Mathematical spectral subtraction (deconvolution) methods supplied by the energy dispersive X-ray spectrometer manufacturer can be employed.

4.8.3.11.2.5.9.4. An alternative method of elemental analysis like XRF may be used.

4.8.3.11.2.5.10. 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 to be consistent with talc and kaolinite, respectively. Polarized light microscopy or infrared spectroscopy can be used to confirm the presence of some of the compounds.

4.8.3.11.2.5.11. 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.

4.8.3.11.2.6. 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 shall be sufficient to produce X-rays from all lines of analytical interest. Because an organic matrix may contain small amounts of some elements, the counting time shall be extended.

4.8.3.11.2.7. In order to compare the average composition of structures, the spectrum used for comparison shall 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 the sample permits.

4.8.3.11.2.8. Generally, comparisons are facilitated by direct spectral comparison.

4.8.3.11.2.8.1. If spectral differences are not detected, it is likely that the materials are similar in elemental composition.

4.8.3.11.2.8.2. If spectral differences are detected, it is likely that the materials are not similar in composition; however, several alternative explanations are possible and shall be evaluated.

4.8.3.11.2.8.2.1. Differences in background shape may result from dissimilar sample geometry.

4.8.3.11.2.8.2.2. Differences in the composition of major peaks may indicate that the spectra are not representative of the bulk composition of a heterogeneous sample. This could occur as a result of the analysis of a sample too small to be representative or the analysis of a raster area too small to be representative.

4.8.3.11.2.8.2.3. 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.

4.8.3.11.2.9. A conclusion regarding similarity results from the comparison of images and elemental composition of individual layers. Spectra may be critically compared by overlaying them.

4.8.3.11.2.9.1. If a comparative analysis did not demonstrate significant differences, then no differences were indicated in structure and composition within the limits of the analytical capability of scanning electron microscopy/energy dispersive X-ray spectroscopy.

4.8.3.11.2.9.2. If ratio differences between peaks exist, it can be concluded that these differences may result from either actual differences in the bulk composition of the materials

or from the analysis of a small sample (or area) whose chemistry is not representative of the bulk composition of a heterogeneous sample. The latter shall only be concluded following an extensive investigation of the heterogeneity of the known samples and demonstration that the range of variation present in one sample encompasses that observed in the other sample.

4.8.3.11.2.9.3. If there are no differences in major peak ratios but there are differences in minor/trace peaks, it can be concluded that no differences in major elemental constituents are indicated, although some differences in the bulk composition are evident. For example, if the specimen was a fragment and unable to be adequately cleaned, a small amount of foreign material may have been present during the analysis. Consequently, some of the minor elemental peaks present in the spectrum may have been produced from elements in the foreign material and not from elements in the questioned material. Equally so, the observed differences may be due to actual differences in the composition of the samples. Therefore, with respect to the elemental composition of these samples, an inconclusive result for this technique is indicated.

4.8.3.11.2.9.4. If, after taking into consideration the previous section a comparative analysis demonstrates significant differences between samples regarding structure and composition, it then can be concluded that the samples are different.

4.8.3.11.2.10. Case notes shall include a copy of the instrumental data. Case notes shall also include a description of the evidence analyzed by SEM, the method of sample preparation, the analytical instrumentation used, and its operating parameters. The case notes shall also include a statement or data confirming system calibration.

4.9. Records: Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics.

4.9.1. Preliminary examination of tape construction shall include its general appearance macroscopically and under a stereomicroscope, including any adhering matter. For all pressure sensitive tapes, document and record any physical damage (e.g., worn, cut, torn, frayed).

4.9.2. The case record shall include the instrumental data. All hard copies shall include a unique sample designation, the operator's name/initials, and the date of analysis. Case notes shall also include a description of the evidence analyzed, the method of sample preparation, the analytical instrumentation used, and its

operating parameters.

4.9.3. Physical edge matches for tape will follow the procedures as described in the Physical Edge Match Comparison Test Method.

4.10. Interpretations of Results:

4.10.1. Typically, a tape examination involves the comparison of samples to determine if they could share a common origin. The goal is to determine if any significant differences exist between the samples. The physical and chemical characteristics are evaluated to associate the questioned and known sample. If a significant difference in a characteristic exists the samples are excluded as having a common origin. If the examination doesn't reveal any significant differences it is concluded that the samples could share a common origin. A limited association can be reported due to the condition/size of one of the samples (i.e. only adhesive residue left to examine and compare). The evaluation of tapes for class characteristics can associate known and questioned tapes to a group but not to a single, individual source. A true physical end match of two tape ends can provide individualizing characteristics that associate the two tapes to one another to the exclusion of all other tapes.

4.11. Report Writing:

4.11.1. If significant differences are found in the tape characteristics, then the report shall state that the tapes are not consistent and did not come from the same source.

4.11.2. If no significant differences are found in the tape characteristics, then the report shall state that the tapes are consistent and could have come from the same source or another source with the same characteristics.

4.11.3. In rare circumstances there are differences in characteristics of the tapes and they can still be evaluated due to the difference in the present conditions of the tapes. In this circumstance it shall be stated in the report.

4.11.4. If a physical end match exists between two tapes, then a more positive association exists. The comparison of the characteristics of the two tapes shall still be conducted and noted in the report of non-conclusive physical edge matches.

4.12. References:

4.12.1. SWGMAT Guideline for the Forensic Examination of Pressure-Sensitive Tapes [Online] (October 2008) Available: http://www.fbi.gov/hq/lab/fsc/backissu/Oct2008/index.htm

4.12.2. SWGMAT Guideline for Assessing Physical Characteristics in Forensic Tape Examinations

4.12.3. SWGMAT Guideline for Using FTIR in Forensic Tape Examinations

4.12.4. SWGMAT Guideline for Using Light Microscopy in Forensic Examination of Tape Components

4.12.5. SWGMAT Guideline for Using Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy in Forensic Tape Examinations

4.12.6. SWGMAT Forensic Fiber Examination Guideline [Online] (April 1999) Available: http://www.fbi.gov/hq/lab/fsc/backissu/april1999/index.htm

4.12.7. SWGMAT Trace Recovery Guidelines [Online] (October 1999). Available: http://www.fbi.gov/hq/lab/fsc/backissu/oct1999/index.htm

4.12.8. Johnston, John, *Pressure Sensitive Adhesive Tapes - A Guide to their Function, Design, Manufacture and Use.* Pressure Sensitive Tape Council, Northbrook, IL. 2003

4.12.9. Blackledge, Robert (Smith, Jenny) *Forensic Analysis on the Cutting Edge – New Methods for Trace Evidence Analysis*, Chapter 12 – Forensic Examination of Pressure Sensitive Tapes

4.12.10. Snodgrass, H., *Duct Tape Analysis as Trace Evidence*. Proceedings of the International Symposium on Trace Evidence, FBI Academy, Quantico, VA. June 1991, p69-73

4.12.11. Keto, R.O., *Forensic Characterization of Black Polyvinyl Chloride Electrical Tape*. Proceedings of the International Symposium on the Analysis and Identification of Polymers, FBI Academy, Quantico, VA. July 31 – Aug 2, 1984, p137-143

4.12.12. Smith, J., *The Forensic Value of Duct Tape Comparisons*. Midwestern Association of Forensic Scientists Newsletter, Vol 27 (1) Jan 1998, p28-33, REPRINTED in CACNews 3rd Quarter, 1998 p14-17

4.12.13. Smith, J., Weaver, R., "*PLM Examinations of Clear Polymer Films",* The Microscope, Volume 52, 3rd and 4th quarter, 2004. p113-118

4.12.14. Hobbs, A.L., et. al., *A New Approach for the analysis of duct Tape Backings* Forensic Science Communications, January 2007, Vol 9, number 1

5. Paint Examinations

5.1. Scope:

This test method is for the analysis, examination, and comparison of evidentiary paint. The scope of this type of evidence includes but is not limited to automotive, industrial, and residential type surface coatings. Typically, a paint examination involves the comparison of samples to determine if they could share a common origin. The goal is to determine if any significant differences exist between the samples. The evaluation of paint for class characteristics can associate known and questioned paint to a group but not to a single, individual source. A physical end match of two paints provides individualizing characteristics that associate the two paint chips to one another. The methods and practices described have been published, peer-reviewed, and are generally accepted within the forensic science community.

5.2. Precautions/Limitations:

5.2.1. The analysis and comparison of paint evidence in the forensic science laboratory can provide valuable information due to the variability of paint. However, some automotive paint exhibits more variability than others. Because of the ever-changing markets, suppliers, and economic factors, it is not practical to establish the statistical probability that a given sample would have the same physical and chemical characteristics as a randomly selected paint sample.

5.2.2. Paint samples submitted as evidence may have been subjected to physical damage. Sometimes only a smear of one of the layers may be present. The strength of an association between a partial paint sample and a more pristine sample could be weakened depending upon the degree of damage. In some cases, the damaged paint may be unsuitable for comparison purposes. These changes may limit the information obtained from the analyses.

5.2.3. The paint sample size does not always allow for the full range of examinations. The examinations and analyses that are performed shall be documented in the forensic scientist's case record.

5.2.4. The information available from a heterogeneous specimen may diminish as its size is reduced and its condition degrades. The smaller a specimen, the less valuable it may become for an association. As sample size is reduced, it may no longer be representative of the original material.

5.2.5. Automotive paint that is not similar to a known sample or that does not have a known sample submitted for comparison purposes may be run with the Paint Data Query (PDQ). This search is described in another section. The database does not have every single paint system that is available. It also does not include repaint systems or utility paint systems. Informational data derived from PDQ shall be evaluated appropriately.

5.2.6. An item where paint particles may be present shall be packaged in a sealed and labeled container in such a way that paint is not lost, transferred, or compromised

for analysis. When individual paint particles are recovered, the paint shall be appropriately packaged (i.e. put into a paper bindle then placed into a properly sealed and identified pill box). Known paint samples shall be submitted with each case and packaged similarly.

5.3. Related Information:

5.3.1. Appendix 1 Worksheets
5.3.2. Appendix 2 Abbreviations
5.3.3. Appendix 3 Definitions
5.3.4. Test Method for Physical Comparisons

5.4. Instruments:

5.4.1. Stereomicroscope – a microscope which gives a "three dimensional view" capable of examining samples up to a magnification of approximately 70x. Stereomicroscopical observations (e.g., layer sequence, colors, layer thickness, and texture) provide initial and discriminating information for paint comparisons.

5.4.2. Polarized Light Microscope (PLM) - a microscope used in the characterization of inorganic materials.

5.4.3. Fourier Transform Infrared (FTIR) Spectrometer– an analytical instrument capable of analyzing the molecular vibrations of the chemical components of paint. Organic and some inorganic constituents may be evaluated with the use of infrared spectroscopy. The components of individual layers can be evaluated with the Microscope FTIR.

5.4.4. Scanning Electron Microscope with energy dispersive spectroscopy (SEM/EDS) – an analytical instrument capable of chemically analyzing paint components and to magnify samples. It can be utilized for the characterization of the inorganic constituents of individual paint layers.

5.4.5. Pyrolysis-Gas Chromatography Mass Spectrometry (Py-GC/MS) – an instrument capable of thermally breaking down, separating, and then analyzing a paint sample.

5.4.6. Microtome – an instrument for cutting thin cross sections of specimens. It can be used to make cross sections of paint samples.

5.5. Reagents/Materials: Chemical solvents are used (i.e. chloroform, acetone, and diphenylamine reagent) in the analyses of paint.

5.5.1. Microscope slides
5.5.2. Microscope cover slips
5.5.3. Mounting medium

5.5.4. Scalpel or razor blade
5.5.5. Low e^- microscope slide

5.5.6. SEM stubs
5.5.7. Liquid nitrogen
5.5.8. Roller knife
5.5.9. Disposable gloves
5.5.10. Laboratory coat
5.5.11. Spot plate
5.5.12. Tweezers
5.5.13. Probes

5.6. Hazards/Safety:

5.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as "Biohazard" shall be handled with gloved hands during the entire examination process. Items handled with gloves shall be disinfected prior to handling with bare hands.

5.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling occurs first, the forensic scientist shall still take precautions during examination for potential additional DNA analysis in the future. During the examination the forensic scientist shall also wear a disposable face mask. Gloves shall be changed after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

5.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to minimize additional fingerprints, or obliterating existing fingerprints.

5.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

5.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS's shall be consulted.

5.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

5.7. Reference Materials/Controls/Calibration Checks:

5.7.1. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

5.7.2. Questioned and known samples shall be analyzed under similar conditions.

5.7.3. The Py-GC/MS shall be performance checked as required in Appendix 7.

5.7.4. The SEM Instrument shall be performance checked as required in Appendix 5.

5.7.5. The FTIR Instrument shall be performance checked as required in Appendix 4.

5.7.6. In the event that performance checks are found to be unsatisfactory, the instrument shall be taken out-of-service and measures taken to restore the instrument to proper working order.

5.7.6.1. Any instrument that is out-of-service shall be visibly marked.

5.7.6.2. When an instrument is taken out-of-service for maintenance and/or repair, performance checks shall be performed prior to resuming casework on that instrument.

5.7.6.3. An infrequently used instrument may be placed in an “Inactive” status and the normal verification procedures may be suspended. Normal quality control procedures shall be resumed prior to use in casework analysis.

5.8. Procedures/Instructions:
This section provides an overview of suggested flow of analytical techniques to be utilized for the analysis of paint. The exact selection and order of methods depends upon the sample size and/or condition.

5.8.1. Initial Evaluation and Handling Due to concerns with the handling of paint as physical evidence, each case shall be evaluated to develop appropriate procedures concerning the sampling size, collection, preservation, and order of examinations.

5.8.1.1. Different forensic disciplines may be called upon to examine the same item of evidence. The order in which the examinations shall be conducted needs to be resolved on a case-by-case basis. The order of examinations shall be selected and conducted so as to preserve the most transient evidence and provide the greatest discrimination and most valuable information. Forensic Scientist may make the submitting agency aware of the effects that some disciplines’ processing and examinations may have upon other specific examination requests. If another discipline is chosen before the paint examination, obtaining an unadulterated representative sample shall be considered.

5.8.1.2. When the amount of a paint specimen present for comparison purposes is adequate in size—as deemed by the forensic scientist —analysis may

include several different analytical techniques. Nondestructive methods should be exhausted before subjecting the sample to any destructive tests.

5.8.1.3. If the initial tests are not exclusionary, the examination shall proceed with additional tests that are selected on the basis of their potential for use in discriminating the questioned and known samples. The size, condition, and type of paint present will dictate the selected analytical scheme.

5.8.1.3.1. Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics. Preliminary examination shall include its general appearance macroscopically and under a stereomicroscope, including any physical damage.

5.8.1.4. Ideally the item of evidence shall be preserved in a manner that does not interfere with future testing. All procedures shall be conducted in such a manner to ensure that no cross-contamination occurs. The item shall be photographed or described prior to conducting any destructive analyses in order to provide documentation of original condition. The images shall not be considered items of evidence.

5.8.1.5. For hit-n-run cases involving personal injury, the victim's clothing shall be processed in the manner outlined below:

5.8.1.5.1. Clean off the examination surface and spread a clean piece of examination paper out on the examination surface.

5.8.1.5.2. Open the container, avoiding the breakage of existing seals when possible. Remove the evidence and mark it with the case number, item number, and initials.

5.8.1.5.3. A careful examination is conducted at this time. The color and location of paint smears and other stains is noted. Visible paint chips are removed and transferred to evidence boxes.

5.8.1.5.4. Clothing items should then be scraped off with a spatula over the paper in order to dislodge paint chips, which may be imbedded in the fabric. Pocket linings and hems deserve particular attention in the scraping process. Clothing articles are then shaken to dislodge any remaining debris. The accumulated debris is transferred from the paper to an evidence box.

5.8.1.5.5. Using a stereomicroscope, note the materials present in the debris. Note the relative number and color of paint particles present.

5.8.1.6. The initial examination may be the final step in an analysis if no questioned paint is located, or only exclusionary samples are identified in comparison to the known sample.

5.8.2. Physical Edge Match Evaluation

Physical end matches can provide individualizing associations. When conducting comparison examinations between two or more paint specimens, the edges shall be carefully examined for possible physical end matches. A physical end match is defined as two separate pieces of paint that physically fit together demonstrating that the two pieces were once one continuous piece. This type of association is the most compelling type of association.

5.8.2.1. Observe the specimens to determine if a physical association is possible. For finer detail, a stereomicroscope shall be used to examine the chips.

5.8.2.2. Depending on the paint chips, manufacturing marks or other continuous surface features may be present across fractured edges and may provide additional points of comparison.

5.8.2.3. Any physical associations shall be documented with descriptive notes. Physical associations between specimens shall be imaged. The imaging method shall be dimensionally accurate and include a measuring scale when possible.

5.8.2.4. Any/all physical edge match associations between a questioned specimen and a known specimen shall be verified by another qualified forensic scientist as outlined in the Physical Edge Match Test Method.

5.8.3. Physical Characteristic Evaluation

Macroscopic and stereomicroscopical observations (e.g., colors, layer sequences, layer thicknesses, and layer texture) provide initial and discriminating information for paint comparisons. Physical characterization of paint specimens is the initial step of a comprehensive forensic paint analysis. The number of layers, the layer sequence, the layer colors, relative thickness of the layers, and the texture of the paint varies and therefore is a useful characteristic for forensic examinations. Visual characteristics are the quickest, most discriminating, and least invasive examinations. If no significant physical differences are found between samples, instrumental analyses are warranted.

5.8.3.1. The stereomicroscopical characteristics are sometimes apparent without sample preparation. For a more definitive evaluation, a manual or microtome cross section can employed.

5.8.3.2. General Characteristics The following characteristics shall be documented when applicable:

5.8.3.2.1. Color of each layer
5.8.3.2.2. Sequence of each layer
5.8.3.2.3. Texture of each layer
5.8.3.2.4. Relative thickness of each layer
5.8.3.2.5. Number of layers present
5.8.3.2.6. Type and characteristics of flake present

5.8.3.3. Paint Comparison The question paint chip is sometimes large enough for a side-by-side comparison to the standard. If possible, for comparative examinations the paint shall be examined both macroscopically and by using a stereomicroscope for color, texture, and appearance under multiple illumination sources. Subtle differences in color, pigment appearance, surface details, inclusions, flake size and distribution, and texture are more readily seen in a side-by-side comparison in the same field of view.

5.8.3.4. Markings on the Paint's Surface Layer Under the stereomicroscope the paint shall be examined for features such as marks, striations, dimples, and inclusions on the layer of paint that is attached to the substrate. The shapes and type of markings shall be documented.

5.8.3.5. Multiple Layer Paint Chips Multiple layers may be present in paint chips and the chip shall be examined to determine if multiple layers are present. There are a number of ways to cross-section paint (e.g. hand sectioning, microtome). The multiple layers, if present, shall be characterized and then analyzed with appropriate analytical instrumentation.

5.8.4. Microchemical Evaluation

Paint samples can be examined for their chemical reaction to various solvents. Differing pigments and binders that are otherwise similar in visual appearance are sometimes discriminated. This is a destructive technique and shall be used only when a significant amount of question paint exists. The reactions shall be evaluated first on the known paint samples. Different paints can have different reactions to various solvents.

5.8.4.1. Reactions of the paint chip include softening, dissolution, curling, swelling, pigment filler effervescence, flocculation, or wrinkling. The results of this testing is difficult to quantify. The testing is primarily used for general classification and comparison.

5.8.4.2. Place a small known paint chip in one of the wells of a spot plate.

5.8.4.2.1. Add one drop of acetone. Observe the reaction of the paint chip.

5.8.4.2.2. Add one drop of chloroform. Observe the reaction of the paint chip.

5.8.4.2.3. Add one drop of diphenylamine reagent. Observe the reaction of the paint chip.

5.8.4.3. Microchemical tests are applied to the known paint chip first in order to evaluate their efficacy to a specific case. When probative then the testing shall commence on the question paint sample only when sufficient questioned paint samples are available.

5.8.5. FTIR Evaluation

Organic and some inorganic constituents may be evaluated with the use of infrared

spectroscopy. It can be used to evaluate binders, pigments, and additives in various types of coating materials. Since many of the samples have limited size, the microscope attachment is often needed to analyze the samples. Transmittance and reflectance techniques can be employed.

5.8.5.1. Samples being compared shall be prepared and analyzed in the same manner. Sample and background scans shall be run under the same instrument conditions. A resolution of 4 cm^{-1} (one data point every 2 cm^{-1}) is optimum and the minimum resolution acceptable. Higher resolution may be used.

5.8.5.2. Attenuated Total Reflectance (ATR) methods are used in the examination of the paint intact. Since ATR is a surface technique it is necessary to remove any extraneous material from the area to be examined. The bench ATR (single reflection) is useful for large paint samples that have a single layer. These accessories utilize an internal reflection crystal to condense the beam onto a spot-sized sampling area.

5.8.5.3. Samples analyzed with the FTIR Microscope accessory. The use of a microscope accessory is preferred for very small samples. Spectra can be obtained from samples as small as 10-20 micrometers in diameter after flattening. The microscope attachment permits the analysis of multiple samples placed on an appropriate support material. The method affords the advantage of viewing the sample optically and choosing the most appropriate area for analysis.

5.8.5.4. Transmission measurements generate spectra with fewer artifacts. However, transmission methods generally entail more sample preparation than reflection techniques. The paint sample shall be rendered thin enough not to over-absorb. This typically requires a sample thickness of approximately 3-5 micrometers.

5.8.5.5. If samples are flattened directly on an infrared light reflecting surface (e.g., low e^- glass or gold mirror), the reflection mode can be used to produce spectra mimicking double-pass transmission spectra. The technique is sometimes referred to as "transflection" or “reflection/absorption”. The FTIR microscope can also be used in the specular reflection mode.

5.8.5.6. ATR objectives are available for infrared microscopes. Consistent pressure shall be applied to each sample to mitigate spectral variations. Intra-sample variations may result from sample heterogeneity; therefore, multiple samplings shall be considered.

5.8.5.7. Spectral comparisons of the paint component spectra can be accomplished by digital overlays with full scale expansion. Comparison of samples may be conducted with both spectra displayed in transmittance and/or absorbance. Certain information may be seen more readily in one format

or the other.

5.8.5.8. There are a number of significant factors that shall be considered when comparing spectra including the presence or absence of absorption bands, and their position (wavenumber), shape and relative intensity. Additional sample replicates may be necessary to evaluate reproducibility of these spectral characteristics.

5.8.5.9. The presence of additional absorption bands could be from true differences between the samples or from extraneous material adhering to the paint. If extraneous material is suspected as the source of the difference, the sample shall be cleaned or additional samples prepared. If the sample cannot be cleaned or resampled, then spectral subtraction may be an option.

5.8.5.10. For spectra to be considered indistinguishable, the position of the absorption bands shall have reasonable agreement with each other. The positions of corresponding peaks in two or more spectra being compared shall be within a few wavenumbers of each other, depending on whether the peak is sharp or broad. For sharp absorption peaks one shall use tighter constraints and with broad peaks the variation shall be slightly greater.

5.8.5.11. For spectra to be considered indistinguishable, the shape of the absorption bands shall be consistent between comparison samples. The peak width and the symmetry of each peak shall be evaluated. Sample thickness may affect the peak width and resolution.

5.8.5.12. For spectra to be considered indistinguishable, the relative intensities of respective absorption bands shall be similar between comparison samples. The relative intensity may be affected by the heterogeneity of the sample.

5.8.5.13. Three possible conclusions can be reached after evaluating and comparing spectra: 1) the spectra are dissimilar, 2) the spectra are indistinguishable, or 3) inconclusive.

5.8.5.13.1. The spectra are dissimilar if there are one or more significant differences in the spectra. Significant differences are differences in which the spectral variation cannot be explained other than as differences between samples.

5.8.5.13.2. The spectra are indistinguishable when there are no significant differences in the spectra. Differences are not significant if the spectral variation can be explained

5.8.5.13.3. An inconclusive determination is one in which the significance of the differences cannot be completely assessed due to the constraints of

sample size and/or condition.

5.8.5.14. Paint is often comprised of a number of layers that can't be completely isolated which can result in overlapping bands in the IR spectra; therefore, caution shall be exercised while evaluating the data.

5.8.5.15. Tools that can assist in the characterization of the spectra include, but are not limited to, spectral libraries, flow charts, and reference materials. Most commercial spectral libraries consist of transmission (as opposed to reflection) spectra. It is desirable to use reference spectra that were obtained using the same sample preparation and collection technique.

5.8.5.16. The case record shall include a description of the evidence analyzed by IR, the method of sample preparation, the analytical instrumentation used, and its operating parameters.

5.8.6. SEM/EDS Evaluation

An analytical technique utilized for the characterization of the inorganic constituents of paint includes scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS). SEM/EDS provides elemental profiles of analyzed specimens. Additionally, SEM has imaging capabilities to evaluate surface topography of paint.

5.8.6.1. When samples are prepared for scanning electron microscopy, a map identifying sample location on an SEM mount can be constructed to assist in locating the sample when performing the analysis. This may be in the form of a sketch, a photomicrograph, or a captured video image and may include an index mark on the mount.

5.8.6.2. The choice of a specific method for sample preparation depends on the size, nature, and condition of the specimen, as well as the analytical objective. It may be necessary to use multiple preparation methods in order to analyze all sample layers.

5.8.6.3. If the analytical objective is to determine elemental composition, then any possible contribution from extraneous materials shall be eliminated or accounted for. For the accurate comparison of elemental composition and structure, samples shall be prepared in the same manner. Because the scanning electron microscopy method is a surface analysis, the presence of even a small amount of this material can prevent an accurate determination and comparison of composition. Therefore, recognition and removal or abatement of this material shall be performed.

5.8.6.4. Depending on sample size and type, extraneous material may be physically removed with a brush, probe, or fine blade. If necessary, a fresh surface may be exposed by scraping or cutting with a fine scalpel blade.

5.8.6.5. When extraneous materials cannot be removed, these materials shall be avoided during analysis if possible.

5.8.6.6. All samples to be analyzed in the scanning electron microscope shall be attached to an appropriate SEM mount. The geometry of each sample, including flatness and take-off angle, shall be similar.

5.8.6.7. The SEM imaging of paint at moderate magnifications (75-250x) yields structural information complementary to that of traditional optical microscopical methods. It can be used to image very small striations, craters, and surface features on the backings of paint. A backscattered electron image is useful for defining layer structure based on the average atomic number of the matrix. SEM micrographs shall include a measuring scale or magnification scale or both. The micrograph shall also display which signal (backscattered electron or secondary electron) was used to produce the image.

5.8.6.8. The following are suggested operating parameters for starting conditions. As the forensic scientist determines specific analytical needs, actual operating conditions may vary. A beam voltage of 20-30 KeV is an adequate compromise between the need for sufficient over-voltage necessary for efficient X-ray excitation and X-ray spatial resolution. Most of the X-ray lines produced may be displayed with an energy range of 0 to 20 KeV. The pulse processor time constant shall be set at a midrange value, which is a compromise between maximum count rate and maximum spectral resolution. The beam current shall be adjusted to yield an X-ray detector dead time of approximately 30 percent. A live time of 100-200 seconds is usually sufficient to provide reasonable counting statistics for minor peaks. Generally, changes in the suggested initial conditions are required under the following circumstances:

5.8.6.8.1. Beam voltage is increased when higher energy line excitation is required.

5.8.6.8.2. Beam voltage is decreased when greater spatial resolution is required.

5.8.6.8.3. Pulse processor time constant is lengthened when greater spectral resolution is required.

5.8.6.8.4. Pulse processor time constant is shortened when a greater count rate is required (e.g., for trace element analysis or construction of elemental distribution maps).

5.8.6.8.5. Detector to sample distance can be reduced to increase X-ray collection efficiency.

5.8.6.8.6. Spectral energy display scale is expanded when sufficient detail is not evident.

5.8.6.8.7. 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.

5.8.6.8.8. 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.

5.8.6.9. Once heterogeneity of the material is evaluated, a spectrum of the average (bulk) elemental composition of the sample is obtained. The raster shall include as much area of the sample 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 shall be collected in the same manner with the same conditions.

5.8.6.10. 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.

5.8.6.10.1. 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.

5.8.6.10.2. 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, shall be evaluated.

5.8.6.10.3. As spectral interpretation alternates between the identification of major and minor peaks, the vertical (counts) scale shall 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 shall 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 forensic scientist shall be familiar with

the characteristic pattern and relative intensities of peaks of various atomic numbers. The identification of major components is usually straightforward.

5.8.6.10.4. Following the identification of major components, lower intensity peaks and overlapped peaks are identified. The number of characteristic peaks present in a spectrum can limit minor element identification.

5.8.6.10.5. 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.

5.8.6.10.6. If identification is unequivocal, each individual peak is labeled with the corresponding elemental symbols (and X-ray line if the software permits). If the identification is probable but not absolute, the peak label shall so indicate.

5.8.6.10.7. Spectra shall 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.

5.8.6.10.8. If an automatic identification application is used, the forensic scientist shall confirm the resulting element identifications by visually evaluating the spectra.

5.8.6.10.9. 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.

5.8.6.10.9.1. The live time count can be increased.

5.8.6.10.9.2. The processing time of the pulse processor may be increased to improve spectral resolution.

5.8.6.10.9.3. Mathematical spectral subtraction (deconvolution) methods supplied by the energy dispersive X-ray spectrometer

manufacturer can be employed.

5.8.6.10.10. 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 paint sample 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 to be consistent with talc and kaolinite, respectively. Polarized light microscopy or infrared spectroscopy can be used to confirm the presence of some of the compounds.

5.8.6.10.11. 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.

5.8.6.10.12. Analysis of a substance that is primarily organic 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 shall be sufficient to produce X-rays from all lines of analytical interest. Because an organic matrix may contain small amounts of some elements, the counting time shall be extended.

5.8.6.10.13. In order to compare the average composition of structures, the spectrum used for comparison shall 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 the sample permits.

5.8.6.11. Generally, comparisons are facilitated by direct spectral comparison.

5.8.6.11.1. If spectral differences are not detected, it is likely that the materials are similar in elemental composition.

5.8.6.11.2. If spectral differences are detected, it is likely that the materials are not similar in composition; however, several alternative explanations

are possible and shall be evaluated.

5.8.6.11.3. Differences in background shape may result from dissimilar sample geometry.

5.8.6.11.4. Differences in the composition of major peaks may indicate that the spectra are not representative of the bulk composition of a heterogeneous sample. This could occur as a result of the analysis of a sample too small to be representative or the analysis of a raster area too small to be representative.

5.8.6.11.5. 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.

5.8.6.11.6. A conclusion regarding similarity results from the comparison of images and elemental composition of individual layers. Spectra may be critically compared by overlaying them.

5.8.6.11.7. If a comparative analysis did not demonstrate significant differences, then no differences were indicated in structure and composition within the limits of the analytical capability of scanning electron microscopy/energy dispersive X-ray spectroscopy.

5.8.6.11.8. If ratio differences between peaks exist, it can be concluded that these differences may result from either actual differences in the bulk composition of the materials or from the analysis of a small sample (or area) whose chemistry is not representative of the bulk composition of a heterogeneous sample. The latter shall only be concluded following an extensive investigation of the heterogeneity of the known samples and demonstration that the range of variation present in one sample encompasses that observed in the other sample.

5.8.6.11.9. If there are no differences in major peak ratios but there are differences in minor/trace peaks, it can be concluded that no differences in major elemental constituents are indicated, although some differences in the bulk composition are evident. For example, if the specimen was a fragment and unable to be adequately cleaned, a small amount of foreign material may have been present during the analysis. Consequently, some of the minor elemental peaks present in the spectrum may have been produced from elements in the foreign material and not from elements in the questioned material. Equally so, the observed differences may be

due to actual differences in the composition of the samples. Therefore, with respect to the elemental composition of these samples, an inconclusive result for this technique is indicated.

5.8.6.11.10. If, after taking into consideration the previous section a comparative analysis demonstrates significant differences between samples regarding structure and composition, it then can be concluded that the samples are different.

5.8.6.12. Case record shall include a copy of the instrumental data. Case notes shall also include a description of the evidence analyzed by SEM, the method of sample preparation, the analytical instrumentation used, and its operating parameters. The case notes shall also include a statement or data verifying system quality assurance check.

5.9. Records: Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics.

5.9.1. Preliminary examination of paint shall include its general appearance macroscopically and under a stereomicroscope, including any adhering matter.

5.9.2. The case record shall include the instrumental data. All hard copies shall include a unique sample designation, the operator's name/initials, and the date of analysis. Case notes shall also include a description of the evidence analyzed, the method of sample preparation, the analytical instrumentation used, and its operating parameters.

5.10. Interpretations of Results:

5.10.1. Typically, a paint examination involves the comparison of samples to determine if they could share a common origin. The goal is to determine if any significant differences exist between the samples. The physical and chemical characteristics are evaluated to associate the questioned and known sample. If a significant difference in a characteristic exists the samples are excluded as having a common origin. If the examination doesn't reveal any significant differences it is concluded that the samples could share a common origin. A limited association can be reported due to the condition/size of one of the samples (i.e. paint smears, paint that does not allow for separation for analysis). The evaluation of paint for class characteristics can associate known and questioned paint to a group but not to a single, individual source. A true physical end match of two paints can provide individualizing characteristics that associate the two paints to one another to the exclusion of all other paints.

5.11. Report Writing:

5.11.1. If significant differences are found in the paint characteristics, then the report shall state that the paints are not consistent and did not come from the same source.

5.11.1.1. In some circumstances the difference in characteristics of the paints can still be evaluated due to the difference in the present conditions of the paints. In this circumstance it shall be stated in the report.

5.11.2. If no significant differences are found in the paint characteristics, then the report shall state that the paints are consistent and could have come from the same source or another source with these same characteristics.

5.11.3. If a physical end match exists between two paints, then a more positive association exists. The comparison of the characteristics of the two paints shall still be conducted and noted in the report of non-conclusive physical edge matches.

5.12. References:

5.12.1. ASTM E1610 Standard Guide for Forensic Paint Analysis and Comparison

5.12.2. ASTM E2808 Standard Guide for Microspectrophotometry and Color Measurement in Forensic Paint Analysis

5.12.3. ASTM E2809 Standard Guide for Using Scanning Electron Microscopy/XRay Spectrometry in Forensic Paint Examinations

5.12.4. ASTM E2937 Standard Guide for Using Infrared Spectroscopy in Forensic Paint Examinations

5.12.5. SWGMAT Trace Recovery Guidelines [Online] (October 1999). Available: http://www.fbi.gov/hq/lab/fsc/backissu/oct1999/index.htm

5.12.6. Crown, D. A. *The Forensic Examination of Paints and Pigments.* Charles C. Thomas, Springfield, Illinois, 1968.

5.12.7. Thornton, J. I. Forensic paint examination. In: *Forensic Science Handbook* (Vol. 1). Saferstein, R. (ed.). Prentice-Hall, New Jersey, 1982, pp. 529-571.

5.12.8. Stoecklein, W. Forensic science: Paints, varnishes and lacquers. In: *Encyclopedia of Analytical Science.* Academic Press, New York, 1995, pp. 1625-1635.

5.12.9. Laing, D. K., Locke, J., Richard, R. A., and Wilkerson, J. M. The examination of paint films and fibers as thin-sections, *Microscope* (1987) 35:233-248.

5.12.10. Allen, T. J. Modification of sample mounting procedures and microtome equipment for paint sectioning, *Forensic Science International* (1991) 5:93-100.

5.12.11. McCrone, W. C. Application of particle study in art and archeology conservation and authentication. In: *The Particle Atlas* (Vol. V). Ann Arbor Science, Ann Arbor, Michigan, 1979, pp. 1402-1413.

5.12.12. Kilbourn, J. H. and Marx, R. B. Polarized light microscopy of extenders in structural paints: Forensic applications, *Microscope* (1994) 42:167-175.

5.12.13. O'Neill, L. A. Analysis of paints by infrared spectroscopy, *Medicine, Science and the Law* (1967) 7:145-147.

5.12.14. Suzuki, E. M. Forensic applications of infrared spectroscopy. In: *Forensic Science Handbook* (Vol. 3). Saferstein, R. (ed.). Regents/Prentice Hall, Englewood Cliffs, New Jersey, 1993, pp. 71-195.

5.12.15. Ryland, S. G. Infrared microspectroscopy of forensic paint evidence. In: *Practical Guide to Infrared M*

5.12.16. Wilkinson, J. M., Locke, J., and Laing, D. K. The examination of paints at thin-sections using visible microspectrophotometry and Fourier transform infrared microscopy, *Forensic Science International* (1988) 38:43-52.

5.12.17. Allen, T. J. Paint sample presentation for Fourier transform infrared microscopy, *Vibrational Spectroscopy* (1992) 3:217-37.

5.12.18. Bartick, E. G. and Tungol, M. W. Infrared microscopy and its forensic applications. In: *Forensic Science Handbook* (Vol. 3). Saferstein, R. (ed.). Regents/Prentice Hall, Englewood Cliffs, New Jersey, 1993, pp. 196-252.

5.12.19. 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* (1983) 16:163-173.

5.12.20. Suzuki, E. M. Infrared spectra of U.S. automobile original topcoats (1974-1989): Differentiation and identification based on acrylonitrile and ferrocyanide C=N stretching absorptions, *Journal of Forensic Sciences* (1996) 41:376-392.

5.12.21. Suzuki, E. M. Infrared spectra of U.S. automobile original topcoats (1974-1989): Identification of some topcoat inorganic pigments using an extended range (4000-220 cm-1) Fourier transform spectrometer, *Journal of Forensic Sciences* (1996) 41:393-406.

5.12.22. Suzuki, E. M. and Marshall, W. P. Infrared spectra of U.S. automobile original topcoats (1974-1989): In situ identification of some organic pigments used in yellow, orange, red and brown nonmetallic and brown metallic finishes - benzimidazolones, *Journal of Forensic Sciences* (1997) 42:619-648.

5.12.23. Infrared Spectroscopy Atlas Working Committee, *An Infrared Spectroscopy Atlas for the Coatings Industry, 4th ed.* (Vols. 1 and 2). Federation of Societies for Coatings Technology, Blue Bell, Pennsylvania, 1991.

5.12.24. Burke, P., Curry, C. J., Davies, L. M., and Cousins, D. R. A comparison of pyrolysis mass spectrometry, pyrolysis gas chromatography, and infrared spectroscopy for the analysis of paint resins, *Forensic Science International*

(1985) 28:201-219.

5.12.25. Fukuda, K. The pyrolysis gas chromatographic examination of Japanese car paint flakes, *Forensic Science International* (1985) 29:227-236.

5.12.26. Cassista, A. R. and 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 Sciences Journal* (1994) 27:209-223.

5.12.27. Blackledge, R. D. Application of pyrolysis gas chromatography in forensic science, *Forensic Science Review* (1992) 4:2-15.

5.12.28. Challinor, J. M. Forensic application of pyrolysis capillary gas chromatography, *Forensic Science International* (1983) 1:269-285.

5.12.29. Wampler, T. P. Analytical pyrolysis: An overview and instrumentation and analysis. In: *Applied Pyrolysis Handbook,* Wampler, T. P. (ed.). Marcel Dekker, New York, 1995, pp. 1-54.

5.12.30. Freed, D. J. and Liebman, S. A. Basic analytical pyrolysis instrumentation. In: *Pyrolysis and GC in Polymer Analysis,* Liebman, S. A. and Levy, E. J. (ed.). Marcel Dekker, New York, 1985, pp. 15-51.

5.12.31. Liebman, S. A. and Wampler, T. P. Analysis of polymeric materials: Advanced pyrolysis instrumentation systems. In: *Pyrolysis and GC in Polymer Analysis,* Liebman, S. A. and Levy, E. J. (eds.). Marcel Dekker, New York, 1985, pp. 15-51.

5.12.32. McMinn, D. G., Carlson, T. L., and Munson, T. O. Pyrolysis capillary gas chromatography-mass spectrometry for analysis of automotive paints, *Journal of Forensic Sciences* (1985) 30:1064-1073.

5.12.33. Challinor, J. M. Examination of forensic evidence. In: *Applied Pyrolysis Handbook,* Wampler, and T. P. (ed.). Marcel Dekker, New York, 1995, pp. 207-217.

5.12.34. The use of microspectrophotometry in the examination of paints, *Forensic Science Review* (1989) 1:141-161.

5.12.35. Derrick, M. R. Infrared microspectroscopy in the analysis of cultural artifacts. In: *Practical Guide to Infrared Microspectroscopy,* Humecki, and H. J. (ed.). Marcel Dekker, New York, 1995, pp. 287-322.

5.12.36. Goldstein, J. I., Newbury, D. E., Echlin, P., Joy, D. C., Romig, A. D., Lyman, C. E., Fiori, C., and Lifshin, E. *Scanning Electron Microscopy and X-Ray Microanalysis, 2nd ed.* Plenum Press, New York, 1992, pp. 79-90, 334-336.

5.12.37. Gardiner, L. R. *The Homogeneity of Modern Household Paints Using Scanning Electron Microscopy: Energy Dispersive X-Ray Analysis (SEM-EDXA),* HOCRE Report 408, 1981, pp. 1-6.

5.12.38. Fischer, R. and Hellmiss, G. Principles and forensic applications of x-ray diffraction and x-ray fluorescence. In: *Advances in Forensic Science* (Vol. 2). Medical, Chicago, 1989, pp. 129-158.

5.12.39. Howden, C. R., Dudley, R. J., and Smalldon, K. W. The non-destructive analysis of single layered household paints using energy dispersive x-ray fluorescence spectrometry, *Journal of the Forensic Science Society* (1977) 17: 161-167.

5.12.40. Snider, A. M. *X-Ray Techniques for Coatings Analysis, Analysis of Paints and Related Materials: Current Techniques for Solving Coatings Problems,* Golton, W. C. (ed.). ASTM STP, Philadelphia, 1992, pp. 82-95.

5.12.41. Allen, T. J. *Effects of Environmental Factors on the Fluorescence of White Alkyd Paint,* CRSE Report 758, 1994.

5.12.42. McCrone, W. C. and Delly, J. G. *The Particle Atlas* (Vol. 1). Ann Arbor Science, Ann Arbor, Michigan, 1976, pp. 221-222.

5.12.43. Brown, R. Light and electron microscopy of inorganic paint constituents. In: *Proceedings of Scanning '93, Scanning,* 15, Supp. III, 1993, pp. III-35.

5.12.44. Goebel, R. and Stoecklein, W. The use of electron microscopic methods for the characterization of paints in forensic science, *Scanning Microscopy* (1987) 1:1007-1015.

6. Paint Database Query Requests

6.1. Scope: This test method is for the analysis, entry, evaluation and reporting of make and model information for a questioned vehicle paint sample by use of the Paint Data Query (PDQ) software. The scope of this type of evidence is limited to OEM (Original Equipment Manufacturer) vehicle paint with at least one primer layer intact. The goal is to determine if the paint layers that are present in an unknown sample are similar to any known OEM vehicle paint standards that have been entered into the database. With this database search, it is sometimes possible to categorize an unknown OEM vehicle paint sample within a group (or groups) of vehicles based upon make, model and/or year of manufacturer. It is not possible to associate an unknown paint sample to a single, individual source. The methods and practices described have been published, peer-reviewed, and are generally accepted within the forensic community.

6.2. Precautions/Limitations:

6.2.1. Care shall be taken with questioned paint samples from cases in which there is no suspect to preserve the sample for possible future analysis. If a known sample from a suspect vehicle becomes available following the PDQ examination it may be necessary to conduct a full paint examination on the questioned sample to compare it to the known sample. See the Test Method for Paint Examinations.

6.2.2. The PDQ analysis and comparison of OEM vehicle paint evidence in the forensic science laboratory can provide valuable information due to the variability of paint. However, some automotive paint exhibits more variability than others. In general, the more complex the paint, the more variable it is. Because of the ever-changing markets, suppliers, and economic factors, it is not practical to establish the statistical probability that a given sample would have the same physical and chemical characteristics as a randomly paint sample.

6.2.3. The most variability in OEM paint samples is found within the primer layers of the paint. For this reason, a successful PDQ search is dependent upon at least one primer layer being present in the layer sequence of the sample.

6.2.4. The information available from a heterogeneous specimen may diminish as its size is reduced and its condition degrades. The smaller a specimen, the less valuable it may become for an association. As sample size is reduced, it may no longer be representative of the original material.

6.2.5. The PDQ database is not all inclusive. The database contains information from OEM automotive paint systems only. The database does not have every single OEM vehicle paint system that is available. It also does not include repaint systems or utility paint systems. Informational data derived from PDQ shall be evaluated appropriately.

6.2.6. An item where paint particles may be present shall be packaged in a sealed and labeled container in such a way that paint is not lost, transferred, or compromised for analysis. When individual paint particles are recovered, the paint shall be

appropriately packaged (i.e. put into a paper bindle then placed into a properly sealed and identified pill box). Known paint samples should be submitted with each case and packaged similarly.

6.3. Related Information:

6.3.1. Appendix 1 Worksheets
6.3.2. Appendix 2 Abbreviations
6.3.3. Appendix 3 Definitions
6.3.4. Test Method for Paint Examination

6.4. Instruments:

6.4.1. Stereomicroscope – a microscope which gives a "three dimensional view" capable of examining samples up to a magnification of approximately 90x. Stereomicroscopical observations (e.g., layer sequence, colors, layer thickness, and texture) provide initial and discriminating information for paint comparisons.

6.4.2. Polarized Light Microscope (PLM) - a microscope which can be used to evaluate the properties of paint samples. Characterization of inorganic materials are accomplished with the use of PLM.

6.4.3. Fourier Transform Infrared (FTIR) Spectrometer– an analytical instrument capable of chemically analyzing the molecular vibrations of the chemical components of paint. Organic and some inorganic constituents may be evaluated with the use of infrared spectroscopy. The components of individual layers can be evaluated with the Microscope FTIR.

6.4.4. PDQ Software and computer – An established paint database of automotive paint that is searchable.

6.5. Reagents/Materials: No chemical solvents are necessary for analysis of paint for entry into the PDQ database.

6.5.1. Scalpel or razor blade
6.5.2. Low e^- microscope slide
6.5.3. SEM stubs
6.5.4. Roller knife
6.5.5. Liquid Nitrogen
6.5.6. Disposable gloves
6.5.7. Laboratory coat
6.5.8. Tweezers
6.5.9. Probes
6.5.10. Dropper bottles

6.6. Hazards/Safety:

6.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial

examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as "Biohazard" shall be handled with gloved hands during the entire examination process. Items handled with gloves shall be disinfected prior to handling with bare hands.

6.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling occurs first, the forensic scientist shall still take precautions during examination for potential additional DNA analysis in the future. During the examination the forensic scientist shall also wear a disposable face mask. Gloves shall be changed after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

6.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to minimize additional fingerprints, or obliterating existing fingerprints.

6.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

6.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS's shall be consulted.

6.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

6.7. Reference Materials/Controls/Calibration Checks:

6.7.1. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

6.7.2. The FTIR Instrument shall be performance checked as required in Appendix 4.

6.7.3. The SEM Instrument shall be performance checked as required in Appendix 5.

6.7.4. The PDQ software shall be performance checked as required in Appendix 16.

6.7.5. In the event that performance checks are found to be unsatisfactory, the instrument shall be taken out-of-service and measures taken to restore the instrument to proper working order.

6.7.5.1. Any instrument that is out-of-service shall be visibly marked.

6.7.5.2. When an instrument is taken out-of-service for maintenance and/or repair, performance checks shall be performed prior to resuming casework on that instrument.

6.7.5.3. An infrequently used instrument may be placed in an "Inactive" status and the normal verification procedures may be suspended. Normal quality control procedures shall be resumed prior to use in casework analysis.

6.8. Procedures/Instructions:
This section provides an overview of suggested flow of analytical techniques to be utilized for the analysis of paint for entry into the PDQ database. The exact selection and order of methods depends upon the sample size and/or condition.

6.8.1. Initial Evaluation and Handling Due to concerns with the handling of paint as physical evidence, each case shall be evaluated to develop appropriate procedures concerning the sampling size, collection, preservation, and order of examinations.

6.8.1.1. Different forensic disciplines may be called upon to examine the same item of evidence. The order in which the examinations shall be conducted needs to be resolved on a case-by-case basis. The order of examinations shall be selected and conducted so as to preserve the most transient evidence and provide the greatest discrimination and most valuable information. Forensic Scientist may make the submitting agency aware of the effects that some disciplines' processing and examinations may have upon other specific examination requests. If another discipline is chosen before the paint examination, obtaining an unadulterated representative sample shall be considered.

6.8.1.2. When the amount of a paint specimen present for comparison purposes is adequate in size—as deemed by the forensic scientist —analysis may include several different analytical techniques. Nondestructive methods shall be exhausted before subjecting the sample to any destructive tests. Questioned paint samples shall be preserved for possible future comparison to a known sample, if one shall become available.

6.8.1.2.1. Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics. Preliminary examination shall include its general appearance macroscopically and under a stereomicroscope, including any physical damage.

6.8.1.3. The item of evidence shall be preserved in a manner that does not interfere with future testing. All procedures shall be conducted in such a manner to ensure that no cross-contamination occurs. The item shall be photographed or described prior to conducting any destructive analyses in order to provide

documentation of original condition.

6.8.1.4. For hit and run cases involving personal injury, the victim's clothing shall be processed in the manner outlined below:

6.8.1.4.1. Clean off the examination surface and spread a clean piece of examination paper out on the examination surface.

6.8.1.4.2. Open the container, avoiding the breakage of existing seals when possible. Remove the evidence and mark it with the case number, item number, and initials.

6.8.1.4.3. A careful examination is conducted at this time. The color and location of paint smears and other stains is noted. Visible paint chips are removed and transferred to evidence boxes.

6.8.1.4.4. Clothing items should then be scraped off with a spatula over the paper in order to dislodge paint chips, which may be imbedded in the fabric. Pocket linings and hems deserve particular attention in the scraping process. Clothing articles are then shaken to dislodge any remaining debris. The accumulated debris is transferred from the paper to an evidence box.

6.8.1.4.5. Using a stereomicroscope, note the materials present in the debris. Note the relative number and color of paint particles present.

6.8.1.5. The initial examination may be the final step in an analysis if no question paint is located.

6.8.2. Physical Characteristic Evaluation
Macroscopic and stereomicroscopical observations (e.g., colors, layer sequences, layer thicknesses, layer texture) provide initial and very discriminating information for paint comparisons. Physical characterization of paint specimens is the initial step of a comprehensive forensic paint analysis. The number of layers, the layer sequence, the layer colors, relative thickness of the layers, and the texture of the paint varies and therefore is a useful characteristic for forensic examinations. Visual characteristics are the quickest, most discriminating, and least invasive examinations. If the sample appears to be an OEM vehicle paint, FTIR analysis may be warranted for entry into PDQ.

6.8.2.1. The stereomicroscopical characteristics are sometimes apparent without sample preparation. For a more definitive evaluation, a manual or microtome cross section can employed.

6.8.2.2. General Characteristics The following characteristics shall be documented when applicable:
6.8.2.2.1. Color of each layer
6.8.2.2.2. Sequence of each layer

6.8.2.2.3. Texture of each layer
6.8.2.2.4. Thickness of each layer
6.8.2.2.5. Number of layers present
6.8.2.2.6. Type and characteristics of flake present

6.8.2.3. Markings on the surface layer of the paint
Under the stereomicroscope the paint shall be examined for features such as marks, striations, dimples, and inclusions on the layer of paint that is attached to the substrate. The shapes and type of markings shall be documented.

6.8.2.4. Multiple Layer Paint Chips Multiple layers may be present in paint chips and the chip shall be examined to determine if multiple layers are present. There are a number of ways to cross-section paint (e.g. hand sectioning, microtome). The multiple layers, if present, shall be characterized and then analyzed by FTIR.

6.8.3. FTIR Evaluation
Organic and some inorganic constituents of the paint samples shall be evaluated with the use of infrared spectroscopy. It can be used to evaluate binders, pigments, and additives in various types of coating materials. Since many of the samples have limited size, the microscope attachment is typically needed to analyze the samples. Transmittance and reflectance techniques can be employed.

6.8.3.1. Sample and background scans shall be run under the same instrument conditions. A resolution of 4 cm^{-1} (one data point every 2 cm^{-1}) shall be the minimum resolution. Higher resolution may be used.

6.8.3.2. Samples to be searched in the PDQ database shall be analyzed with the FTIR Microscope accessory. The use of a microscope accessory is preferred for very small samples. Spectra can be obtained from samples as small as 10-20 micrometers in diameter after flattening. The microscope attachment permits the analysis of multiple samples placed on an appropriate support material. The method affords the advantage of viewing the sample optically and choosing the most appropriate area for analysis.

6.8.3.3. Transmission measurements generate spectra with fewer artifacts. However, transmission methods generally entail more sample preparation than reflection techniques. The paint sample shall be rendered thin enough not to over-absorb. This typically requires a sample thickness of approximately 3-5 micrometers.

6.8.3.4. If samples are flattened directly on an infrared light reflecting surface (e.g., low e-glass or gold mirror), the reflection mode can be used to produce spectra mimicking double-pass transmission spectra. The technique is sometimes referred to as "transflection" or “reflection/absorption”. The FTIR microscope can also be used in the specular reflection mode.

6.8.3.5. ATR objectives are available for infrared microscopes. Consistent pressure shall be applied to each sample to mitigate spectral variations. Intra-sample variations may result from sample heterogeneity; therefore, multiple samplings shall be considered.

6.8.3.6. Spectral comparisons of the paint component spectra can be accomplished by digital overlays with full scale expansion. Comparison of samples may be conducted with both spectra displayed in transmittance and/or absorbance. Certain information may be seen more readily in one format or the other.

6.8.3.7. There are a number of significant factors that shall be considered when comparing spectra including the presence or absence of absorption bands, and their position (wavenumber), shape and relative intensity. Additional sample replicates may be necessary to evaluate reproducibility of these spectral characteristics.

6.8.3.8. The presence of additional absorption bands could be from true differences between the samples or from extraneous material adhering to the paint. If extraneous material is suspected as the source of the difference, the sample shall be cleaned or additional samples prepared. If the sample cannot be cleaned or resampled, then spectral subtraction may be an option.

6.8.3.9. For spectra to be considered indistinguishable, the position of the absorption bands shall have reasonable agreement with each other. A rule of thumb is that the positions of corresponding peaks in two or more spectra being compared shall be within a few wavenumbers of each other, depending on whether the peak is sharp or broad. For sharp absorption peaks one shall use tighter constraints and with broad peaks the variation shall be slightly greater.

6.8.3.10. For spectra to be considered indistinguishable, the shape of the absorption bands shall be consistent between comparison samples. The peak width and the symmetry of each peak shall be evaluated. Sample thickness may affect the peak width and resolution.

6.8.3.11. For spectra to be considered indistinguishable, the relative intensities of respective absorption bands shall be similar between comparison samples. The relative intensity may be affected by the heterogeneity of the sample.

6.8.3.12. Three possible conclusions can be reached after evaluating and comparing spectra: 1) the spectra are dissimilar, 2) the spectra are indistinguishable, or 3) inconclusive.

6.8.3.13. The spectra are dissimilar if there are one or more significant differences in the spectra. Significant differences are differences in which the spectral

variation cannot be explained other than as differences between samples.

6.8.3.14. The spectra are indistinguishable when there are no significant differences in the spectra. Differences are not significant if the spectral variation can be explained.

6.8.3.15. An inconclusive determination is one in which the significance of the differences cannot be completely assessed due to the constraints of sample size and/or condition.

6.8.3.16. Paint is often comprised of a number of layers that can't be completely isolated which can result in overlapping bands in the IR spectra; therefore, caution shall be exercised while evaluating the data.

6.8.3.17. The use of the PDQ database requires that spectra be coded based upon the components present in each individual paint layer.

6.8.3.18. Tools that can assist in the characterization of the spectra include, but are not limited to, spectral libraries, flow charts, and reference materials. Most commercial spectral libraries consist of transmission (as opposed to reflection) spectra. It is desirable to use reference spectra that were obtained using the same sample preparation and collection technique.

6.8.3.19. Spectra can be searched in tandem, or individually using PDQ or another software program. The coding for the paint with the best hit quality may help in PDQ coding.

6.8.3.20. Case record shall include a description of the evidence analyzed by IR, the method of sample preparation, the analytical instrumentation used, and its operating parameters.

6.9. Records: Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics.

6.9.1. Preliminary examination of paint shall include its general appearance macroscopically and under a stereomicroscope, including any adhering matter.

6.9.2. The case record shall include the instrumental data. All hard copies shall include a unique sample designation, the operator's name/initials, and the date of analysis. Case record shall also include a description of the evidence analyzed, the method of sample preparation, the analytical instrumentation used, and its operating parameters.

6.9.3. The PDQ spectral codings that are searched for each layer, along with search results and hit lists shall be included with the case record.

6.9.4. It may be necessary to contact the PDQ Administrators to confirm PDQ spectra coding. All communications should be documented and included in the case record.

6.10. Interpretations of Results:

6.10.1. A successful PDQ search will produce results that report information including make(s), model(s) and range(s) of years in which the OEM sequence from the questioned paint sample may have been used.

6.10.2. Partial incomplete results may be reported, when appropriate. Examples of these include possible makes without a production year range, possible makes without a model, etc.

6.11. Report Writing:

6.11.1. For samples where a search of the PDQ database was successful:

6.11.1.1. "A search of the PDQ (Paint Data Query) indicated that the Item ___ multilayered red metallic paint chip was consistent with paint used on the following vehicles:

1998 – 1999 Toyota Avalon
1998 – 2001 Toyota Camry

6.11.1.1.1. It shall be noted that not all makes/models/years of vehicles produced by each manufacturer are present in the PDQ database."

6.11.2. For samples where a search of the PDQ database was unsuccessful:

6.11.2.1. "A search of the PDQ (Paint Data Query) for Item ___ multilayered red metallic paint chip was not consistent with any paint in the PDQ database. It shall be noted that not all makes/models/years of vehicles produced by each manufacturer are present in the PDQ database."

6.11.3. For samples where an OEM sequence is not present:

6.11.3.1. "The red metallic paint (particles, smears, etc.) present (in the debris, from, on) Item ___ was not suitable for make/model determination.

6.12. References:

6.12.1. SWGMAT Trace Recovery Guidelines [Online] (October 1999). Available: http://www.fbi.gov/hq/lab/fsc/backissu/oct1999/index.htm

6.12.2. PDQ Manual

6.12.3. Crown, D. A. *The Forensic Examination of Paints and Pigments.* Charles C. Thomas, Springfield, Illinois, 1968.

6.12.4. Thornton, J. I. Forensic paint examination. In: *Forensic Science Handbook* (Vol. 1). Saferstein, R. (ed.). Prentice-Hall, New Jersey, 1982, pp. 529-571.

6.12.5. Stoecklein, W. Forensic science: Paints, varnishes and lacquers. In: *Encyclopedia of Analytical Science.* Academic Press, New York, 1995, pp. 1625-1635.

6.12.6. Laing, D. K., Locke, J., Richard, R. A., and Wilkerson, J. M. The examination of paint films and fibers as thin-sections, *Microscope* (1987) 35:233-248.

6.12.7. Allen, T. J. Modification of sample mounting procedures and microtome equipment for paint sectioning, *Forensic Science International* (1991) 5:93-100.

6.12.8. McCrone, W. C. Application of particle study in art and archeology conservation and authentication. In: *The Particle Atlas* (Vol. V). Ann Arbor Science, Ann Arbor, Michigan, 1979, pp. 1402-1413.

6.12.9. Kilbourn, J. H. and Marx, R. B. Polarized light microscopy of extenders in structural paints: Forensic applications, *Microscope* (1994) 42:167-175.

6.12.10. O'Neill, L. A. Analysis of paints by infrared spectroscopy, *Medicine, Science and the Law* (1967) 7:145-147.

6.12.11. Suzuki, E. M. Forensic applications of infrared spectroscopy. In: *Forensic Science Handbook* (Vol. 3). Saferstein, R. (ed.). Regents/Prentice Hall, Englewood Cliffs, New Jersey, 1993, pp. 71-195.

6.12.12. Ryland, S. G. Infrared microspectroscopy of forensic paint evidence. In: *Practical Guide to Infrared M*

6.12.13. Wilkinson, J. M., Locke, J., and Laing, D. K. The examination of paints at thin-sections using visible microspectrophotometry and Fourier transform infrared microscopy, *Forensic Science International* (1988) 38:43-52.

6.12.14. Allen, T. J. Paint sample presentation for Fourier transform infrared microscopy, *Vibrational Spectroscopy* (1992) 3:217-37.

6.12.15. Bartick, E. G. and Tungol, M. W. Infrared microscopy and its forensic applications. In: *Forensic Science Handbook* (Vol. 3). Saferstein, R. (ed.). Regents/Prentice Hall, Englewood Cliffs, New Jersey, 1993, pp. 196-252.

6.12.16. 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* (1983) 16:163-173.

6.12.17. Suzuki, E. M. Infrared spectra of U.S. automobile original topcoats (1974-1989): Differentiation and identification based on acrylonitrile and ferrocyanide C=N stretching absorptions, *Journal of Forensic Sciences* (1996) 41:376-392.

6.12.18. Suzuki, E. M. Infrared spectra of U.S. automobile original topcoats (1974-1989): Identification of some topcoat inorganic pigments using an extended range (4000-220 cm-1) Fourier transform spectrometer, *Journal of Forensic Sciences* (1996) 41:393-406.

6.12.19. Suzuki, E. M. and Marshall, W. P. Infrared spectra of U.S. automobile original topcoats (1974-1989): In situ identification of some organic pigments used in yellow, orange, red and brown nonmetallic and brown metallic finishes - benzimidazolones, *Journal of Forensic Sciences* (1997) 42:619-648.

6.12.20. Infrared Spectroscopy Atlas Working Committee, *An Infrared Spectroscopy Atlas for the Coatings Industry, 4th ed.* (Vols. 1 and 2). Federation of Societies for Coatings Technology, Blue Bell, Pennsylvania, 1991.

6.12.21. Burke, P., Curry, C. J., Davies, L. M., and Cousins, D. R. A comparison of pyrolysis mass spectrometry, pyrolysis gas chromatography, and infrared spectroscopy for the analysis of paint resins, *Forensic Science International* (1985) 28:201-219.

6.12.22. Fukuda, K. The pyrolysis gas chromatographic examination of Japanese car paint flakes, *Forensic Science International* (1985) 29:227-236.

6.12.23. Cassista, A. R. and 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 Sciences Journal* (1994) 27:209-223.

6.12.24. Blackledge, R. D. Application of pyrolysis gas chromatography in forensic science, *Forensic Science Review* (1992) 4:2-15.

6.12.25. Challinor, J. M. Forensic application of pyrolysis capillary gas chromatography, *Forensic Science International* (1983) 1:269-285.

6.12.26. Wampler, T. P. Analytical pyrolysis: An overview and instrumentation and analysis. In: *Applied Pyrolysis Handbook,* Wampler, T. P. (ed.). Marcel Dekker, New York, 1995, pp. 1-54.

6.12.27. Freed, D. J. and Liebman, S. A. Basic analytical pyrolysis instrumentation. In: *Pyrolysis and GC in Polymer Analysis,* Liebman, S. A. and Levy, E. J. (eds.). Marcel Dekker, New York, 1985, pp. 15-51.

6.12.28. Liebman, S. A. and Wampler, T. P. Analysis of polymeric materials: Advanced pyrolysis instrumentation systems. In: *Pyrolysis and GC in Polymer Analysis,* Liebman, S. A. and Levy, E. J. (eds.). Marcel Dekker, New York, 1985, pp. 15-51.

6.12.29. McMinn, D. G., Carlson, T. L., and Munson, T. O. Pyrolysis capillary gas chromatography-mass spectrometry for analysis of automotive paints, *Journal of Forensic Sciences* (1985) 30:1064-1073.

6.12.30. Challinor, J. M. Examination of forensic evidence. In: *Applied Pyrolysis Handbook,* Wampler, T. P. (ed.). Marcel Dekker, New York, 1995, pp. 207-

217.

6.12.31. The use of microspectrophotometry in the examination of paints, *Forensic Science Review* (1989) 1:141-161.

6.12.32. Derrick, M. R. Infrared microspectroscopy in the analysis of cultural artifacts. In: *Practical Guide to Infrared Microspectroscopy,* Humecki, H. J. (ed.). Marcel Dekker, New York, 1995, pp. 287-322.

6.12.33. Goldstein, J. I., Newbury, D. E., Echlin, P., Joy, D. C., Romig, A. D., Lyman, C. E., Fiori, C., and Lifshin, E. *Scanning Electron Microscopy and X-Ray Microanalysis, 2nd ed.* Plenum Press, New York, 1992, pp. 79-90, 334-336.

6.12.34. Gardiner, L. R. *The Homogeneity of Modern Household Paints Using Scanning Electron Microscopy: Energy Dispersive X-Ray Analysis (SEM-EDXA),* HOCRE Report 408, 1981, pp. 1-6.

6.12.35. Fischer, R. and Hellmiss, G. Principles and forensic applications of x-ray diffraction and x-ray fluorescence. In: *Advances in Forensic Science* (Vol. 2). Medical, Chicago, 1989, pp. 129-158.

6.12.36. Howden, C. R., Dudley, R. J., and Smalldon, K. W. The non-destructive analysis of single layered household paints using energy dispersive x-ray fluorescence spectrometry, *Journal of the Forensic Science Society* (1977) 17: 161-167.

6.12.37. Snider, A. M. *X-Ray Techniques for Coatings Analysis, Analysis of Paints and Related Materials: Current Techniques for Solving Coatings Problems,* Golton, W. C. (ed.). ASTM STP, Philadelphia, 1992, pp. 82-95.

6.12.38. Allen, T. J. *Effects of Environmental Factors on the Fluorescence of White Alkyd Paint,* CRSE Report 758, 1994.

6.12.39. McCrone, W. C. and Delly, J. G. *The Particle Atlas* (Vol. 1). Ann Arbor Science, Ann Arbor, Michigan, 1976, pp. 221-222.

6.12.40. Brown, R. Light and electron microscopy of inorganic paint constituents. In: *Proceedings of Scanning '93, Scanning,* 15, Supp. III, 1993, pp. III-35.

6.12.41. Goebel, R. and Stoecklein, W. The use of electron microscopic methods for the characterization of paints in forensic science, *Scanning Microscopy* (1987) 1:1007-1015.

7. Glass Comparison Examinations

7.1. Scope:

This test method is for the analysis, examination, and comparison of evidentiary glass. The scope of this type of evidence includes but is not limited to automotive glass, architectural glass, bottle glass, and decorative glass. A search is made of a myriad of different items for the presence of glass which may be of evidentiary value. The goal is to determine if any significant differences exist between the samples. The evaluation of glass for class characteristics can associate known and questioned glass to a group but not to a single, individual source. A physical edge match of two glass fragments provides individualizing characteristics that associate the two glass fragments to one another. The methods and practices described have been published, peer-reviewed, and are generally accepted within the forensic community.

7.2. Precautions/Limitations:

7.2.1. The analysis and comparison of glass evidence in the forensic science laboratory can provide valuable information due to the variability of glass. Because of the unknown characteristics of all of the old glass present in an environment and the unknown properties of the manufacture of additional glass, it is not practical to establish the statistical probability that a given sample would have the same physical, optical and chemical characteristics as a randomly selected glass sample.

7.2.2. Glass samples by nature are brittle. Submitted items may have glass fragments present that are extremely small. Sometimes questioned samples may not allow for all characteristics to be examined and compared to a known glass standard. The strength of an association between a small questioned sample and a larger sample might be weakened depending upon the degree of comparison. In some cases, the glass may be unsuitable for all comparison properties except for refractive index. These changes may limit the information obtained from the analyses. When the glass sample size limits the full range of examinations, the examinations and analyses that are performed shall be documented in the forensic scientist's case record.

7.2.3. An item on which glass fragments may be present shall be packaged in a sealed and labeled container in such a way that glass is not lost, transferred, or compromised for analysis. When individual glass fragments are recovered, the glass shall be appropriately packaged (i.e. placed into a properly sealed and identified pill box). Glass standards shall be submitted with each case. The standards shall be packaged separately and in such a way that the glass is not lost, transferred, or compromised for analysis.

7.3. Related Information:

7.3.1. Appendix 1 Worksheets
7.3.2. Appendix 2 Abbreviations
7.3.3. Appendix 3 Definitions
7.3.4. Test Method for Physical Edge Match Evaluations

7.3.5. Test Method for Glass Fractures

7.4. Instruments:

7.4.1. Stereomicroscope – a microscope which gives a "three dimensional view" capable of examining samples up to a magnification of approximately 90x. Stereomicroscopical observations (e.g., fracture lines, transparency, sharp edges) provide initial and discriminating information for selecting fragments to identify as glass and then to use for glass comparisons.

7.4.2. Calipers – an instrument capable of accurately and precisely measuring the thickness of different samples. It can be used to measure the thickness of glass when both edges are present.

7.4.3. Polarized Light Microscope (PLM) - a microscope which can be used to evaluate the properties of glass samples. Characterization and identification of glass is accomplished with the use of PLM.

7.4.4. UV Box – an instrument designed to allow for the viewing of specimens in short and long wavelengths of light. It can be used to look at the fluorescence of float glass and also coatings on glass.

7.4.5. Scanning Electron Microscope with Energy Dispersive Spectroscopy (SEM/EDS) – an analytical instrument capable of chemically analyzing a glass sample. It can be utilized for the characterization of the inorganic constituents of glass.

7.4.6. X-ray Fluorescence (XRF) – an analytical instrument capable of chemically analyzing a glass sample. It can be utilized for the characterization of the inorganic constituents of glass down to the trace elements that are present.

7.4.7 Glass Refractive Index Measurement System (GRIM III) – an instrument used to determine the refractive index of a glass sample.

7.5. Reagents/Materials: Chemical solvents are used (i.e. bromoform, methanol, and acetone) in the analysis of glass.

7.5.1. Microscope slides
7.5.2. Microscope cover slips
7.5.3. Suitable solvents
7.5.4. Mounting medium
7.5.5. Refractive index liquids
7.5.6. Disposable gloves
7.5.7. Laboratory coat
7.5.8. Tweezers
7.5.9. Probes

7.6. Hazards/Safety:

7.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as "Biohazard" shall be handled with gloved hands during the entire examination process.

7.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling occurs first, the forensic scientist shall take precautions during examination for potential additional DNA analysis in the future. During the examination the forensic scientist shall wear a disposable face mask, gloves and a lab coat. Gloves shall be changed after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

7.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to prevent obliterating fingerprints and/or depositing additional fingerprints.

7.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

7.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS's shall be consulted.

7.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

7.6.7. Care shall be taken to prevent lacerations from glass samples.

7.7. Reference Materials/Controls/Calibration Checks:

7.7.1. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

7.7.2. Questioned and known samples shall be analyzed under similar conditions.

7.7.3. The XRF shall be performance checked as required in Appendix 8.

7.7.4. The GRIM III Instrument shall be performance checked as required in Appendix 9.

7.7.5. The Digital Micrometer shall be performance checked as required in Appendix 13.

7.7.6. The Polarized Light Microscope shall be performance checked as required in Appendix 14.

7.7.7. In the event that performance checks are found to be unsatisfactory, the instrument shall be taken out-of-service and measures taken to restore the instrument to proper working order.

7.7.7.1. Any instrument that is out-of-service shall be visibly marked.

7.7.7.2. When an instrument is taken out-of-service for maintenance and/or repair, performance checks shall be performed prior to resuming casework on that instrument.

7.7.7.3. An infrequently used instrument may be placed in an "Inactive" status and the normal verification procedures may be suspended. Normal quality control procedures shall be resumed prior to use in casework analysis.

7.8. Procedures/Instructions:
This section provides an overview of suggested flow of analytical techniques to be utilized for the analysis of glass. The exact selection and order of methods depends upon the sample size and/or condition. Fragment size sometimes precludes the determination of some or all of a glass fragment's physical properties. In this situation, physical properties are determined to the extent possible, and conclusions are drawn based on those properties that can be determined. Glass comparison examinations shall be considered for fracture match examination, if feasible. Physical fitting provides the only conclusive association between glass samples. Another examination that may be requested is glass identification. A request may be that a submitted sample be identified as glass, or it may be to identify the type of glass in a sample. Physical properties and/or refractive index measurements are determined to the extent necessary to reach a conclusion.

7.8.1. Initial Evaluation and Handling Due to concerns with the handling of glass as physical evidence, each case shall be evaluated to develop appropriate procedures concerning the sampling size, collection, preservation, and order of examinations.

7.8.1.1. Different forensic disciplines may be called upon to examine the same item of evidence. The order in which the examinations shall be conducted needs to be resolved on a case-by-case basis. The order of examinations shall be selected and conducted so as to preserve the most transient evidence and provide the greatest discrimination and most valuable information. If another discipline is chosen before the glass examination, obtaining an unadulterated representative sample shall be considered.

7.8.1.2. When the amount of a glass specimen present for comparison purposes is adequate in size, analysis may include several different analytical techniques. Non-destructive methods shall be exhausted before subjecting the sample to any destructive tests.

7.8.1.3. If the initial tests are not exclusionary, the examination shall proceed with additional tests that are selected on the basis of their potential for use in discriminating the questioned and known samples. The color, size, condition, and type of glass present will dictate the selected analytical scheme.

7.8.1.4. Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics. Preliminary examination shall include its general appearance macroscopically and under a stereomicroscope.

7.8.1.5. The item of evidence shall be preserved in a manner that does not interfere with future testing whenever possible. All procedures shall be conducted in such a manner to ensure that no cross-contamination occurs. The item shall be photographed or described prior to conducting any destructive analyses in order to provide documentation of original condition. The forensic scientist shall retain the actual sample analyzed.

7.8.1.6. If laminated glass is submitted in its complete form, samples shall be taken from both panes and treated as two separate samples.

7.8.1.7. For glass examinations where clothing has been submitted for examination, the clothing should be processed in the manner outlined below:

7.8.1.7.1. Clean off the examination surface and spread a clean piece of examination paper out on the examination surface.

7.8.1.7.2. Open the container, avoiding the breakage of existing seals when possible. Remove the evidence and mark it with the case number, item number, analyst's PE number, and initials if possible to do so.

7.8.1.7.3. A careful examination is conducted at this time. Visible glass fragments are removed and transferred to evidence boxes.

7.8.1.7.4. For clothing items, the general type, color(s), size, and manufacturer should be noted. The overall condition of each item (new, worn, clean, soiled, apparent blood stains, fabric separations, etc.) should be noted.

7.8.1.7.5. Clothing items should then be scraped with a spatula over the paper in order to dislodge glass fragments, which may be imbedded in the fabric. Pocket linings and hems deserve particular attention in the scraping process. Clothing articles should then be shaken and/or tape

lifted to dislodge any remaining debris. The clothing should be turned inside-out and the scrape down and/or tape lift should be repeated. The accumulated debris from the shake down should then be transferred from the paper to an evidence box.

7.8.1.7.6. Rigid objects such as tools and weapons should be processed by the most appropriate means, depending on the item. If fragments are embedded in a soft object such as plastic, wood or aluminum, hand picking, or probing in gouges may be the most effective means of removal.

7.8.1.7.6.1. For objects such as tools or weapons, the type, color, manufacturer, size and appearance should be noted. Objects may exhibit scratches, gouges or other damage, which should be noted.

7.8.1.7.7. For objects such as shoes or boots, the type, color, manufacturer, size, and appearance should be noted. Objects may exhibit unique features, inclusion, or wear patterns, which should be noted.

7.8.1.7.8. When removing debris from footwear, not only the exterior of the footwear shall be thoroughly examined, but the interior of the footwear shall also be thoroughly examined. The laces and insoles shall be removed and the debris from the footwear shall be collected and analyzed. Macroscopically visible glass fragments may be removed by particle picking.

7.8.1.7.9. Using a stereomicroscope, note the materials present in the debris. Note the relative number and physical characteristics of potential glass fragments present.

7.8.1.7.10. Any possible glass fragments removed from debris should be checked to ensure the fragment is glass. Using a microscope fitted with polarizing light capabilities should be used. Glass fragments are insoluble in water, exhibit sharp edges and are isotropic under crossed polarized light. Additional features that could be present include hackle marks and conchodial fractures.

7.8.1.7.11. Glass fragments shall be cleaned prior to further analysis if necessary. When glass fragments are cleaned, the cleaning method and solvents used shall be reflected in the case record.

7.8.1.8. The initial examination may be the final step in an analysis if no questioned glass is located, or if only exclusionary samples are identified in comparison to the known sample.

7.8.2. Thickness

7.8.2.1. The thickness of a glass fragment shall be measured if the fragment is large enough and has both flat sides present.

7.8.2.2. Place the known fragment of glass into the opened clamps of the caliper. Close the clamps onto the flat surfaces of the known glass fragment. Measure the thickness of the glass fragments and record the measurement. Measure several additional fragments and record the thickness to establish a thickness range.

7.8.2.3. Repeat these steps for the questioned glass fragments, record, and compare the results.

7.8.3. Fluorescence

7.8.3.1. The fluorescence of a glass fragment shall be measured if the fragment is large enough and has one or both flat sides present..

7.8.3.2. Place the known or standard glass fragment into the UV view booth. Turn on the short wave lamp and record the observations for both flat sides of the known glass fragment

7.8.3.3. Turn off the short wave lamp and turn on the long wave lamp. Record any observations for both flat sides of the known glass fragment.

7.8.3.4. Repeat these steps for the questioned fragment(s) and record and compare the results/observations.

7.8.3.5. If the questioned and known pieces are of suitable size, the intensity of the fragments shall be evaluated with a side by side comparison.

7.8.4. Elemental Analysis (XRF)

7.8.4.1. If the glass fragment(s) have not been cleaned, this shall be done prior to elemental analysis. Any cleaning methods and chemicals used shall be noted.

7.8.4.2. To prepare a glass fragment for analysis, mount the fragment onto a plastic slide holder containing a piece of double stick carbon tape.

7.8.4.3. Mark on the surface of the glass a spot if possible with a marker so the instrument can focus on the surface of the glass.

7.8.4.4. Glass fragments of similar size shall be compared. If only microscopic fragments of glass have been recovered, prepare a sample of the known glass of a similar size. Crushing the known sample may be necessary.

7.8.4.5. Place the mounted questioned and known glass fragments into the XRF chamber, and close the chamber to start the evacuation of the air inside the chamber. When the vacuum status reaches the green area, analysis can

begin.

7.8.4.6. Bring the sample into focus and select the appropriate spot size. Obtain a spectrum for 1200 live seconds. Save the spectrum including the appropriate case, item, and fragment designation.

7.8.4.7. A shorter analysis time may be used for screening purposes but not for conducting a semi-quantitative analysis.

7.8.4.8. Obtain the data for each element properly identified in the spectrum. The comparison of known vs. questioned fragments shall be conducted on a semi-quantitative basis using elemental ratios.

7.8.4.9. The elemental ratios that may be used for the comparison of glass fragments include Ca/Mg, Ca/Ti, Ca/Fe, Sr/Ar, Fe/Zr, and Ca/K. Many other elements may be detected and compared. From these other elements, it is at the analyst's discretion which shall be used. Generally, elements close to one another in kV value shall be used for establishing elemental ratios.

7.8.4.10. The inverse of these ratios can also be used if desired.

7.8.4.11. When practical, a minimum of three replicates on each questioned specimen and nine replicates on each known glass source should be analyzed.

7.8.4.12. Compare the elemental ratios by establishing a range for each elemental ratio for the known fragment(s). The elemental ratios for the questioned fragments(s) shall be compared to those of the known fragment(s). If the elemental ratios of the questioned glass are within the range of the known glass, then the glass fragment(s) are considered to be similar for that particular ratio.

7.8.4.13. If all of the elemental ratios are similar, then the glass fragment(s) shall be considered similar in elemental composition. There are allowances for slight differences in elemental ratio comparison, and any conclusions drawn from the data shall be reviewed by another proficient analyst.

7.8.5. Refractive Index Determination

7.8.5.1. Questioned glass fragments recovered from debris from clothing or objects shall be cleaned prior to mount preparation to obtain the most suitable data.

7.8.5.2. Start the GRIM 3 system. Open the GRIM 3 software and create a data file for the ISP case.

7.8.5.2.1. Prepare a sample, on a GRIM 3 slide, of fine, crushed glass fragments using the appropriate immersion oil. Any glass pulverizer and work area shall be cleaned before and after each use. Locke Silicone Oil B is used for most window and container glasses. Locke Silicone Oil C is

used with headlight glasses. Locke Silicone Oil A is used for samples with high refractive index that are above the refractive index range of the Locke Silicone Oil B. A GRIM 3 cover slip is then placed over the prepared sample. The GRIM 3 slide shall be marked for identification purposes.

7.8.5.2.2. The prepared sample is inserted into the hot stage on the phase contrast microscope. The glass fragments are located and the microscope is focused on the glass fragments.

7.8.5.2.3. Using the phase contrast microscope, align the phase rings. This is accomplished by pushing in and turning the adjustment knobs for the phase annulus, which is located on the microscope's condenser assembly. The phase ring alignment shall be checked and adjusted for each sample, as the alignment may shift while locating a series of glass fragments to analyze.

7.8.5.3. Select fragments to measure which exhibit edges with the highest degree of contrast. Obtain RI measurements on a minimum of ten fragments for each crushed mounting, per sample. Do not take multiple readings from the same microscopic glass fragment. If a question glass is clearly not in the range of RI as the known, it is not required to conduct analysis on the full ten fragments.

7.8.5.4. When ten measurements cannot be obtained, determine the refractive index of as many fragments as possible that are available. Do not take multiple readings from the same microscopic glass fragment.

7.8.5.5. The quality of the refractive index measurement on the GRIM 3 system can be assessed by the “Edge Contrast” value assigned to each measured value. This value is shown in parentheses next to the match temperature measurement. The best value is “99", with the value decreasing as edge contrast quality decreases. Glass fragments with an edge contrast value of less than “50", in either the cooling or heating measurement cycles, are not desirable for comparison purposes. If a value is to be rejected the strikethrough feature shall be utilized.

7.8.5.6. Glass fragments with an edge contrast value of less than “50” may be used, if the analyst does not have a sufficient sample to work with. It is also possible to re-measure the fragment on a different edge to obtain an acceptable edge contrast value.

7.8.5.7. The refractive index measurement for questioned glass fragments shall be compared to the observed refractive index range for the known glass sample(s).

7.8.5.8. Questioned glass fragments with an average refractive index value that is within the observed RI range of the known glass sample(s) are considered

to be similar in refractive index.

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

When refractive index values are determined by the GRIM 3 system a printed copy of the GRIM 3 data and the equipment and calibration oils used shall be included in each case record.

7.9. Records Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics.

7.9.1. Preliminary examination of glass fragments shall include its general appearance macroscopically and under a stereomicroscope, including any adhering matter.

7.9.2. The case record shall include the instrumental data. All hard copies shall include a unique sample designation, the operator's initials, laboratory case number, and the date of analysis. The case record shall also include a description of the evidence analyzed, the method of sample preparation, the analytical instrumentation used, and its operating parameters.

7.10. Interpretations of Results:

7.10.1. Typically, a glass examination involves the comparison of samples to determine if they could share a common origin. The goal is to determine if any significant differences exist between the samples. The physical, chemical, and optical characteristics are evaluated to associate the questioned and known sample. The evaluation of glass for class characteristics can associate known and questioned glass to a group but not to a single, individual source.

7.10.1.1. If a significant difference in a characteristic exists the samples are excluded as having a common origin.

7.10.1.2. If the examination doesn't reveal any significant differences it is concluded that the samples could share a common origin.

7.10.1.3. A limited association can be reported due to the condition/size of one of the samples.

7.11. Report Writing:

7.11.1. If significant differences are found in the glass fragment characteristics, then the report shall state that the glass fragments are not similar and did not come from the same source.

7.11.2. If no significant differences are found in the glass fragment characteristics, then the report shall state that the glass fragments are similar and could have come from the same source or another source with these same characteristics.

7.12. References:

7.12.1. General References

7.12.1.1. SWGMAT Trace Recovery Guidelines [Online] (October 1999). Available: http://www.fbi.gov/hq/lab/fsc/backissu/oct1999/index.htm

7.12.1.2. Forensic Interpretation of Glass Evidence –James Michael Curran, Tacha Natalie Hicks, and John S. Buckleton – *Chapter 1: Examination of Glass* p.1 – 26

7.12.1.3. Forensic Examination of Glass and Paint – Brian Caddy – *Chapter 2: The Composition and Manufacture of Glass and its Domestic and Industrial Applications* - Geoffrey J. Copley p.27 – 46

7.12.1.4. Glass in the Modern World by FJT Maloney 1958

7.12.1.5. SWGMAT Introduction to Forensic Glass Analysis – Forensic Science Communications January 2005 – Volume 7 – Number 1

7.12.1.6. SWGMAT Initial Examinations of Glass – Forensic Science Communications January 2005 – Volume 7 – Number 1

7.12.1.7. Polarized Light Microscopy - McCrone - Sections IX and X

7.12.1.8. Transfer and Persistence of Glass Fragments on Garments – T Hicks, R Vanina, P Margot; 1995

7.12.1.9. Forensic Glass Comparison: Background Information Used in Data Interpretation – Maureen Botrell, *Forensic Science Communications* - April 2009

7.12.2. Elemental References

7.12.2.1. ASTM E2926 Standard Test Method for Forensic Comparison of Glass Using Micro X-ray Fluorescence (u-XRF) Spectroscopy

7.12.2.2. Forensic Examination of Glass and Paint – Brian Caddy – *Chapter 4, Elemental Analysis of Glass Fragments*– Jose R. Almirall p. 65 -83

7.12.2.3. Micro-XRF for Forensic Glass Analysis: Our Experience Over the Past Seven Years – Scott Ryland – *Powerpoint from Trace Symposium 2006*

7.12.2.4. The Forensic Significance of Glass Composition and Refractive Index Measurements – Robert D. Koons and JoAnn Buscaliglia; 1999

7.12.2.5. EDAX Eagle III Computer Tutorial

7.12.2.6. EDAX Eagle III Instrumental Information Book

7.12.2.7. SWGMAT Elemental Analysis of Glass – Forensic Science Communications January 2005 – Volume 7 – Number 1

7.12.3. Refractive Index References

7.12.3.1. Forensic Examination of Glass and Paint – Brian Caddy – *Chapter 3, section 3.6 Refractive Index Measurement* – Pamela S. Hamer p. 56 -62

7.12.3.2. ASTM E1967 Standard Test Method for the Automated Determination of Refractive Index of Glass Samples Using the Oil Immersion Method and a Phase Contrast Microscope

7.12.3.3. GRIM 3 Manual

7.12.3.4. Distribution of Refractive Index Values in Sheet Glasses – Robert D. Koons, JoAnn Buscaglia; FBI; 2001

7.12.3.5. The Use of Interference Filters in Refractive Index Measurements– John Locke; 1982

7.12.3.6. The Evidential Value of Dispersion in the Examination of Glass - John Locke; 1986

7.12.3.7. SWGMAT Glass Refractive Index Determination – Forensic Science Communications January 2005 – Volume 7 – Number 1

8. Glass Fracture Examinations

8.1. Scope:

This test method is for the analysis and examination of glass fractures. The scope of this type of evidence includes; the direction of force used to produce the fracture, the point or points of impact, the type of shock which resulted in the fracture (e.g. thermal or mechanical), the relative velocity of the fracture, and the sequence of fractures. The associative glass examination that involves the comparison of samples to determine if they could share a common origin is outlined in the Glass Comparison Test Method. The following methods and practices described have been published, peer-reviewed, and are generally accepted within the forensic community.

8.2. Precautions/Limitations:

8.2.1. There are instances for which direction of force is not able to be performed. The broken glass should not have a point of impact which is close to the edge of a frame. Some of the classical characteristics used in determining direction of force are not applicable to reinforced glass (e.g. wire re-enforced).

8.2.2. For direction of force, the submitted glass fragments taken directly from a window frame shall be properly labeled as to inside/outside orientation. This shall be accomplished by marking the fragments still in the frame with the correct orientation at the time of collection by the investigator.

8.2.3. For direction of force, a sufficient amount of the total glass fragments from the broken pane shall be submitted in order to reconstruct the pane and subsequently make an appropriate conclusion with regard to direction of force. This shall be accomplished by collecting and submitting all of the larger pieces, including those from the floor, those from the ground and those found in the frame. There shall be enough of the glass present to determine if a specific crack is a radial or concentric fracture.

8.2.4. For sequence of impact, the glass pane shall hold together in order to retain the pattern. Laminated or wire reinforced panes will retain a pattern suitable for examination, but sequencing can also be performed on other forms of glass panes that are intact and have not gone under extensive, additional breakage. The investigator shall collect and preserve as much of the window pane as possible for this determination.

8.2.5. Caution shall be used when comparing multiple impacts on a laminated pane. Cracks may extend after the impacts have initially occurred, which will interfere with any conclusions made. Extensive photographs should be taken by the investigator of the laminated pane before the pane is moved/disturbed in any way (i.e. transporting/moving a vehicle with an impacted windshield).

8.2.6. An item which glass fragments may be present should be packaged in a sealed and labeled container in such a way that glass is not lost, transferred, or compromised for analysis. When a pane of glass or glass fragments are

recovered, the glass should be appropriately packaged (i.e. packaged in a container so no further cracking or breakage occurs).

8.3. Related Information:

8.3.1. Appendix 1 Worksheets
8.3.2. Appendix 2 Abbreviations
8.3.3. Appendix 3 Definitions
8.3.4. Test Method for Physical Edge Match Examinations
8.3.5. Test Method for Glass Comparison

8.4. Instruments:

8.4.1. Stereomicroscope – a microscope which gives a "three dimensional view" capable of examining samples up to a magnification of approximately 90x. Stereomicroscopical observations (e.g., fracture lines, transparency, sharp edges) provide initial and discriminating information for selecting fragments to identify as glass and then to use for glass comparisons.

8.4.2. Calipers – an instrument capable of accurately and precisely measuring the thickness of different samples. It can be used to measure the thickness of glass when both edges are present.

8.4.3. Polarized Light Microscope (PLM) - a microscope which can be used to evaluate the properties of glass samples. Characterization and identification of glass is accomplished with the use of PLM.

8.4.4. UV Box – an instrument designed to allow for the viewing of specimens in short and long wavelengths of light. It can be used to look at the fluorescence of float glass and also coatings on glass.

8.4.5. Ohm meter – an instrument capable of testing conductivity.

8.5. Reagents/Materials: Not applicable

8.6. Hazards/Safety:

8.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as "Biohazard" shall be handled with gloved hands during the entire examination process.

8.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling occurs first, the forensic scientist shall still take precautions during examination for potential additional DNA analysis in the future. During the examination the

forensic scientist shall wear a lab coat, use a disposable face mask, and change gloves after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

8.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to prevent obliterating fingerprints and/or depositing additional prints.

8.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

8.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS's should be consulted.

8.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

8.6.7. Care shall be taken to prevent lacerations from glass samples.

8.7. Reference Materials/Controls/Calibration Checks:

8.7.1. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

8.7.2. Any measuring device (tape measure or ruler) will be evaluated appropriately.

8.7.3. The Digital Micrometer shall be performance checked as required in Appendix 13.

8.8. Procedures/Instructions:

This section provides an overview of suggested flow of analytical techniques to be utilized for the analysis of glass for physical end matching, direction of force, and sequencing. The exact selection and order of methods depends upon the sample size and/or condition.

8.8.1. **Initial Evaluation and Handling** Due to concerns with the handling of broken glass as physical evidence, each case shall be evaluated to select appropriate procedures concerning the sampling size, collection, preservation, and order of examinations.

8.8.1.1. Different forensic disciplines may be called upon to examine the same item of evidence. The order in which the examinations shall be conducted needs to be resolved on a case-by-case basis. The order of examinations shall be

selected and conducted so as to preserve the most transient evidence and provide the greatest discrimination and most valuable information. The forensic scientist shall make the submitting agency aware of the effects that some disciplines' processing and examinations may have upon other specific examination requests.

8.8.1.2. Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics. Preliminary examination shall include its general appearance macroscopically and under a stereomicroscope.

8.8.1.3. Ideally the item of evidence shall be preserved in a manner that does not interfere with future testing. All procedures shall be conducted in such a manner to ensure that no cross-contamination occurs. The item shall be photographed or described prior to conducting any destructive analyses in order to provide documentation of original condition.

8.8.1.4. Glass sources sometimes have other materials such as paint, decals, or tint films associated with them. These materials may be transferred with glass fragments and may increase the evidentiary value of the glass. The presence of these materials can also be used to physically fit one piece of glass into another.

8.8.2. Physical Edge Matching. A physical edge match may be requested for a submitted glass sample. This procedure is used to characterize the physical properties of a glass sample or an individual glass fragment for comparison of these properties with other samples or fragments.

8.8.2.1. Work area shall be cleaned before and after each use.

8.8.2.2. Record visual observations. These observations include, but are not limited to, color, type, thickness, temper, float properties, shape, inclusions, surface texture, and surface markings. Record the amount of glass received. As applicable, this may be accomplished by indicating either the number of fragments of an approximate size (1 fragment ~1 ¼" x 3", 6 fragments ~ 1/2") or by indicating a general description as to amount present. Examine a sufficient number of fragments in the sample to determine if multiple broken glass sources may be present in the sample.

8.8.2.3. If two parallel manufactured surfaces are present, measure the thickness of the glass sample using a digital caliper. Take a representative number of measurements to determine if a thickness range exists. The maximum, minimum, and number of measurements shall be recorded. The digital caliper used shall be noted.

8.8.2.4. Observe the fractured surfaces of a glass sample for indications of temper. Tempered glass sources typically "dice" when broken, forming approximate cube shaped fragments. The broken surfaces of these fragments are

generally perpendicular to the manufactured surfaces. Other characteristics of tempered glass may include symmetrical, or mirror-imaged, ridges on the broken surfaces, or the presence of a “frost line”, which generally divides the symmetrical ridges in the broken surfaces. For samples of limited size, examination of the fragments with a polarized light microscope (PLM) using crossed polarized light may be useful to determine temper. Tempered glass exhibits internal stresses that can be observed as interference colors that appear to move through the fragment as the PLM stage is rotated.

8.8.2.5. Examine flat manufactured surfaces using long and short wavelength UV. If a flat glass sample exhibits a marked fluorescence on one side and not on the other, it is determined to be a float glass. Record whether the sample is a float or a non-float glass. Glass samples may also exhibit an overall fluorescence rather than only on the float surfaces.

8.8.2.6. If it is readily apparent that a float glass is present in the case samples, then proceed with the determination of physical properties.

8.8.2.7. If the glass sample has a highly reflective but transparent manufactured surface, determine if the glass is from a low emissivity (low e^-) source. Check the continuity tester prior to use and record in your notes. Then press the two probes of the continuity tester against the glass surface without allowing them to touch each other. If the surface conducts electrical current, the sample is a low-E glass. Low e^- glass conducts electrical current on one manufactured surface but not the other. The continuity tester used shall be noted.

8.8.2.8. If two glass samples are fitted together, it shall be documented through imaging, followed by a microscopic examination to confirm the fit.

8.8.2.9. To confirm the physical edge match, the microscopic examination shall be conducted for the identification of microscopic scratches, stains, or any other marks that may begin on the questioned fragment, and then continue on the known fragment. Images shall be taken to document the forensic scientist’s conclusion. The images shall not be considered items of evidence.

8.8.3. Direction of Force. This procedure is used to determine from which side a window pane was broken. For direction of force, the broken pane is reconstructed (if necessary) and, if possible, the point-of-impact and cracks radiating from that point are identified to determine the direction of force.

8.8.3.1. Determining direction of force for a broken pane

8.8.3.1.1. Lay out broken pane fragments on a clean sheet of paper.

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

8.8.3.1.3. Reconstruct the pane as completely as possible.

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

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

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

8.8.3.2. Determining the direction of force when analyzing a laminated pane.

8.8.3.2.1. Examine the laminate for any coning or beveling present and record any observations. The direction of any coning present should indicate the direction of force.

8.8.3.2.2. Observe the displaced glass at the area of impact and record any observations.

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

8.8.3.2.4. Images shall be taken to document the forensic scientist's conclusion. The images shall not be considered items of evidence.

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

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

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

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

8.8.4.4. Images shall be taken to document the forensic scientist's conclusion. The images shall not be considered items of evidence.

8.8.4.5. Determining the type of impact

8.9. Records: Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics.

8.9.1. Preliminary examination of the pane of glass shall include its general appearance macroscopically, including any adhering matter.

8.9.2. Observed characteristics shall be fully documented.

8.10. Interpretations of Results:

8.10.1. The conclusions reached shall be supported by the documented characteristics that are exhibited in the items of evidence. The weight of the conclusions shall be summarized.

8.10.2. If conflicting characteristics exist, all of the characteristics shall be noted and summarized.

8.10.3. All conclusions shall be verified by a second qualified examiner. This verification shall be documented in the case notes.

8.11. Report Writing:

8.11.1. If a direction of force determination can be made, the results shall describe the determination made, in detail.

8.11.2. If sequencing of multiple impacts is possible, the results shall describe the determination made, in detail.

8.12. References

8.12.1. Forensic Examination of Glass and Paint – Brian Caddy – *Chapter 6, Interpretation of Physical Aspects of Glass Evidence* – John I. Thornton p. 97 - 121

8.12.2. Glass Fracture Documentation at the Scene and Reconstruction Techniques – Keith Webb, Houston Police Department

8.12.3. SWGMAT Glass Fractures – Forensic Science Communications January 2005 – Volume 7 – Number 1

9. Lamp Examinations

9.1. Scope:

This test method is for the examination of lamp filaments to determine if they were lighted or unlighted at the time of an impact. This test method provides an overview of techniques applied to the analysis of lamp filaments. Methods for the analysis of lamp filaments include examination of physical characteristics observable both macroscopically and with a stereomicroscope and the testing of filament continuity with an ohm meter. The forensic scientist has a familiarity with the techniques used in the methods described. The methods and practices described have been published, peer-reviewed, and are generally accepted within the forensic community.

9.2. Precautions/Limitations:

9.2.1. The analysis of lamp filaments in the forensic science laboratory can provide valuable information due to the characteristics that may be imparted on a filament during a collision or impact. Any lamp having a tungsten filament which has been involved in a collision or has been impacted may yield some information; however, there may not always be sufficient information to make a definitive conclusion.

9.2.2. Lamps not containing tungsten filaments (such as neon lamps and light emitting diodes) are not able to be analyzed by the methods described. These types of lamps do not exhibit any reproducible characteristics when involved in a collision or impact.

9.2.3. A lamp submitted for filament examination should be packaged in a sealed and labeled container in such a way that filaments are not lost, damaged, or compromised for analysis. There is a possibility that a lamp could be damaged in a prior event or damaged in removal and transport for the analysis. In these cases a conclusion shall be qualified. Lamps submitted for analysis are to be packaged separately.

9.3. Related Information:

9.3.1. Appendix 1 Worksheets
9.3.2. Appendix 2 Abbreviations
9.3.3. Appendix 3 Definitions

9.4. Instruments:

9.4.1. Stereomicroscope – a microscope which gives a “three dimensional view” capable of examining lamps up to a magnification of approximately 90x.

9.4.2. Ohm meter – an instrument capable of testing the conductivity of filaments.

9.4.3. Scanning Electron Microscope with energy dispersive spectroscopy (SEM/EDS) – an analytical instrument capable of chemically analyzing any precipitates found on

or around a filament.

9.5. Reagents/Materials:

9.5.1. Disposable gloves
9.5.2. Laboratory coat
9.5.3. Tweezers
9.5.4. Probes
9.5.5. Glass scorer
9.5.6. Dremel tool
9.5.7. Electrical charcoal starter loop
9.5.8. Hammer
9.5.9. Pliers

9.6. Hazards/Safety:

9.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as “Biohazard” shall be handled with gloved hands during the entire examination process. Items handled with gloves shall be disinfected prior to handling with bare hands.

9.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling occurs first, the forensic scientist shall still take precautions during examination for potential additional DNA analysis in the future. During the examination the forensic scientist shall also wear a disposable face mask. Gloves shall be changed after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

9.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to minimize additional fingerprints, or obliterating existing fingerprints.

9.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

9.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS’s shall be consulted.

9.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

9.7. Reference Materials/Controls/Calibration Checks:

9.7.1. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

9.7.2. Known reference lamps may be used for comparison. These lamps may be purchased from any retail lamp distributor.

9.7.3. The Ohm meter shall be performance checked as required in Appendix 15.

9.7.4. The SEM Instrument shall be performance checked as required in Appendix 5.

9.7.5. In the event that performance checks are found to be unsatisfactory, the instrument shall be taken out-of-service and measures taken to restore the instrument to proper working order.

9.7.5.1. Any instrument that is out-of-service shall be visibly marked.

9.7.5.2. When an instrument is taken out-of-service for maintenance and/or repair, performance checks shall be performed prior to resuming casework on that instrument.

9.7.5.3. An infrequently used instrument may be placed in an "Inactive" status and the normal verification procedures may be suspended. Normal quality control procedures shall be resumed prior to use in casework analysis.

9.8. Procedures/Instructions:
This section provides an overview of suggested flow of analytical techniques to be utilized for the analysis of lamps to determine if they were lighted or unlighted at the time of an impact. The exact selection and order of methods depends upon the sample condition.

9.8.1. Initial Evaluation and Handling The evidence shall be marked in accordance with the Quality Assurance Manual. A systematic approach shall be used for the vehicle lamp examination, with findings and observations recorded in the case record.

9.8.1.1. Different forensic disciplines may be called upon to examine the same item of evidence. The order in which the examinations shall be conducted shall be resolved on a case-by-case basis. The order of examinations shall be selected and conducted so as to preserve the most transient evidence and provide the greatest discrimination and most valuable information. The forensic scientist shall make the submitting agency aware of the effects that some disciplines' processing and examinations may have upon other specific

examination requests. If another discipline is chosen before the lamp examination the possible effects on the evidence shall be considered.

9.8.1.2. Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics. Preliminary examination shall include its general appearance macroscopically and under a stereomicroscope, including any physical damage.

9.8.1.3. Ideally the item of evidence shall be preserved in a manner that does not interfere with future testing. The item shall be photographed or described prior to conducting any destructive analyses in order to provide documentation of original condition.

9.8.2. General Lamp Examination Procedure:

9.8.2.1. Classify the lamp as to the brand, type, base arrangement, bulb shape, electrical rating (voltage/wattage), filament configuration, and usage in the vehicle if possible.

9.8.2.2. Document the physical appearance of the lamp through imaging (and sketches if appropriate).

9.8.2.3. Note the following:

9.8.2.3.1. Physical condition of the glass bulb (color, breakage, discoloration, etc.).

9.8.2.3.2. Physical condition of the base (substrate, markings, etc.).

9.8.2.3.3. Physical description of the post(s) to include color and luster of the metal and general appearance.

9.8.2.3.4. Physical appearance of the filament(s) to include color, luster, or metal, descriptions of the coil spacing and any out of plane bending.

9.8.2.3.5. Presence or absence of any discoloration of the metal and/or oxide deposits on posts or filaments.

9.8.2.3.6. Presence or absence of age sag and pitting of metal.

9.8.2.3.7. Presence of cold fracture or burn out.

9.8.2.3.8. Presence of molten or melted glass.

9.8.2.3.9. Any other physical phenomena observed.

9.8.2.4. Determine, using the Ohm meter, whether each filament is operational.

9.8.2.5. If observations are inconsistent, it may be advantageous to use comparison lamps to show similarities or differences in lamp configurations.

9.8.2.6. It may be necessary to open the lamp to better visualize the filament(s) being examined, as described later in this test method. Photos shall be taken of the lamp to be opened in its original condition. Continuity testing shall be conducted with an Ohm meter before opening any lamps.

9.8.2.6.1. Opening a Small Lamp for Examination

9.8.2.6.1.1. Wear protective eye coverings.

9.8.2.6.1.2. Using a glass scribe, etch all the way around the glass bulb to stress the glass.

9.8.2.6.1.3. Place the lamp in a pill box covered with a clean piece of paper.

9.8.2.6.1.4. Place the lamp into the mouth of an adjustable wrench. Tighten the wrench until the pressure exerted on the glass causes it to break.

9.8.2.6.1.5. Remove the lamp from the adjustable wrench. Turn the lamp upside down over the pill box to catch any material that has fallen into the base.

9.8.2.6.1.6. Remove any sharp edges of glass with a pair of needle nosed pliers.

9.8.2.6.1.7. Note the current condition of the filament and compare it to how it appeared prior to opening the lamp.

9.8.2.6.1.8. Record the method of opening, findings, and observations in the notes by documenting and/or photographing.

9.8.2.6.2. Opening a Sealed Beam Lamp for Examination

9.8.2.6.2.1. Using a glass scorer, make a score mark around the lugs on the reflector in a circular pattern.

9.8.2.6.2.2. Hold the hot electrical charcoal starter loop on the scored circle.

9.8.2.6.2.3. If the reflector glass has not yet cracked, place water drop wise where the reflector and the electric charcoal starter meet. The reflector will crack along the score line with an audible click.

9.8.2.6.2.4. Rotate the lamp slowly repeating the previous step until the crack as gone all the way around the reflector. Then remove the

electric charcoal starter.

9.8.2.6.2.5. Grasp one of the lugs with pliers and tap the reflector on the scored, cracked line with a small hammer or pliers to separate the part of the reflector attached to the filament from the lens.

9.8.2.6.2.6. Record the method of opening, findings, and observations in the notes by documenting and/or imaging.

9.8.2.6.3. Opening a Plastic Sealed Tractor/Trailer Light

9.8.2.6.3.1. Using a Dremel Tool with a sanding disc attachment, sand the plastic area where the lens and the back of the light meet until the plastic is melted.

9.8.2.6.3.2. Continue this all the way around the light until the lens is able to be removed from the back of the light.

9.8.2.6.3.3. Use wire cutters to cut the wires attached to the lamp backing. Use scissors to remove the lamp from the lamp backing.

9.8.2.6.3.4. Place the lamp in a pill box for storage and analysis.

9.8.2.6.3.5. Record the method of opening, findings, and observations in the notes by documenting and/or imaging.

9.9. Records: Written descriptions, sketches, photography, or other imaging methods shall be used to document the characteristics of each item.

9.9.1. Examination of a vehicle lamp and filament(s) shall include appearance macroscopically and under a stereomicroscope.

9.9.2. The case record shall include the instrumental data. All hard copies shall include a unique sample designation, the operator's name/initials, and the date of analysis. Case notes shall also include a description of the evidence analyzed and the methodology used for analysis.

9.10. Interpretations of Results:

9.10.1. All interpretations of results to include lamp description, observations, and conclusions shall be recorded in notes.

9.10.2. The filament was definitely “on” at or near the time of an impact.

9.10.2.1. Minimum requirements include a broken glass bulb with filament distortion and the presence of oxidation and/or fused glass on the filament. There could also be white tungsten oxide deposits on the colder surfaces (posts, other filament).

9.10.2.1.1. Note: when the glass bulb is broken there is the possibility of mechanical stretching of the filament. As a result it is important that one of the other characteristics listed in the paragraph above is present to make this determination.

9.10.3. The filament exhibits characteristics that indicate it was "on" at or near the time of an impact.

9.10.3.1. Minimum requirements include any combination of the following characteristics: significant filament distortion (hot stretch) with or without a broken filament with one beaded end and one tapered end (burn out.)

9.10.4. The filament exhibits characteristics that indicate it was "off" at or near the time of impact.

9.10.4.1. Minimum requirements include a broken filament with jagged ends (cold fracture) or a broken filament with one beaded end and one tapered end and no filament distortion.

9.10.5. The filament does not exhibit any characteristics that indicated whether it was "on" or "off" at the time of impact.

9.10.5.1. The lamp appears to be a normal, functioning lamp and/or there is insufficient distortion to make a determination.

9.11. Report Writing:

9.11.1. Examination of the lamp from (Item #) revealed physical characteristics consistent with it having been lighted when it was subjected to impact shock and the glass envelope was broken.

9.11.2. Examination of the lamp from (Item #) revealed physical characteristics that indicate it was lighted when it was subjected to impact shock.

9.11.3. Examination of the lamp from (Item #) revealed it not to have been lighted when it was subjected to impact shock.

9.11.4. Examination of the lamp from (Item #) revealed it to be a normal, functioning lamp. There were no physical characteristics observed to indicate this lamp was "on" or "off" at the time of vehicle impact.

9.12. References:

9.12.1. Badger, Joseph E., "Casting New Light On Lamp Investigation", Law Enforcement Technology, September 1989, pp. 38-39, 53

9.12.2. Baker, J.S., Aycock, T.L., and Lindquist, T., Lamp Examination for On or Off in Traffic Accidents, Northwestern University Traffic Institute, 1993

9.12.3. Rioux, J.M., Murphy, K.J., Stone, H.S, and Stuart, A.W., "Determination of the Cooling Rate For an Incandescent Headlamp Filament", Can. Soc. Forensic Sci. J., Vol. 30, No. 2, 1997, pp. 61-64

9.12.4. Saferstein, R., Forensic Science Handbook, Chapter 4, "Forensic Glass Comparisons", pp. 139 – 182, 1982

9.12.5. Stauffer, Eric, M.S., "Interpretation of Automotive Light Bulb Examination Results: An Intriguing Case," J. Forensic Sci., January 2007, Vol. 52, No.1, 119 – 124

9.12.6. Tobin, William A., "Noninvasive Evaluation of Vehicular Lamp Bulbs", Crime Laboratory Digest, Vol. 21, No. 1, January 1994, pp. 5-7

9.12.7. Traffic Collision Investigation, "Chapter 7 – Lamp Examination for "on" or "off" in Vehicle Collisions," 2001 Northwestern University Center for Public Safety, Evanston, IL, pp. 301-365

10. Fiber Examinations

10.1. Scope:

This test method is for the analysis, examination, and comparison of fibers. The scope of this type of evidence includes but is not limited to textiles, apparel, ropes and cordage and carpeting. Typically, a fiber examination involves the comparison of samples to determine if they could share a common origin. The goal is to determine if any significant differences exist between the samples. The evaluation of fibers for class characteristics can associate known and questioned fibers to a group but not to a single, individual source. A physical edge match of two pieces of cloth or rope provides individualizing characteristics that associate the two pieces of rope or cloth to one another. The following methods and practices have been published, peer-reviewed, and are generally accepted within the forensic community.

10.2. Precautions/Limitations:

10.2.1. The analysis and comparison of fiber evidence in the forensic science laboratory can provide valuable information due to the variability of fibers. Because of the unknown characteristics of all of the fibers present in an environment and the unknown properties of the manufacture of additional fibers, it is not possible to establish the statistical probability that a given sample would have the same physical, optical and chemical characteristics as a randomly selected fiber sample.

10.2.2. The microscopic comparison of human hair and the species identification for animal hairs is not conducted at the Indiana State Police laboratory.

10.2.3. Fibers by nature can be transient. Submitted items may have fibers adhering as evidence that are extremely small. Care shall be taken in packaging items of evidence and in the collection of evidence to help ensure that fibers are not transferred. Care should also be taken to eliminate the possibility of introducing common contaminants which can include fibers.

10.2.4. An item where fibers may be present should be packaged in a sealed and labeled container in such a way that the fibers are not lost, transferred, or compromised for analysis. When individual fibers are recovered, the fibers should be appropriately packaged. (i.e. put into a paper bindle then placed into a properly sealed and identified pill box) Fiber standards need to be packaged separately and should be submitted with each case and packaged accordingly.

10.3. Related Information:

10.3.1. Appendix 1 Worksheets
10.3.2. Appendix 2 Abbreviations
10.3.3. Appendix 3 Definitions
10.3.4. Test Method for Physical Edge Match Comparisons

10.4. Instruments:

10.4.1. Stereomicroscope – a microscope which gives a “three dimensional view” capable of examining samples up to a magnification of approximately 90x.

Stereomicroscopical observations provide initial and discriminating information for selecting fibers to use for fiber comparisons.

10.4.2. Polarized Light Microscope (PLM) - a microscope which can be used to evaluate the properties of fibers. Characterization and identification of fibers is accomplished with the use of PLM.

10.4.3. UV Box – an instrument designed to allow for the viewing of specimens in short and long wavelengths of light. It can be used to look at the fluorescence of fibers.

10.4.4. Fourier Transform Infrared (FTIR) Spectrometer– an analytical instrument capable of analyzing the molecular vibrations of the chemical components of fibers.

10.4.5. Microspectrophotometer – an analytical instrument capable of characterizing color and fluorescence of the fiber as well as its properties under ultraviolet light.

10.4.6. Microtome – an instrument for cutting thin cross sections of specimens. It can be used to make cross sections of fibers.

10.4.7. Comparison Microscope - a microscope capable of simultaneous microscopic examinations of two different items.

10.4.8. Hot stage Microscope - a microscope capable of determining the melting point of fibers.

10.5. Reagents/Materials:

10.5.1. Microscope slides
10.5.2. Microscope cover slips
10.5.3. Suitable solvents
10.5.4. Mounting medium
10.5.5. Cargille refractive index liquids (1.3 to 1.8)
10.5.6. Hotplate
10.5.7. Scalpel or razor blade
10.5.8. Low e^- microscope slide
10.5.9. SEM stubs
10.5.10. Roller knife
10.5.11. Disposable syringes
10.5.12. Disposable gloves
10.5.13. Laboratory coat
10.5.14. Spoon/watch glass
10.5.15. Beakers
10.5.16. Test Tubes
10.5.17. Tweezers
10.5.18. Probes
10.5.19. Dropper bottles

10.5.20. Polypropylene or polyethylene bag.

10.6. Hazards/Safety:

10.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as "Biohazard" shall be handled with gloved hands during the entire examination process. Items handled with gloves shall be disinfected prior to handling with bare hands.

10.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling occurs first, the forensic scientist shall still take precautions during examination for potential additional DNA analysis in the future. During the examination the forensic scientist shall also wear a disposable face mask. Gloves shall be changed after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

10.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to minimize additional fingerprints, or obliterating existing fingerprints.

10.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

10.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS's shall be consulted.

10.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

10.7. Reference Materials/Controls/Calibration Checks:

10.7.1. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

10.7.2. Questioned and known samples shall be analyzed under similar conditions.

10.7.3. The SEM Instrument shall be performance checked as required in Appendix 5.

10.7.4. The FTIR Instrument shall be performance checked as required in Appendix 4.

10.7.5. The melting point microscope shall be performance checked as required in Appendix 11.

10.7.6. The Microspectrophotometer Instrument shall be performance checked as required in Appendix 10.

10.7.7. The Polarized Light Microscope shall be performance checked as required in Appendix 14.

10.7.8. In the event that performance checks are found to be unsatisfactory, the instrument shall be taken out-of-service and measures taken to restore the instrument to proper working order.

10.7.8.1. Any instrument that is out-of-service shall be visibly marked.

10.7.8.2. When an instrument is taken out-of-service for maintenance and/or repair, performance checks shall be performed prior to resuming casework on that instrument.

10.7.8.3. An infrequently used instrument may be placed in an "Inactive" status and the normal verification procedures may be suspended. Normal quality control procedures shall be resumed prior to use in casework analysis.

10.7.9. Reference Materials and Controls:

10.7.9.1. Fibers: Sufficient reference materials of common fibers shall be maintained in the laboratory reference collection for comparison purposes.

10.7.9.2. Animal Hairs: Sufficient reference materials of known hairs from various species of animals shall be maintained in the laboratory reference collection for comparison purposes.

10.7.10. Following the microscopic examination, the slides should be packed in slide mailers for protection. The mailer shall be sealed and marked and then placed with the rest of the evidence. The forensic scientist's initials shall be marked across the seal.

10.8. Procedures/Instructions:

This section provides an overview of suggested flow of analytical techniques to be utilized for the analysis of fibers. The exact selection and order of methods depends upon the sample size, type of fiber, and/or condition.

10.8.1. Analytical Policy

10.8.1.1. Fibers received as physical evidence by the ISP Laboratory shall be analyzed by a variety of microscopic, chemical, and instrumental techniques.

10.8.1.2. The classification of a manufactured fiber shall be confirmed by at least two different methods.

10.8.1.3. Fiber examinations shall typically be conducted only when an appropriate known sample has been submitted.

10.8.1.4. The known or control sample shall be analyzed in a like manner to questioned samples.

10.8.1.5. Additional laboratory techniques may be employed when deemed necessary by the forensic scientist and approved by the Unit Supervisor.

10.8.1.6. The forensic scientist shall retain the actual sample analyzed.

10.8.2. Initial Sample Treatment

10.8.2.1. Upon receiving evidence for fiber examination, label the outer container of the evidence with case number, item number, examiner's initials, and PE number.

10.8.2.2. Clean the examination surface and spread a clean piece of examination paper out on the examination surface. Avoid drafts around the examination area.

10.8.2.3. Open the container, avoiding the breakage of seals when possible. Remove the evidence and, when possible, mark it with the case number, item number, and your initials.

10.8.2.4. A visual examination of the evidence is conducted at this point. A careful visual search and collection procedure is conducted for fiber evidence and/or other debris which is easily observed. Any visible fibers or debris shall be removed from the item with tweezers and placed into a paper bindle or pill box for preservation and possible analysis. If pieces of fabric are present, the weave or knit pattern and fiber composition should be characterized. Care shall be taken so as not to lose fibers from the item being examined.

10.8.3. Evidence Collection

10.8.3.1. After any visible fiber evidence and/or debris are removed from the item of evidence an acceptable method of evidence collection shall be used. This includes, but is not limited to, tape lifting, vacuuming, and scraping.

10.8.3.2. Tape lifting: A roll of clear plastic tape on a tape roller is folded back upon itself exposing the adhesive side of the tape. The tape is then systematically rolled over the item being examined. Fibers and other debris will adhere to the adhesive on the tape. Once no more debris is adhering to the tape, the tape is removed from the roll and placed onto a clear plastic page protector for analysis. The page protector is heat sealed to protect and preserve the evidence on the tape lift. This process is repeated until the fibers and debris from the entire item of evidence have been collected on the tape lifts. The tape lifts are searched by use of a stereomicroscope for fibers of interest. Fibers may be removed from the tape using an appropriate tool of choice (i.e. tweezers, probe). When necessary, a solvent can be used to remove the adhesive from the fiber.

10.8.3.3. Vacuuming: An approved vacuum fitted with an evidence capturing filter may be used to collect hairs, fibers, dust, and debris from a particular item. This method can become cumbersome in most cases because of the sheer volume of debris it collects. In certain limited cases (such as cases involving large amounts of debris where a tape lifter would be ineffective), using a vacuum may be the preferred method of collection.

10.8.3.4. Scraping: The item to be examined is suspended above the examination surface and gently scraped with a spatula. Scraping in a downward direction allows surface hairs, fibers, and debris to fall onto the examination surface. To store and preserve the evidence, the fibers and debris can be collected using an adhesive tape or transferred to a pillbox. The item shall not be scraped too vigorously as debris can become airborne and lost. Take appropriate precautions when collecting debris from heavily bloodstained articles this includes but is not limited to wearing a disposable lab coat, face mask, and goggles.

10.8.4. Examination Methodology:

10.8.4.1. Upon collecting debris from an item of evidence take an inventory of the debris through the use of a stereomicroscope. This includes, but is not limited to items such as hair, fibers, soil, vegetable material, mineral aggregate, and paint chips.

10.8.4.2. If a fiber of interest is found clean the fiber to remove any foreign material which may be adhering to it. Water, methanol, xylene substitute, or other solvents may be employed to clean a dirty fiber. However, a color comparison of a questioned fiber to standard fibers may need to be performed prior to cleaning since certain solvents (such as xylene substitute) may remove dye from fibers.

10.8.4.3. Look for the presence of any foreign material that may warrant examination by a different unit within the laboratory (notify the appropriate unit if such debris is found).

10.8.4.4. Prepare temporary or permanent mounts of the standard and questioned fibers.

10.8.4.4.1. Acceptable mounting mediums include, but are not limited to Refractive index oils (RI = 1.500 – 1.700), glycerine (RI = 1.47), Norland Optical Adhesive 60® (R.I. = 1.560), and Norland Optical Adhesive 65® (R.I. = 1.524).

10.8.4.5. View the questioned and known fibers using a polarized light microscope (PLM) and take note of the fiber's physical and optical properties.

10.8.4.5.1. Observations should include, when appropriate: type of fiber (i.e. animal, synthetic, vegetable), color, diameter, cross section, delusterant, relative refractive index and sign of elongation.

10.8.4.5.1.1. The sign of elongation is determined by viewing the retardation colors of the fiber when a compensator is inserted into the microscope. The fiber has a positive sign of elongation if the fiber has a higher interference color when it is aligned parallel to the compensator and it is negative if the fiber has a lower interference color when it sits perpendicular to the compensator.

10.8.4.5.1.2. Relative refractive index is determined by observing the Becke line's direction of movement in relation to the mounting medium and the fiber. The Becke line will move into the medium that has the higher refractive index. A medium with a refractive index of 1.52 to 1.56 can provide the most useful information for comparisons.

10.8.4.5.1.2.1. A more specific refractive index can be determined by changing the oils based on the Becke line's direction of movement in both the perpendicular and parallel positions of extinction. The fiber shall be mounted in different oils in a systematic fashion until the Becke line or fiber's edge becomes faint or disappears. The disappearance of the fiber edge indicates that the oil and the fiber have the same refractive index.

10.8.4.6. The microscopic characteristics of the questioned fiber shall be compared to the known to determine if they are similar. If any substantive characteristics are found to be different, the examination shall be concluded.

10.8.4.7. If the microscopic characteristics of the questioned fiber are similar to the microscopic characteristics of the standard, then a comparison microscope shall be used to perform a side-by-side comparison of the fibers.

10.8.5. Cross-Sectioning - polyethylene bag method

10.8.5.1. Procedure:

10.8.5.1.1. Using the stereomicroscope, place the fiber on two or more sheets of polypropylene (PP) or polyethylene (PE) (e.g. zip-lock plastic bag) and cover with two or more sheets of PP/PE.

10.8.5.1.2. Melt the PP/PE between two microscope slides at a low heat setting. Some pressure may need to be applied to the slides during the process to remove any pockets of air that may be forming. This is usually done with a pencil eraser.

10.8.5.1.3. Slowly remove the PP/PE when it has cooled to room temperature.

10.8.5.1.4. Using a microtome cut thin cross sections of the fiber. A microtome is the preferable tool, however, a scalpel or razor blade are acceptable tools to perform the cross section.

10.8.5.1.5. Mount the cross sections on a microscope slide using an oil or permanent mount and observe the cross-sectional shape using the PLM.

10.8.5.1.6. Compare the cross sections of the known and unknown fibers using the comparison microscope to provide a split-field, superimposed or right/left field of view.

10.8.5.1.7. It may be necessary to take multiple cross sections of some types of fibers (regenerated cellulosic fibers) to establish a range of diameter and shape.

10.8.5.2. Cross-Sectioning - SEM Method

10.8.5.2.1. Procedure:

10.8.5.2.1.1. Laterally mount the sample on a scanning electron microscope sample stub using double sided carbon tape or another appropriate mounting medium.

10.8.5.2.1.2. Load the mount into the SEM chamber and saturate the filament using an accelerating voltage (i.e. ≤ 10 keV) and spot size (i.e. ≤ 60) that will minimize charging and damage to the fiber A working distance of approximately 8mm to 10mm will obtain the best image (refer to SEM handbook for reference).

10.8.5.2.1.3. Acquire images of the fiber at low magnifications (30X to 200X). This will provide resolved images of a fiber's cross section that

could not be seen with an optical microscope (i.e. fiber damage).

10.8.5.2.2. A PP/PE cross section prepared using the procedure described above can also be used at the discretion of the forensic scientist. This may provide a more detailed image of a cross section and provide elemental information on the fiber's delusterant.

10.8.6. Fourier Transform Infrared Spectroscopy – FTIR

10.8.6.1. Procedure:

10.8.6.1.1. Using the roller knife, flatten the fiber on the mirrored side of the low e^- microscope slide.

10.8.6.1.2. Select the microscope method and detector of choice (narrow band or wide band) on the FTIR. The wide band detector has the benefit of a wider spectral response range (4000 cm^{-1} to 450 cm^{-1})

10.8.6.1.3. A background scan shall be performed before the fiber is scanned.

10.8.6.1.4. The fiber may need to be thinner if the spectrum is not resolved or has an unacceptable quality

10.8.6.1.5. Compare the spectrum from the known and unknown fibers to conclude similarity or differences. If necessary, the fiber library can be searched to confirm the type of fiber.

10.8.7. Microspectrophotometry (MSP)

10.8.7.1. Procedure:

10.8.7.1.1. Turn the system on and allow a sufficient period of time for the MSP to warm up and stabilize. Performing a check of the instruments calibration shall not take place until this condition is met.

10.8.7.1.2. Adjust the field diaphragm to assure that it is in focus and maximized for the field of view.

10.8.7.1.3. Follow the microspectrophotometer manual for calibration of instrument and collection of reference and background spectra.

10.8.7.1.4. Mount the fiber using a microscope slide, cover slip and a suitable mounting medium. Glycerin is the preferred mounting medium and shall be used for fluorescence.

10.8.7.1.5. Adjust the aperture relative to the size of the fiber and begin to collect transmission (or reflectance) and fluorescence spectra. (Refer to microspectrophotometer manual for reference).

10.8.7.1.6. Several spectra shall be obtained along the length of the fiber to demonstrate any variation in color intensity.

10.8.7.1.7. Compare the spectra from the questioned fiber to the spectra from the standard.

10.8.8. Melting Point Determination

10.8.8.1. Procedure:

10.8.8.1.1. Determining the melting point of a fiber is a destructive test and under most circumstances the fiber standard shall be analyzed first. This will help establish a potential starting temperature for the unknown fiber.

10.8.8.1.2. Mount the fiber using a microscope slide and cover slip without any mounting medium.

10.8.8.1.3. Place the slide into the hot stage and under crossed poles orient the fiber to its brightest point of illumination.

10.8.8.1.4. Heat the stage to within approximately 30°C of a potential melting point (reference a fiber melting point chart).

10.8.8.1.5. Program a temperature range and heat rate into the temperature monitoring system with the heat rate decreasing as the fiber begins to reach a potential melting point (i.e. 10°C/min →1.0°C/min →0.5°C/min).

10.8.8.1.6. Record the temperature at which the fiber begins to melt and the temperature at which the fiber is completely melted. The fiber is considered completely melted when it is observed to be white (first order) under crossed polars.

10.8.8.1.7. Compare the melting point range of the questioned fiber to the melting point range of standard fibers.

10.8.9. Following the microscopic examinations:

10.8.9.1. The permanently mounted slides shall be packed in slide mailers for protection. The mailer shall be sealed, marked, and then placed with the rest of the evidence. The examiner's initials shall be marked across the seal.

10.8.9.2. The fibers that are not in a permanent mount shall be transferred to a paper bindle and placed in appropriate packaging (i.e. pill box). The pillbox shall be sealed, marked, and then placed with the rest of the evidence. The examiner's initials shall be marked across the seal.

10.9. Records:
Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics.

10.9.1. Preliminary examination of fibers shall include its general appearance macroscopically and under a stereomicroscope, including any adhering matter.

10.9.2. The case record shall include the instrumental data. All hard copies shall include a unique sample designation, the operator's name/initials, and the date of analysis. Case notes shall also include a description of the evidence analyzed, the method of sample preparation, the analytical instrumentation used, and its operating parameters.

10.10. Interpretations of Results:

10.10.1. Typically, a fiber examination involves the comparison of samples to determine if they could share a common origin. The goal is to determine if any significant differences exist between the samples. The physical and chemical characteristics are evaluated to associate the question and known sample. If a significant difference in a characteristic exists the samples are excluded as having a common origin. If the examination doesn't reveal any significant differences it is concluded that the samples could share a common origin. A limited association can be reported due to the condition/size/type of one of the samples. The evaluation of fibers for class characteristics can associate known and questioned fibers to a group but not to a single, individual source. A true physical edge match of two fabrics or ropes can provide individualizing characteristics that associate the two items to one another to the exclusion of all other items.

10.11. Report Writing:

10.11.1. If significant differences are found in the fiber characteristics, then the report shall state that the fibers are not consistent and did not come from the same source.

10.11.2. If no significant differences are found in the fiber characteristics, then the report shall state that the fibers are consistent and could have come from the same source or another source with these same characteristics.

10.11.3. In rare circumstances the difference in characteristics of the fibers can still be evaluated due to the difference in the present conditions of the fibers. In this circumstance it shall be stated in the report.

10.11.4. Fibers of a ubiquitous nature may not be directly associated with a particular known reference sample (e.g. white cotton, blue denim).

10.12. References:

10.12.1. SWGMAT Trace Recovery Guidelines [Online] (October 1999). Available: http://www.fbi.gov/hq/lab/fsc/backissu/oct1999/index.htm

10.12.2. Polarized Light Microscopy - McCrone - Sections IX and X

10.12.3. Gaudette, Barry D., "The Forensic Aspects of Textile Fiber Examination", Forensic Science Handbook, Volume II, Chapter 5, pp. 209 – 272, 1988

10.12.4. DeForest, P.R., "Foundations of Forensic Microscopy", in Forensic Science Handbook. Vol. 1, 1st ed, Richard Saferstein, 416-528, 1982

10.12.5. Bartick, Edward G. and Tungol, Mary W., "Infrared Microscopy and Its Forensic Applications", in Forensic Science Handbook. Vol. 3, 1st ed, Richard Saferstein, 196-252, 1993

10.12.6. Robertson, James and Greive, Michael, "Forensic Examination of Fibres", (Taylor and Francis Group Publishers), 1992

10.12.7. Moody, Von and Needles, Howard, L, "Tufted Carpet: Textile Fibers, Dyes, Finishes, and Processes", (William Andrew Inc.), 2004

10.12.8. ASTM E2224 Standard Guide of Forensic Analysis of Fibers by Infrared Spectroscopy

10.12.9. ASTM E2225 Standard Guide for Forensic Examination of Fabrics and Cordage

10.12.10. ASTM E2228 Standard Guide for Microscopical Examination of Textile Fibers

11. Ignitable Liquid Examinations

11.1. Scope:

This test method is for the identification of ignitable liquid residues in extracts from fire debris samples. This analytical procedure can be performed on burned fire debris, unburned material, or liquids of any type in which ignitable liquids are suspected. Ignitable liquid residues examinations arise due to suspicious fires in buildings, homes, cars, etc. This test method provides an overview of techniques applied to the analysis of fire debris. The forensic scientist has a familiarity with techniques and instrumentation used in the methods described. The methods and practices described have been published, peer-reviewed, and are generally accepted within the forensic community.

11.2. Precautions/Limitations:

11.2.1. All items requested for ignitable liquid analysis shall be packaged in airtight containers. Failure to do so may result in contamination and/or loss of volatiles.

11.2.1.1. Glass jars with screw caps are acceptable; however, care shall be taken to avoid breakage.

11.2.1.2. Heat sealed nylon or polyester bags (such as Grand River brand bags) are acceptable; however, care shall be taken to avoid puncture.

11.2.2. Microbial degradation, which occurs from bacterial action in soil, can affect ignitable liquid residues in fire debris. The degradation is very selective resulting in the absence of many distinguishing components associated with ignitable liquid residues. In order to minimize the rate of microbial degradation of hydrocarbons in soil, fire debris containing soil should be frozen as soon as possible when it is practical to do so.

11.2.3. Weathering can also affect ignitable liquid residues in fire debris. Weathering can occur when an ignitable liquid is consumed during the fire or when an ignitable liquid evaporates after the fire. Weathered ignitable liquid residues may still be properly classified by comparison to an ignitable liquid reference library containing known weathered samples.

11.2.4. Competitive adsorption can occur in the adsorption-elution extraction technique. Competitive adsorption is when the charcoal strip and the charred debris both have an inherent tendency to adsorb and retain hydrocarbons. It is possible to heat the container for a longer amount of time at a lower temperature if competitive adsorption occurs.

11.2.5. Although the GC/MS is extremely sensitive, there is a limit of detection. An ignitable liquid residue may be present in a sample; however, due to an instrument's detection limits, it may not be able to be identified.

11.2.6. Pyrolysis products that are common in ignitable liquid residue data come from the breakdown of polymers. The background of these pyrolysis products could mask an ignitable liquid residue that may also be present.

11.2.7. Although an extensive ignitable liquid reference library has been created, this library is not all inclusive and might not have a reference sample for comparison to an unknown.

11.3. Related Information:

11.3.1. Appendix 1 Worksheets
11.3.2. Appendix 2 Abbreviations
11.3.3. Appendix 3 Definitions

11.4. Instruments:

11.4.1. Gas Chromatograph/Mass Spectrometer (GC/MS) – An instrument that separates a mixture into its components. The resulting chromatogram shows how much of each singular ion is present, and this is used for identification of ignitable liquid residues.

11.5. Reagents/Materials:

11.5.1. Pentane
11.5.2. Disposable syringes
11.5.3. ASTM E1618 test mixture
11.5.4. Gasoline standard
11.5.5. Activated charcoal strips
11.5.6. Oven
11.5.7. Thermometer
11.5.8. Grand River bags
11.5.9. Metal paint cans
11.5.10. Reference ignitable liquids
11.5.11. Spoon/watch glass

11.6. Hazards/Safety:

11.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as “Biohazard” shall be handled with gloved hands during the entire examination process. Items handled with gloves shall be disinfected prior to handling with bare hands.

11.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling occurs first, the forensic scientist shall still take precautions during examination for potential additional DNA analysis in the future. During the examination the

forensic scientist shall also wear a disposable face mask. Gloves shall be changed after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

11.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to minimize additional fingerprints, or obliterating existing fingerprints.

11.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

11.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS's shall be consulted.

11.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

11.6.7. Intentional inhalation of the odor of the contents of items for ignitable liquid analysis is prohibited due to potential health concerns related to this activity.

11.6.8. Visual examination and extractions shall be performed under proper ventilation.

11.7. Reference Materials/Controls/Calibration Checks:

11.7.1. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

11.7.2. Questioned and known samples shall be analyzed under similar conditions.

11.7.3. Each new lot of charcoal strips is to be checked for contaminants.

11.7.3.1. Pentane – Each new bottle or lot of pentane shall be tested prior to its use in casework. Approximately 500 ml of the new solvent is evaporated to a few drops. 1-2 µl of this evaporated solvent is injected into the GC/MS. If the resulting data has no significant peaks which interfere with the analysis, then the solvent is available for use in casework. The data is labeled with: manufacturer, lot number, start volume, end volume and is placed in a file.

11.7.4. The GC/MS shall be performance checked as required in Appendix 6.

11.7.5. Blanks shall be run before and after each case sample.

11.7.6. In the event that performance checks are found to be unsatisfactory, the instrument shall be taken out-of-service and measures taken to restore the instrument to proper working order.

11.7.6.1. Any instrument that is out-of-service shall be visibly marked.

11.7.6.2. When an instrument is taken out-of-service for maintenance and/or repair, performance checks shall be performed prior to resuming casework on that instrument.

11.8. Procedures/Instructions:
This section provides an overview of analytical techniques to be utilized for the analysis of fire debris. If the exhibit is not a liquid, or "neat" sample, the volatile components are separated from solid debris by one of three techniques: heated headspace, solvent extraction, or adsorption-elution (charcoal strip method). The volatile fraction is examined by temperature-programmed gas chromatography/mass spectrometry.

11.8.1. Initial Evaluation and Handling

11.8.1.1. Different forensic disciplines may be called upon to examine the same item of evidence. The order in which the examinations shall be conducted needs to be resolved on a case-by-case basis. The order of examinations shall be selected and conducted so as to preserve the most transient evidence and provide the greatest discrimination and most valuable information. Forensic Scientist shall make the submitting agency aware of the effects that some disciplines' processing and examinations may have upon other specific examination requests.

11.8.1.2. Label all submitted (and additional packaging if necessary) containers with case number, item number, and your initials.

11.8.1.3. Fire debris samples should be submitted in sealed airtight containers – repackage as necessary at the time of submission.

11.8.1.4. Before beginning analysis, clean off the examination surface and spread a new piece of examination paper out on the examination surface.

11.8.1.5. Briefly open each container. Visually inspect and record a description of the contents. Do not intentionally smell the contents of the container while observing the contents but do make a notation of any unavoidable odor from the contents.

11.8.1.5.1. Observe the contents under proper ventilation.

11.8.1.5.2. The description of the evidence may include the condition of the seal, the container, and the container's labeling.

11.8.1.5.3. If initial examination indicates a strong possible volatile ignitable liquid odor, then heat those containers separately from the containers with no noticeable odor.

11.8.1.6. The forensic scientist shall retain samples.

11.8.1.7. If the autosampler is used, vials shall be recapped and retained.

11.8.2. Heated Headspace

11.8.2.1. Purpose

11.8.2.1.1. The purpose of a heated headspace extraction is to sample volatile components present in the air space above the debris. After heating, the headspace is sampled and injected directly into the gas chromatograph-mass spectrometer (GC/MS).

11.8.2.2. Significance and Use

11.8.2.2.1. This technique is useful for screening fire debris samples to determine possible ignitable liquid class prior to extraction with other techniques.

11.8.2.2.2. This procedure is useful when volatile oxygenated products such as alcohols or lacquer thinners are suspected.

11.8.2.2.3. High concentrations of highly volatile compounds may saturate the headspace, inhibiting the recovery of less volatile components.

11.8.2.2.4. Can follow-up with solvent extraction or adsorption-elution if more complex hydrocarbon mixtures are suspected.

11.8.2.3. Minimum Standards and Controls

11.8.2.3.1. Use a new disposable syringe for each sample and standard injected.

11.8.2.3.2. A syringe/room air blank shall be run prior to each sample. These blank runs shall be satisfactory, having no significant peaks in the region of interest, before injecting case samples.

11.8.2.4. Sample preparation

11.8.2.4.1. Punch or drill a small hole in the container lid and cover the hole with tape.

11.8.2.4.2. If the sample is going to be heated, place the container in an oven until it reaches a temperature no greater than 90 °C.

11.8.2.4.2.1. Forensic scientist shall decide to perform headspace extraction without heating too.

11.8.2.4.3. The syringe may be heated prior to sampling.

11.8.2.5. Sampling Procedure

11.8.2.5.1. Remove the container from the oven and push the syringe through the tape. Slowly pump the syringe three times, withdraw from the lid, and inject 0.5 to 2.0 mL into the GC/MS.

11.8.2.5.2. After the headspace sample has been removed, reseal the hole with tape.

11.8.3. Solvent Extraction

11.8.3.1. Purpose

11.8.3.1.1. The purpose of solvent extraction is to remove petroleum products which may be present in the debris. This extract may be concentrated by evaporation, if needed, and a portion injected into the GC/MS.

11.8.3.2. Significance and Use

11.8.3.2.1. This technique is useful in the event that heated headspace and/or adsorption-elution indicated the presence of a heavy petroleum ignitable liquid residue in a less than conclusive manner.

11.8.3.2.2. Many components from the fire debris are also soluble in pentane and these may interfere with chromatogram interpretation. Generally, this technique is useful for the extraction of nonporous surfaces such as glass or metal.

11.8.3.2.3. This is potentially a destructive technique. Whenever possible, this technique should only be used when a representative portion of the sample can be reserved for future analysis.

11.8.3.3. Minimum Standards and Controls

11.8.3.3.1. Previously cleaned glassware is rinsed with pentane prior to use.

11.8.3.3.2. With this procedure an extraction blank shall be obtained at the same time as the debris extraction. Compare the extraction blank to the debris extract(s).

11.8.3.3.3. If the sample extract is to be concentrated by evaporation, the extraction blank shall be evaporated a similar amount. A copy of the

extraction blank shall be placed in each case file extracted at that time.

11.8.3.4. Procedure

11.8.3.4.1. Select a beaker size which is capable of holding twice the volume of the sample.

11.8.3.4.2. If possible, select a representative portion of the sample to extract.

11.8.3.4.3. Record a description of the portion of the sample extracted.

11.8.3.4.4. Add a sufficient amount of pentane to the debris. The amount needed will vary with the amount and kind of debris: a normal range is 20 – 100 ml. If the debris is non-porous then agitate by gentle swirling to induce further solvent – debris contact. With porous debris, a second (one-size smaller) beaker may be used like a plunger to promote extraction.

11.8.3.4.5. Filter the extract, collecting the filtrate in a suitable small beaker or evaporating dish. (If water is present, separate out using phase separating paper).

11.8.3.4.6. Evaporate the solvent with filtered air to a volume of approximately 1 mL.

11.8.3.4.6.1. The amount of concentrating will be based on the sample.

11.8.3.4.7. Transfer extract into a labeled vial.

11.8.3.4.8. Inject an appropriate volume of pentane into the GC/MS.

11.8.4. Passive Adsorption-Elution

11.8.4.1. Purpose

11.8.4.1.1. The purpose of the passive adsorption-elution extraction is to remove volatile compounds, petroleum products and other ignitable liquids which may be present in the debris and put them into a liquid which can be analyzed via gas chromatography/mass spectrometry (GC/MS). Passive adsorption-elution is also referred to as the charcoal strip method.

11.8.4.1.2. Special considerations:

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

11.8.4.1.2.2. This extraction method may not fully recover petroleum products of low volatility. If a fuel oil or other heavy petroleum product is indicated by the data, then a follow-up solvent extraction may be conducted.

11.8.4.2. Significant and Use

11.8.4.2.1. This technique is useful for preparing extracts from fire debris for later analysis by GC/MS.

11.8.4.2.2. This is a very sensitive separation procedure.

11.8.4.3. Minimum Standards and Controls

11.8.4.3.1. Each time samples are prepared using this method, a method blank will be prepared in the same manner as the evidentiary samples. A copy of the method blank will be placed in each case file extracted that day.

11.8.4.3.2. A new piece of charcoal is used for every case item.

11.8.4.3.3. The oven shall be checked each day of use to ensure it is functioning properly (50 – 85 ^{o}C).

11.8.4.4. Procedures

11.8.4.4.1. The sample shall be in an appropriate fire debris container or placed into one for heating purposes.

11.8.4.4.2. Suspend a pre-cut charcoal strip inside the container via a paper clip and string and close the container.

11.8.4.4.3. Heat the container to a temperature of 50 to 85 °C for 2 to 24 hours

11.8.4.4.3.1. The adsorption temperature and duration may vary based on the sample.

11.8.4.4.3.2. When other evidentiary considerations arise such as document or latent print examinations, it may be appropriate to conduct the adsorption at ambient temperature for extended periods to minimize damage.

11.8.4.4.4. Remove the container from the oven and allow it to cool.

11.8.4.4.5. Remove the charcoal strip from the container and place in a labeled sample vial.

11.8.4.4.6. Add approximately 500 µL of pentane to the vial. A vortex mixer can be used to agitate the sample.

11.8.4.4.7. Inject a portion of the pentane extract into the GC/MS.

11.8.5. Direct Injection

11.8.5.1. When a whole liquid is present, a direct injection may be performed.

11.8.5.2. Procedure

11.8.5.2.1. Nonaqueous liquid samples shall be diluted in pentane and injected directly into the GC/MS.

11.8.6. Flame Test

11.8.6.1. If enough of the liquid exists in the item, a portion shall be analyzed to see if it ignites.

11.8.6.2. Procedure

11.8.6.2.1. The procedure shall be conducted in the hood with no other flammable or combustible materials present.

11.8.6.2.2. A small amount of the liquid is placed in a spoon or on a watch glass.

11.8.6.2.3. A flame source is moved slowly toward the liquid and the ignition (if any) is noted.

11.8.6.2.3.1. The flame (if it is visible) color and if smoke is present shall be noted.

11.8.6.2.3.2. If the liquid boils and does not ignite this shall be noted.

11.8.6.2.4. Some heavy petroleum distillates will not ignite even when an ignition source is placed physically at the surface of the liquid. For these samples a match will sometimes “wick up” and sustain a flame.

11.9. Records: Written descriptions, sketches, or imaging shall be used to document each sample’s characteristics.

11.9.1. Preliminary examination of a fire debris sample shall include its general appearance macroscopically, including amount of sample and presence of noticeable odor.

11.9.2. The case record shall include the instrumental data. All hard copies shall include a unique sample designation, the operator's name/initials, and the date of analysis. Case notes shall also include a description of the evidence analyzed,

the method of sample preparation, the analytical instrumentation used, and its operating parameters.

11.10. Interpretations of Results:

11.10.1. Gas Chromatography/Mass Spectrometry (GC/MS)

11.10.1.1. GC/MS provides a Total Ion Chromatogram (TIC) pattern for the sample as well as the ability to extract patterns for groups of specific ions. Extracted ion chromatography is especially useful when samples show a high degree of debris interference. This test method is used for the identification of single compounds, simple mixtures, or non-petroleum based ignitable liquids.

11.10.1.2. Data Analysis

11.10.1.2.1. Initial data analysis consists of a visual comparison of the total ion chromatograms to reference ignitable liquid chromatograms as described below.

11.10.1.2.1.1. The essential requirement for making a classification using this procedure is the matching of the sample chromatogram with a reference ignitable liquid chromatogram obtained under similar conditions.

11.10.1.2.1.2. Additional data analysis may be carried out using extracted ion profiling (mass chromatography).

11.10.1.2.1.2.1. A data station is used to extract and draw extracted ion profiles for major ions characteristic of each compound type. The following ions are used for the individual classes of hydrocarbons:

11.10.1.2.2. Alkanes: Ions 57, 71, 85, 99
11.10.1.2.3. Aromatics: Ions 91, 105, 119
11.10.1.2.4. Cycloalkanes/cycloparaffins: Ions 55, 69, 83
11.10.1.2.5. Alkylnaphthalenes: Ions 128, 142, 156

11.10.1.2.5.1. Extracted ion profiles for an unknown sample are compared against corresponding extracted ion profiles from reference ignitable liquids. This is generally done by visual pattern recognition.

11.10.2. Flame Test

11.10.2.1. A positive flame test is a definite indication of an ignitable liquid.

11.10.2.1.1. The data analysis from the GC/MS shall be used to classify the ignitable liquid.

11.11. Report Writing:

11.11.1. Petroleum Based Ignitable Liquids Class Designation: Samples demonstrating the presence of a liquid petroleum-based ignitable liquid shall be reported by class as outlined below:

11.11.1.1. Light Petroleum Distillates (LPD)/Products

11.11.1.1.1. Examples: Petroleum ether, some cigarette lighter fluids, some camping fuels

11.11.1.1.2. Example: A light petroleum distillate was identified in item __. Examples of LPDs include, but are not limited to…

11.11.1.2. Medium Petroleum Distillates (MPD)/Products

11.11.1.2.1. Examples: Some charcoal starters, some paint thinners, some cleaning solvents

11.11.1.2.2. Example: A medium petroleum distillate was identified in item __. Examples of MPDs include, but are not limited to…

11.11.1.3. Heavy Petroleum Distillates (HPD)/Products

11.11.1.3.1. Examples: Kerosene (fuel oil #1), Diesel (fuel oil #2), some charcoal starters

11.11.1.3.2. Example: A heavy petroleum distillate was identified in item __. Examples of HPDs include, but are not limited to…

11.11.1.4. Gasoline

11.11.1.4.1. Gasoline is a unique product and shall be named as such in the report.

11.11.1.4.1.1. Example: Gasoline was identified in item __.

11.11.1.4.1.2. Example: The liquid from Item __ was identified as gasoline.

11.11.2. Miscellaneous Compounds

11.11.2.1. Turpentine

11.11.2.1.1. When data suggests compounds present in both wood and turpentine, the examiner may report as follows:

11.11.2.1.1.1. Examination of Item __ revealed the presence of terpenes, which occur naturally in some woods and are also found in turpentine and some cleaning products.

11.11.2.1.1.2. No ignitable liquids were identified in item __ except for compounds consistent with the background material.

11.11.2.1.1.2.1. The forensic scientist shall document exact compounds in notes and not in the report when they are known background compounds.

11.11.2.2. Limited Component Compounds

11.11.2.2.1. Ignitable liquids consisting of single compounds or simple mixtures of compounds shall be reported based upon identification rather than classification.

11.11.2.2.1.1. Example: Item __ contained methyl ethyl ketone.

11.11.2.2.1.2. Example: Acetone was present in item __.

11.11.2.2.2. Some single compounds or simple mixtures are often the result of background interference, pyrolysis, or incomplete combustion of various materials. Therefore, these compounds shall not be reported when they are likely due to the sample matrix unless a qualifier is added.

11.11.2.2.2.1. Example: Toluene was identified in item __; it shall be noted that toluene is commonly used in the manufacturing of shoes.

11.11.2.3. No ignitable liquid residues

11.11.2.3.1. Example: No ignitable liquid residues were identified in item __.

11.11.2.4. Inconclusive

11.11.2.4.1. Inconclusive comparisons shall be reported that no ignitable liquid residues were identified in item __. It shall be documented in the forensic scientist's notes how this conclusion was reached.

11.11.3. Qualifiers

11.11.3.1. The forensic scientist may elect to include a qualifier, or disclaimer to the report.

11.11.3.1.1. If an ignitable liquid was identified, the following disclaimer may be added:

11.11.3.1.1.1. The identification of an ignitable liquid residue in a fire scene does not necessarily lead to the conclusion that a fire was incendiary in nature. Further investigation may reveal a legitimate reason for the presence of ignitable liquid residues.

11.11.3.1.2. If a negative result was obtained, the following disclaimer may be added:

11.11.3.1.2.1. Negative results do not preclude the possibility that ignitable liquids were present at the scene before, during, or after the fire.

11.12. References:

11.12.1. ASTM E1386, Standard Practice for Separation and Concentration of Flammable or Combustible Liquid Residues from Fire Debris Samples by Solvent Extraction

11.12.2. ASTM E1388, Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples

11.12.3. ASTM E1412, Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris by Passive Headspace Concentration with Activated Charcoal

11.12.4. ASTM E1618, Standard Guide for Identification of Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry

12. Clandestine Laboratory Precursor and Reagents Examinations

12.1. Scope:

This is designed for the guidance of laboratory personnel who support investigation of clandestine drug laboratories by analyzing non-drug samples for the presence of chemical reagents used in the illegal manufacture of controlled substances. The scope is limited to chemicals outlined by Indiana Criminal Code IC 35-48-4-14.5 and most frequently encountered in the illegal manufacture of controlled substances, namely, organic solvents, inorganic reagents, and acids and alkalis. Examples include Toluene, camping fuel, Acetone, Red Phosphorus, Iodine, Ammonium Nitrate, Hydrochloric Acid, Sulfuric Acid, and Sodium Hydroxide. This test method provides an overview of techniques applied to the analysis of chemical reagents and precursors in clandestine laboratories. The methods and practices described have been published, peer-reviewed, and are generally accepted within the forensic community.

12.2. Precautions/Limitations:

12.2.1. Clandestine laboratories employ a variety of methods, equipment, precursors, and reagents to illegally produce controlled substances. Clandestine laboratories often times utilize unorthodox, dangerous, and evolving production methods and materials. Realizing the variation in submitted cases, alternative procedures other than those listed may be employed with the approval of the Unit Supervisor.

12.2.2. All chemicals and materials seized from suspected clandestine laboratories may not be able to be fully analyzed by the Indiana State Police laboratory. Chemicals such as elemental lithium, anhydrous ammonia, hydrogen chloride gas, and chemical mixtures that include reaction sludge and hydrogen chloride gas generators possess great chemical hazards as well as analytical limitations and are generally not accepted for analysis.

12.2.3. Red phosphorus may be submitted in a mixture of other inorganic and organic materials or in a purified form. Red phosphorous is found on the strike plates of matchbooks and fusees and is often removed from these sources for the clandestine manufacture of methamphetamine. The purity of red phosphorous on matchbook strike plates has been demonstrated through strike plate analysis to vary widely. Compositions of red phosphorous and silicates are fixed to the strike plate by an adhesive. To release the red phosphorous mixture, alcohol is added to dissolve the adhesive. Recovered red phosphorous may have some organic component with remaining adhesive and also with paper fibers from filters that have been introduced to the red phosphorus. Red phosphorus that has been used with Iodine in clandestine reactions and reclaimed to be used again has demonstrated that a trace amount of Iodine may be detected by SEM-EDS.

12.3. Related Information:

12.3.1. Appendix 1 Worksheets

12.3.2. Appendix 2 Abbreviations
12.3.3. Appendix 3 Definitions
12.3.4. Test Method for General Chemical Comparisons

12.4. Instruments:

12.4.1. Stereomicroscope – a microscope which gives a "three dimensional view" capable of examining samples up to a magnification of approximately 70x. Stereomicroscopical observations (e.g., color, deliquescence, crystal formations) provide initial and discriminating information for selecting fragments to identify.

12.4.2. Polarized Light Microscope (PLM) - a microscope which can be used to evaluate microscopic particles and crystals. Characterization and identification of some materials is accomplished with the use of PLM.

12.4.3. Heating and stirring plate. A device capable of heating to evaporate liquids, sublime iodine, and provide stirring of liquids by means of magnetic stir bars inserted in a vessel.

12.4.4. Reflux column. A water-cooled reflux column may be used to condense Iodine vapors to Iodine crystals.

12.4.5. Gas Chromatograph-Mass Spectrometer – an analytical instrument capable of separating vapor and liquid mixtures into identifiable component compounds. Once separated by the gas chromatograph, the mass spectrometer provides a mass spectrum of the separated compounds.

12.4.6. Scanning Electron Microscope with energy dispersive spectroscopy (SEM/EDS) – an analytical instrument capable of chemically analyzing suspected clandestine materials. It can be utilized for the identification of several inorganic materials including Red Phosphorus, Ammonium Nitrate, and Sodium Chloride.

12.5. Reagents/Materials:

12.5.1. Chemical solvents (i.e. chloroform and acetone), acids (i.e. sulfuric, hydrochloric, and nitric), bases (i.e. sodium hydroxide), and oxidizers (i.e. iodine and hydrogen peroxide) are used in the analyses of clandestine laboratory precursors and reagents.
12.5.2. Microscope slides
12.5.3. Microscope cover slips
12.5.4. Hotplate
12.5.5. Scalpel or razor blade
12.5.6. Low e^- microscope slide
12.5.7. SEM stubs
12.5.8. Roller knife
12.5.9. Disposable syringes
12.5.10. Disposable gloves
12.5.11. Laboratory coat

12.5.12. pH test strips
12.5.13. Water finding paper
12.5.14. Oven
12.5.15. Spoon/watch glass
12.5.16. Beakers
12.5.17. Test tubes
12.5.18. Tweezers
12.5.19. Probes
12.5.20. Dropper bottles

12.6. Hazards/Safety:

12.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as "Biohazard" shall be handled with gloved hands during the entire examination process. Items handled with gloves shall be disinfected prior to handling with bare hands.

12.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling occurs first, the forensic scientist shall still take precautions during examination for potential additional DNA analysis in the future. During the examination the forensic scientist shall also wear a disposable face mask. Gloves shall be changed after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

12.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to minimize additional fingerprints, or obliterating existing fingerprints.

12.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

12.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS's shall be consulted.

12.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

12.6.7. Evidence shall be opened in an area of adequate ventilation such as a fume hood or a workstation snorkel.

12.7. Reference Materials/Controls/Calibration Checks:

12.7.1. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

12.7.2. Questioned and known samples shall be analyzed under similar conditions.

12.7.3. The GC/MS instrument shall be performance checked as required in Appendix 6.

12.7.4. The SEM Instrument shall be performance checked as required in Appendix 5.

12.7.5. The FTIR Instrument shall be performance checked as required in Appendix 4.

12.7.6. The Polarized Light Microscope shall be performance checked as required in Appendix 14.

12.7.7. In the event that performance checks are found to be unsatisfactory, the instrument shall be taken out-of-service and measures taken to restore the instrument to proper working order.

12.7.7.1. Any instrument that is out-of-service shall be visibly marked.

12.7.7.2. When an instrument is taken out-of-service for maintenance and/or repair, performance checks shall be performed prior to resuming casework on that instrument.

12.7.7.3. An infrequently used instrument may be placed in an “Inactive” status and the normal verification procedures may be suspended. Normal quality control procedures shall be resumed prior to use in casework analysis.

12.8. Procedures/Instructions:
This section provides an overview of suggested flow of analytical techniques to be utilized for the analysis of clandestine laboratory precursors and reagents. The exact selection and order of methods depends upon the type of chemical being analyzed.

12.8.1. Initial Evaluation and Handling Due to concerns with the handling of chemical reagents and precursors as physical evidence, each case shall be evaluated to develop appropriate procedures concerning the sampling, preservation, and order of analysis.

12.8.1.1. Different forensic disciplines may be called upon to examine the same item of evidence. The order in which the examinations shall be conducted needs to be resolved on a case-by-case basis. The order of examinations shall be selected and conducted so as to preserve the most transient evidence and provide the greatest discrimination and most valuable

information. Forensic Scientists shall make the submitting agency aware of the effects that some disciplines' processing and examinations may have upon other specific examination requests. If another discipline is chosen before the clandestine laboratory precursor and reagent examination, obtaining an unadulterated representative sample shall be considered.

12.8.1.2. When the amount of an unknown chemical specimen present is adequate in size—as deemed by the forensic scientist —analysis may include several different analytical techniques. Nondestructive methods shall be exhausted before subjecting the sample to any destructive tests.

12.8.1.3. If the initial tests are not exclusionary, the examination shall proceed with additional tests that are selected on the basis of their potential for use in discriminating the question and known chemicals. The size, condition, and nature of the unknown chemicals present will dictate the selected analytical scheme.

12.8.1.3.1. Written descriptions, photography, or other imaging methods shall be used to document each sample's characteristics. Preliminary examination shall include its general appearance macroscopically and under a stereomicroscope if applicable.

12.8.1.4. Liquid clandestine drug lab samples will be characterized by pH, gas-chromatography-mass spectrometry, infrared spectroscopy, or by other appropriate methods depending upon the nature of the sample.

12.8.1.5. Inorganic solid clandestine drug lab samples shall be examined visually and will be analyzed using appropriate methods based on preliminary observations.

12.8.1.6. Identification of phosphorus is conducted through X-ray analysis using SEM-EDS.

12.8.1.7. Solubility and starch tests shall be performed on samples containing Iodine. A sublimation-recrystallization test may also be conducted in a sealed vial with gentle heating and the crystals formed shall be compared with crystals formed by an iodine standard. Identification of iodine is conducted by comparison of crystal formations with those of a standard.

12.8.1.8. The presence of a dichromate compound is confirmed through infrared spectroscopy. Identification of sodium dichromate or potassium dichromate is conducted through X-ray analysis using SEM-EDS.

12.8.1.9. The pH of acidic solutions shall be measured and ammonium hydroxide will be added to the sample. The precipitate shall be analyzed and identified using infrared spectroscopy.

12.8.1.10. The pH of basic solutions shall be measured and sodium chloride salt will be made by reacting concentrated hydrochloric acid with the sodium hydroxide. Identification of the salt is conducted through X-ray analysis using SEM-EDS.

12.8.1.11. Reference chemical samples are analyzed in like manner to case samples.

12.8.1.12. Other laboratory techniques such as chemical conversion may be employed when deemed necessary by the examiner or the Unit Supervisor. The complex nature of these supplemental tests precludes analytical specification as such treatment lies beyond the scope of this general methods presentation.

12.8.1.13. The item of evidence shall be preserved in a manner that does not interfere with future testing. All procedures shall be conducted in such a manner to ensure that no cross-contamination occurs. The item shall be imaged or described prior to conducting any destructive analyses in order to provide documentation of original condition.

12.8.2. Physical Characteristic Evaluation

Macroscopic and stereomicroscopical observations (e.g., color, deliquescence, artifacts, and texture) provide initial and discriminating information for chemical comparisons. Physical characterization of chemical specimens is the initial step of a comprehensive forensic analysis.

12.8.2.1. Organic Solvents

12.8.2.1.1. Liquids shall first be characterized with pH paper and water finding paper. Aqueous liquids will be indicated by the water finding paper and the subsequent pH paper value shall be performed. Strong base pH results may indicate the aqueous solution is part of a clandestine manufacture process that still has available controlled substances in the liquid. The case investigator and the Drug Unit may be contacted to determine if an examination for controlled substances is appropriate.

12.8.2.1.1.1. If a liquid sample has two layers, both layers shall be examined to determine the nature of the liquids.

12.8.2.1.1.2. Organic solvent samples and layers shall be characterized by gas chromatography/mass spectroscopy, or by other appropriate methods depending upon the nature of the sample.

12.8.2.1.1.3. If enough of the liquid exists, a portion may be analyzed to determine if it ignites.

12.8.2.2. Red Phosphorus

12.8.2.2.1. Visual Inspection

12.8.2.2.1.1. Red to violet solid

12.8.2.2.2. Solubility

12.8.2.2.2.1. Solubility of the material may be tested in three solvents including deionized water, acetone, and chloroform. Red phosphorus is insoluble in all three solvents. Place a small amount of the sample may be tested in three (3) different wells of a spot plate.

12.8.2.2.3. pH Change

12.8.2.2.3.1. The change in pH may be examined when a sample of material is placed in the presence of iodine and water. Red Phosphorus makes the pH decrease by at least two (2) units as hydroiodic acid is formed.

12.8.2.3. Iodine

12.8.2.3.1. Visual Inspection

12.8.2.3.1.1. Bluish-black solid (e.g. scales or plates), metallic luster, blue-violet vapor, irritating odor.

12.8.2.3.2. Solubility

12.8.2.3.2.1. Solubility may be tested by adding chloroform to the material. If iodine is present, the chloroform layer will turn purple.

12.8.2.3.3. pH Change

12.8.2.3.3.1. The change in pH may be examined when a sample of material is placed in the presence of red phosphorus and water. Iodine makes the pH decrease by at least two (2) units as hydroiodic acid is formed.

12.8.2.3.4. Starch Test

12.8.2.3.4.1. A color change reaction may be observed when a sample of material is placed in water and introduced to starch paper. The presence of iodine in the water makes the starch paper turn purple.

12.8.2.3.5. Sublimation-Recrystallization Test

12.8.2.3.5.1. Crystals may be observed when the material is sealed in a vial and gently heated. Iodine will form characteristic crystals when heated in a sealed vial. Crystals formed by the material being analyzed will be compared to those formed by a known iodine standard to identify iodine.

12.8.2.4. Iodine Tincture

12.8.2.4.1. Visual Inspection

12.8.2.4.1.1. Opaque, brown to dark brown liquid

12.8.2.4.2. Extraction

12.8.2.4.2.1. Combine equal volumes of sample and hydrogen peroxide (3% - 4% concentration). For best results use small volumes of sample and hydrogen peroxide (<2mL each). Iodine crystals will precipitate and migrate to the top surface of the liquid. The crystals shall be filtered and analyzed using the methods described above.

12.8.2.5. Povidone-Iodine

12.8.2.5.1. Visual Inspection

12.8.2.5.1.1. Opaque, brown to dark brown liquid.

12.8.2.5.1.2. Develops soap-like bubbles or foam with vigorous shaking that persist for a time after shaking has stopped.

12.8.2.5.2. Extraction

12.8.2.5.2.1. A solution 5:1 (water: sample) is placed in an Erlenmeyer flask and fit a water-cooled reflux condensing to the flask. As the solution is heated a purple vapor will form in the flask, followed by the formation of silver-gray metallic crystals in the bottom portion of the condensing column. The crystals are collected and analyzed using the methods described above.

12.8.2.6. Sodium Dichromate and Potassium Dichromate

12.8.2.6.1. Visual Inspection

12.8.2.6.1.1. Sodium dichromate: reddish to bright orange, somewhat deliquescent crystals.

12.8.2.6.1.2. Potassium dichromate: bright orange-red crystals

12.8.2.6.2. Infrared Spectroscopy

12.8.2.6.2.1. An appropriate method using the FTIR with ATR accessory is selected and the sample is analyzed. Compare the spectrum generated for the sample with the library or reference material

chemical spectrum. The presence of a dichromate (Cr_2O_7) compound will be confirmed through infrared spectroscopy.

12.8.2.6.3. Elemental Analysis.

12.8.2.6.3.1. Place a small amount of sample on an SEM stub. Analyze the sample using SEM-EDS (refer to SEM-EDS user's manual). The presence of sodium dichromate or potassium dichromate will be confirmed through X-ray analysis.

12.8.2.7. Acids

12.8.2.7.1. pH

12.8.2.7.1.1. Record the pH of the sample. Associated with clandestine drug labs (e.g. hydrochloric acid, sulfuric acid, hydroiodic acid) are strong acids with a pH less than or equal to 2.

12.8.2.7.2. Hydrion water finding test paper

12.8.2.7.2.1. Dip a piece of hydrion water finding paper in the sample. An aqueous solution is indicated by a magenta color change. An organic or concentrated acid solution is indicated by no color change. Sulfuric acid is indicated by an orange color change.

12.8.2.7.3. Ammonium salt precipitate.

12.8.2.7.3.1. When mixing strong acids and strong bases, violent reactions may occur (sulfuric acid reacts with bases in a violent manner). It may be necessary to dilute the sample before mixing with ammonium hydroxide. Care shall be taken when making and disposing of these ammonium salts.

12.8.2.7.3.1.1. Complete ionization of monoprotic and polyprotic acids.

12.8.2.7.3.1.1.1. Drops of the acid sample are added to ammonium hydroxide and the ammonium salt is precipitated with the addition of acetone. The precipitate is then collected and analyzed.

12.8.2.7.3.1.1.2. An appropriate method using the FTIR with ATR accessory is selected and the sample is analyzed. Compare the spectrum generated for the sample with the library or reference material chemical spectrum. The presence of ammonium chloride, ammonium sulfate, ammonium iodide, etc. shall be confirmed through infrared spectroscopy.

12.8.2.7.3.1.1.3. Single ionization of polyprotic acids.

12.8.2.7.3.1.1.4. Drops of the acid sample are added to acetone and the polyprotic acid salt will be precipitated, without ionizing the remaining proton(s), with the addition of ammonium hydroxide. The precipitate is then collected and analyzed.

12.8.2.7.3.1.1.5. An appropriate method using the FTIR with ATR accessory is selected and the sample is analyzed. Compare the spectrum generated for the sample with the library or reference material chemical spectrum. The presence of ammonium hydrogen sulfate, ammonium hydrogen phosphate, etc. will be confirmed through infrared spectroscopy.

12.8.2.8. Sodium Hydroxide

12.8.2.8.1. Visual Inspection

12.8.2.8.1.1. White, deliquescent solid; usually beads or pellets

12.8.2.8.2. pH

12.8.2.8.2.1. A small amount of the sample is tested in a well of a spot plate and deionized water is added. The presence of sodium hydroxide is indicated by a pH greater than 12.

12.8.2.8.3. Sodium salt

12.8.2.8.3.1. Approximately 0.5g of the sample is dissolved in a beaker containing approximately 10mL of water and concentrated hydrochloric acid is added to bring the pH between 6 and 8. When the solution is dried sodium chloride crystals will form and shall be confirmed through X-ray analysis.

12.8.2.9. Ammonium Nitrate and Ammonium Sulfate

12.8.2.9.1. Visual Inspection

12.8.2.9.1.1. White solid, usually beads, pellets, or granulated

12.8.2.9.2. Elemental Analysis

12.8.2.9.2.1. A small amount of sample shall be analyzed using SEM-EDS (refer to SEM-EDS user's manual).

12.8.2.9.3. Infrared Spectroscopy

12.8.2.9.3.1. An appropriate method using the FTIR with ATR accessory is selected and the sample is analyzed. Compare the spectrum generated for the sample with the library or reference material chemical spectrum. The presence of ammonium nitrate and ammonium sulfate shall be confirmed through infrared spectroscopy.

12.9. Records:

12.9.1. Written descriptions, photography, or other imaging methods shall be used to document each sample's characteristics.

12.9.2. Preliminary examination of materials shall include its general appearance macroscopically and under a stereomicroscope when applicable.

12.9.3. The case record shall include the instrumental data. All hard copies shall include a unique sample designation, the operator's name/initials, and the date of analysis. Case notes shall also include a description of the evidence analyzed, the method of sample preparation, the analytical instrumentation used, and its operating parameters.

12.10. Interpretations of Results:

12.10.1. Typically, a clandestine laboratory precursor and reagent examination involves the identification of chemical samples or solutions. The physical and chemical characteristics are evaluated to identify sample material.

12.11. Report Writing:

12.11.1. If a sample is identified as an organic solvent or petroleum product, liquid as an ignitable liquid examples of the type of organic solvent or petroleum product shall be provided (e.g. toluene, acetone, camp fuel).

12.11.2. The presence of an element or inorganic compound in a sample, which has been analyzed and identified, will be reported as such (e.g. iodine, red phosphorus, ammonium nitrate).

12.11.3. The presence of an acid or alkali, which has been analyzed and identified, will be reported as such (e.g. hydrochloric acid, sulfuric acid, sodium hydroxide).

12.12. References:

12.12.1. ASTM E1459 Standard Guide for Physical Evidence Labeling and Related Documentation
12.12.2. ASTM E1492 Standard Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Lab

12.12.3. Microgram Bulletin, Vol. XXXVII, No. 2, Feb. 2004, Identification of Iodine and Red Phosphorus

12.12.4. Journal of the Clandestine Laboratory Investigating Chemists Assoc., Vol. 5, No. 4, Oct. 1995, Analysis of Inorganic Components Found in Clandestine Drug Laboratory Evidence

12.12.5. Clandestine Laboratory Investigating Chemists Assoc. Training Seminar, Sept. 1998, Identification of Common Inorganic Acids Encountered at Clandestine Laboratories

12.12.6. The Merck Index, Twelfth Ed. (CD-ROM Version 12.1), 1996

12.12.7. Indiana Criminal Code, IC 35-48-4-14.5 Possession or Sale of Drug Precursors

13. Physical Edge Match Examinations

13.1. Scope:

This test method is for the analysis, examination, and comparison of items of physical evidence for physical edge match evaluation to determine if they have common origin. The scope of this type of evidence includes but is not limited to glass, plastic, metal, paint, fabric, and paper. A physical edge match of two items can provide individualizing characteristics that associate the two items to one another. The methods and practices described have been published, peer-reviewed, and are generally accepted within the forensic community.

13.2. Precautions/Limitations:

13.2.1. The analysis and comparison of items for a physical edge match in the forensic science laboratory is dependent on having the separated edge of the two resulting items present. If pieces of the original broken item are lost or not collected, the pieces on either side may not be able to be associated to each other. The examination of the two items for a physical edge match is dependent on them originally being in direct contact with one another.

13.2.2. For all examinations a determination if the ends can be puzzle matched to each other shall be evaluated. The item may only be a two dimensional material (e.g. tape). Also the material may be pliable, and thus the edge is often distorted (e.g. black electrical tape). With only a two dimensional fit or with distortion of the item a lock and key type of puzzle match may not occur. Depending on the condition, a determination that the edges were once connected may be implied but not lend itself to a conclusive opinion to the association. Depending on the types of characteristics present, a full chemical analysis may also be appropriate.

13.2.3. An item which is broke or fractured may be compromised after the initial separation to the point where it is not possible to conduct a physical edge match examination. Materials that are brittle in nature can be broken or distorted to the point where an association is no longer possible.

13.3. Related Information:

13.3.1. Appendix 1 Worksheets
13.3.2. Appendix 2 Abbreviations
13.3.3. Appendix 3 Definitions
13.3.4. Test Method for Glass Examinations
13.3.5. Test Method for Fiber Examinations
13.3.6. Test Method for Paint Examinations
13.3.7. Test Method for Physical Comparisons

13.4. Instruments:

13.4.1. Stereomicroscope – a microscope which gives a “three dimensional view” capable of examining samples up to a magnification of approximately 70x.

Stereomicroscopical observations (e.g., fracture lines, Contours, etc...) provide initial and discriminating information for considering questioned and known pieces for possible physical edge match.

13.4.2. Calipers – an instrument capable of accurately and precisely measuring the thickness of different samples. It can be used to measure the thickness of items when both edges are present.

13.4.3. Comparison microscope - a microscope capable of simultaneous microscopic examinations of two different items.

13.4.4. Magnifying lens – an optical instrument designed to allow for the viewing of items of physical evidence.

13.4.5. Imaging equipment – capable of reproducing and storing images.

13.5. Reagents/Materials:

13.5.1. Ruler or measuring tape – a device capable of making measurements of size and length.

13.6. Hazards/Safety:

13.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as “Biohazard” shall be handled with gloved hands during the entire examination process. Items handled with gloves shall be disinfected prior to handling with bare hands.

13.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling occurs first, the forensic scientist shall still take precautions during examination for potential additional DNA analysis in the future. During the examination the forensic scientist shall also wear a disposable face mask. Gloves shall be changed after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

13.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to minimize

additional fingerprints, or obliterating existing fingerprints.

13.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

13.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS's shall be consulted.

13.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

13.7. Reference Materials/Controls/Calibration Checks:

13.7.1. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

13.7.2. Questioned and known samples shall be analyzed under similar conditions.

13.7.3. All cases shall be peer technically reviewed. The forensic scientist doing the peer technical review shall verify all positive physical edge matches. The name of the person conducting the verification shall be noted in the case notes for the physical edge match. The forensic scientist conducting the verification shall be noted as the technical reviewer in LIMS for that case.

13.8. Procedures/Instructions:
This section provides an overview of suggested flow of analytical techniques to be utilized for the analysis of physical edge matches. The exact selection and order of methods depends upon the sample size and/or condition as well as the type of material that is present. If the material is paint, glass, or fibers there may be additional guidelines in the respective test method.

13.8.1. **Initial Evaluation and Handling** Due to concerns with the handling physical evidence, each case shall be evaluated to develop appropriate procedures concerning the collection, observation, preservation, and order of examinations.

13.8.1.1. Different forensic disciplines may be called upon to examine the same item of evidence. The order in which the examinations shall be conducted needs to be resolved on a case-by-case basis. The order of examinations shall be selected and conducted so as to preserve the most transient evidence and provide the greatest discrimination and most valuable information. Forensic Scientist shall make the submitting agency aware of the effects that some disciplines' processing and examinations may have upon other specific examination requests. If another discipline is chosen before the physical edge match examination, obtaining documentation of the unadulterated sample shall be considered.

13.8.1.2. If the initial tests are not exclusionary and do not provide for a definitive physical edge match, the examination shall proceed with additional tests that are selected on the basis of their potential for use in discriminating the questioned and known samples. The size, condition, and type of material present will dictate the selected analytical scheme.

13.8.1.2.1. Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics. Preliminary examination shall include its general appearance macroscopically and under a stereomicroscope, including any physical damage.

13.8.1.3. Ideally the item of evidence shall be preserved in a manner that does not interfere with future testing. All procedures shall be conducted in such a manner to ensure that no cross-contamination occurs. The item shall be photographed or described prior to conducting any destructive analyses in order to provide documentation of original condition.

13.8.2. Physical Characteristic Evaluation Macroscopic and stereomicroscopical observations provide initial and very discriminating information for physical edge match comparisons. Physical characterization of specimens is the initial step of a comprehensive forensic analysis. This visual examination is needed to determine the type and extent of the examination to be conducted. This visual examination also allows for the determination of the correct orientation of the items. Contours, edges, colors, surface features, and similar characteristics may assist in aligning the items correctly. It can be helpful to fit together pieces of a given source (known pieces) before comparing them to pieces that have already been fit together from an additional source (question piece). Visual characteristics are the quickest, most discriminating, and least invasive examinations.

13.8.2.1.1. Experience and judgment on the part of the forensic scientist are a necessity to decide the manner of comparing submitted evidence. The examiner will choose the method of examination based on the questioned evidence submitted.

13.8.3. Broken or torn items

13.8.3.1. Determine the class characteristics of the questioned item(s).

13.8.3.2. Determine the class characteristics of the known item(s).

13.8.3.3. If class characteristics are similar, determine if broken or torn edges of the questioned and standard items exist. Examine the items to see if the edges of the questioned and standard pieces fit together like a puzzle. Hard substances often break leaving unique features. Some physical end matches have a three dimensional fit which is helpful in making a conclusive determination. Matching hackle marks and defects shall also be examined

to reinforce the physical fit. The topography of the two pieces are often mirror images of each other.

13.8.3.4. Determine if individual marks appear to run across the break or tear on both the questioned and known item.

13.8.3.5. Ductile materials are pliable; therefore the fractured edge is often distorted. With the distortion a lock and key type of puzzle match will not occur. When matching flexible materials (e.g. fabric, tape, some plastics) edge rolling, stretching and twisting shall be taken into account. Depending on the condition of the material, a determination that the edges were once connected is possible if enough characteristics exist at the edges.

13.8.3.6. If a physical edge match is not present, the general class characteristics and chemistry can be evaluated to provide association between the two items.

13.8.3.7. If appropriate, a chemical analysis can be conducted to help with associating the two materials even if a non-conclusive physical edge match exists.

13.9. Records: Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics.

13.9.1. Preliminary examination of each item shall include its general appearance macroscopically and under a stereomicroscope, including any adhering matter.

13.9.2. The case record shall include the instrumental data. All hard copies shall include a unique sample designation, the operator's name/initials, and the date of analysis. Case notes shall also include a description of the evidence analyzed, the method of sample preparation, the analytical instrumentation used, and its operating parameters.

13.10. Interpretations of Results:

13.10.1. Typically, a physical edge match examination involves the comparison of samples to determine if they share a common origin. The goal is to determine if any the two items were once attached. The physical and chemical characteristics can be evaluated to associate the questioned and known sample. If a significant difference in a characteristic exists the samples are excluded as having a common origin. If the examination doesn't reveal any significant differences it is concluded that the samples could share a common origin. A limited association can be reported due to the condition/size of one of the samples. The conclusions reached as to common origin or positive identification are based on the significance of similarities or dissimilarities in the characteristics observed.

13.11. Report Writing:

13.11.1. If significant differences are found in the items characteristics, then the report shall state that the different characteristics and/or properties suggest that they did not come from the same source.

13.11.2. If no significant differences are found in the two items characteristics, then the report shall state that the two items are consistent and could have come from the same source but that a physical edge match was not found.

13.11.3. If a physical end match is located, but it is not a conclusive end match, then the association will be stated as such. The report shall state that an association exists and that there is a possible physical edge match, but due to the condition of the items it is not conclusive.

13.11.4. If a conclusive physical end match exists between two items, then a more positive association exists. The report shall state that the items were once connected and a part of each other.

13.12. References:

13.12.1. Thornton J., “Fractal Surfaces as Models of Physical Matches”, Journal of Forensic Sciences, Vol. 28, No. 2, 1983, pp 463-467

14. Physical Characteristics Examinations

14.1. Scope:

This test method is for the analysis, examination, and comparison of items of physical evidence for a physical characteristic evaluation. The scope of this type of evidence includes but is not limited to plastic, metal, paint, glass, fabric, and paper. A comparison is made of the different items for class characteristics and then the items are examined for physical characteristics if they do not fit along a fractured edge. If the two items fit along a fractured edge then the physical edge comparison test method shall be followed. Two items can also be evaluated to determine if they have common chemistry and physical properties and could be from a similar manufacturing source (e.g. two plastic bags from the same roll, a lost button compared to the remaining ones on a shirt). The methods and practices described have been published, peer-reviewed, and are generally accepted within the forensic community.

14.2. Precautions/Limitations:

14.2.1. The complete identification or classification of a random assortment of materials is often not possible. If miscellaneous debris is collected, the wide assortment of materials present may preclude any probative information.

14.2.2. The Microanalysis Unit is limited by the instrumentation and experience of the analyst. There are some materials that are better suited to be analyzed by instrumentation that the laboratory does not possess or operate (e.g. LC/MS or ICP/MS).

14.2.3. There are other forensic laboratories that routinely conduct examinations of certain materials (e.g. geological samples – are better suited going to a laboratory that routinely does soil examinations).

14.2.4. If a broken material is deemed to have similar design, composition, color, size, etc. to the known and there is no physical edge match it is still possible that the material has a different origin. Two possibilities exist: 1) that they originated from the same source, but connecting pieces of material are missing. 2) that they are two random pieces of broken material which just happen to share common physical characteristics.

14.3. Related Information:

14.3.1. Appendix 1 Worksheets
14.3.2. Appendix 2 Abbreviations
14.3.3. Appendix 3 Definitions
14.3.4. Test Method for Physical Edge Match Comparisons
14.3.5. Test Method for Glass
14.3.6. Test Method for Paint
14.3.7. Test Method for Fibers

14.4. Instruments:

14.4.1. Stereomicroscope – a microscope which gives a "three dimensional view" capable of examining samples up to a magnification of approximately 70x. Stereomicroscopical observations provide initial and discriminating information for selecting the examination and analysis protocol.

14.4.2. Calipers – an instrument capable of accurately and precisely measuring the thickness of different samples. It can be used to measure the thickness of materials when both edges are present.

14.4.3. Polarized Light Microscope (PLM) - a microscope which can be used to evaluate the properties of samples. Characterization and identification of material can be accomplished with the use of PLM.

14.4.4. UV Box – an instrument designed to allow for the viewing of specimens in short and long wavelengths of light. It can be used to look at the fluorescence of a material.

14.4.5. Scanning Electron Microscope with energy dispersive spectroscopy (SEM/EDS) – an analytical instrument capable of chemically analyzing a sample. It can be utilized for the characterization of the inorganic constituents of a material.

14.4.6. X-ray Fluorescence (XRF) – an analytical instrument capable of chemically analyzing a sample. It can be utilized for the characterization of the inorganic constituents of a material down to the trace elements that are present.

14.4.7. Densitometer – an instrument for determining the density of a solid or liquid. It can be used to determine the density of a material.

14.4.8. Fourier Transform Infrared (FTIR) Spectrometer– an analytical instrument capable of analyzing the molecular vibrations of the chemical components of a sample. Organic and some inorganic constituents may be evaluated with the use of infrared spectroscopy. The components of individual particles can be evaluated with the Microscope FTIR.

14.4.9. Pyrolysis-Gas Chromatography/Mass Spectrometry (Py-GC/MS) – an instrument capable of thermally breaking down, separating, and then analyzing a sample.

14.4.10. Gas Chromatograph/Mass Spectrometer (GC/MS) – An instrument that separates a compound into a pure sample so that each component can be identified. The resulting chromatogram shows how much of each singular ion is present, and this is used for identification of unknown liquids.

14.4.11. Microspectrophotometer – an analytical instrument capable of analyzing a sample. It can be utilized for the characterization of the color and fluorescence of the material as well as its properties under ultraviolet light.

14.4.12. Microtome – an instrument for cutting thin cross sections of specimens. It can be used to make cross sections of materials.

14.4.13. Comparison Microscope - a microscope capable of simultaneous microscopic examinations of two different items.

14.4.14. Hot Stage Microscope - a microscope capable of determining the melting point of a material.

14.5. Reagents/Materials:

14.5.1. Microscope slides
14.5.2. Microscope cover slips
14.5.3. Suitable solvents
14.5.4. Mounting medium
14.5.5. Cargille refractive index liquids (1.3 to 1.8)
14.5.6. Hotplate
14.5.7. Scalpel or razor blade
14.5.8. Low e^- microscope slide
14.5.9. SEM stubs
14.5.10. Roller knife.
14.5.11. Disposable syringes
14.5.12. Disposable gloves
14.5.13. Laboratory coat
14.5.14. pH test strips
14.5.15. Water finding paper
14.5.16. Oven
14.5.17. Thermometer
14.5.18. Spoon/watch glass
14.5.19. Beakers
14.5.20. Test tubes
14.5.21. Tweezers
14.5.22. Probes
14.5.23. Dropper bottles

14.6. Hazards/Safety:

14.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as “Biohazard” shall be handled with gloved hands during the entire examination process. Items handled with gloves shall be disinfected prior to handling with bare hands.

14.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling

occurs first, the forensic scientist shall still take precautions during examination for potential additional DNA analysis in the future. During the examination the forensic scientist shall also wear a disposable face mask. Gloves shall be changed after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

14.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to minimize additional fingerprints, or obliterating existing fingerprints.

14.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

14.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS's shall be consulted.

14.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

14.7. Reference Materials/Controls/Calibration Checks:

14.7.1. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

14.7.2. Questioned and known samples shall be analyzed under similar conditions.

14.7.3. The Py-GC/MS shall be performance checked as required in Appendix 7.

14.7.4. The SEM Instrument shall be performance checked as required in Appendix 5.

14.7.5. The FTIR Instrument shall be performance checked as required in Appendix 4.

14.7.6. The melting point microscope shall be performance checked as required in Appendix 11.

14.7.7. The Microspectrophotometer Instrument shall be performance checked as required in Appendix 10.

14.7.8. The Polarized Light Microscope shall be performance checked as required in Appendix 14.

14.7.9. The GC/MS shall be performance checked as required in Appendix 6.

14.7.10. In the event that performance checks are found to be unsatisfactory, the instrument shall be taken out-of-service and measures taken to restore the instrument to proper working order.

14.7.10.1. Any instrument that is out-of-service shall be visibly marked.

14.7.10.2. When an instrument is taken out-of-service for maintenance and/or repair, performance checks shall be performed prior to resuming casework on that instrument.

14.7.10.3. An infrequently used instrument may be placed in an "Inactive" status and the normal verification procedures may be suspended. Normal quality control procedures shall be resumed prior to use in casework analysis.

14.8. Procedures/Instructions:
This section provides an overview of suggested flow of techniques to be utilized for the analysis of items. The exact selection and order of methods depends upon the sample size and/or condition.

14.8.1. Initial Evaluation and Handling Each case shall be evaluated to develop appropriate procedures concerning the sampling size, collection, preservation, and order of examinations. The type and number of class characteristics present to evaluate and compare shall be assessed.

14.8.1.1. Different forensic disciplines may be called upon to examine the same item of evidence. The order in which the examinations shall be conducted needs to be resolved on a case-by-case basis. The order of examinations shall be selected and conducted so as to preserve the most transient evidence and provide the greatest discrimination and most valuable information. Forensic Scientist shall make the submitting agency aware of the effects that some disciplines' processing and examinations may have upon other specific examination requests. If another discipline is chosen before the physical comparison examination, obtaining an unadulterated representative sample shall be considered.

14.8.1.2. When the amount of a specimen present for comparison purposes is adequate in size—as deemed by the forensic scientist —analysis may include several different analytical techniques. Nondestructive methods shall be exhausted before subjecting the sample to any destructive tests.

14.8.1.3. If the initial tests are not exclusionary, the examination shall proceed with additional tests that are selected on the basis of their potential for use in discriminating the questioned and known samples. The size, condition, and type of material present will dictate the selected analytical scheme.

14.8.1.3.1. Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics. Preliminary examination shall include its general appearance macroscopically and under a stereomicroscope, including any physical damage.

14.8.1.4. The item of evidence shall be preserved in a manner that does not interfere with future testing. All procedures shall be conducted in such a manner to ensure that no cross-contamination occurs. The item shall be imaged or described prior to conducting any destructive analyses in order to provide documentation of original condition.

14.8.1.5. The initial examination may be the final step in an analysis if the items are different upon the initial evaluation. All of the pieces in a questioned item need to be evaluated in comparison to the known sample.

14.8.1.6. The forensic scientist shall retain the evidence tested.

14.8.2. Physical Characteristic Evaluation
Macroscopic and stereomicroscopical observations (e.g., colors, thicknesses, texture, and markings) provide initial and very discriminating information for comparisons. Physical characterization of specimens is the initial step of a comprehensive forensic analysis. Visual characteristics are the quickest, most discriminating, and least invasive examinations. If no significant physical differences are found between samples, instrumental analyses are warranted.

14.8.2.1. The stereomicroscopical characteristics are sometimes apparent without sample preparation. For a more definitive evaluation, a manual or microtome cross section can employed.

14.8.2.2. General Characteristics The following characteristics shall be documented when applicable:

14.8.2.2.1. The general form of the material shall be noted (e.g. solid, flat, and cylindrical).

14.8.2.2.2. The amount of the material present shall be noted.

14.8.2.2.3. The homogeneity of the material shall be noted.

14.8.2.2.4. If more than one type of material is present they all shall be characterized.

14.8.2.2.5. The color and texture of each of the material(s).

14.8.2.2.6. The hardness of the material (s).

14.8.2.2.7. The shape of the material (e.g. cubic).

14.8.2.2.8. The number of layers present (if applicable).

14.8.2.3. Comparison The questioned item is sometimes large enough for a side-by-side comparison to the standard. If possible, for comparative examinations the item shall be examined both macroscopically and by using a stereomicroscope for color, texture, and appearance under multiple illumination sources. Subtle differences are more readily seen in a side-by-side comparison in the same field of view.

14.8.2.4. Markings on the Surface Layer Under the stereomicroscope the item shall be examined for features such as marks, striations, dimples, and inclusions on the top surface layer. The shapes and type of markings shall be documented.

14.8.2.5. Multiple Layer Samples Multiple layers may be present in an item and the item shall be examined to determine if multiple layers are present. There are a number of ways to cross-section an item (e.g. hand sectioning, microtome). The multiple layers, if present, shall be characterized and then analyzed with appropriate analytical instrumentation.

14.8.3. Instrumental Analysis

14.8.3.1. Instrumental analysis offers a method to characterize solid materials both organically and inorganically. The initial step of the instrumental analysis shall normally follow a set protocol using instrument parameters that are similar to one used in one of the sub-discipline test methods or in a published testing protocol.

14.8.3.2. The examiner shall refer to the specific type of material Test Method for examination (e.g. Fibers, Glass, Paint).

14.8.3.2.1. The instrument parameters shall be noted in the case record.

14.8.4. Testing following guidelines from other sources shall be referenced and included in the case record.

14.9. Records: Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics.

14.9.1. Preliminary examination of an item shall include its general appearance macroscopically and under a stereomicroscope, including any adhering matter.

14.9.2. The case record shall include the instrumental data. Case notes shall include a description of the evidence analyzed, the method of sample preparation, the analytical instrumentation used, and its operating parameters.

14.10. Interpretations of Results:

14.10.1. Typically, an examination involves the comparison of samples to determine if they could share a common origin or manufacturer. The goal is to determine if any significant differences exist between the samples. The physical and chemical characteristics are evaluated to associate the questioned and known sample. If a significant difference in a characteristic exists the samples are excluded as having a common origin or manufacturing process. If the examination doesn't reveal any significant differences it is concluded that the samples could share a common origin or manufacturing process. A limited association can be reported due to the condition/size of one of the samples or limited class characteristics. The evaluation of class characteristics can associate known and questioned items to a group but not to a single, individual source.

14.11. Report Writing:

14.11.1. For Fractured Items

14.11.1.1. If significant differences are found in the item characteristics, then the report shall state that the items are not consistent in characteristics and did not originate from the same source or manufacturing process.

14.11.1.1.1. If the known sample is fractured and it is believed that it includes all of the properties and characteristics of the complete original item.

14.11.1.2. If no significant differences are found in the characteristics, then the report shall state that the items are consistent and could have come from the same source or another source with these same characteristics.

14.11.1.3. If not enough of the questioned item is present for comparison purposes, or if the known item does not appear to be a complete representation of the complete item, the report shall state as such.

14.11.2. For a Similar Manufactured Physical Comparison

14.11.2.1. If significant differences are found in the item characteristics, then the report shall state that the items are not consistent and probably did not originate from the same source or manufacturing process. The explanation shall be noted. (e.g. The questioned and known sample had different design or physical dimensions.)

14.11.2.2. If no significant differences are found in the characteristics, then the report shall state that the items are consistent and could have come from the same manufacturing source or another source with these same characteristics.

14.12. References:

14.12.1. SWGMAT Trace Recovery Guidelines [Online] (October 1999). Available: http://www.fbi.gov/hq/lab/fsc/backissu/oct1999/index.htm

14.12.2. Thornton J., "Fractal Surfaces as Models of Physical Matches", Journal of Forensic Sciences, Vol. 28, No. 2, 1983, pp 463-467

15. General Chemical Examinations

15.1. Scope:

This test method is for the analysis, examination, and comparison of evidentiary materials that do not fall into a specific test method. The scope of this type of evidence includes but is not limited to solids and liquids encountered as physical evidence. There are general outlines for some forensic types of materials (e.g. building materials, safe insulation, bank dyes, irritants, tire rubber). Each case shall be evaluated to see if it falls within the general approach using scientifically accepted methodology. The methods and practices described have been published, peer-reviewed, and are generally accepted.

15.2. Precautions/Limitations:

15.2.1. The complete identification or classification of a random assortment of materials is often not possible. If miscellaneous debris is collected, the wide assortment of materials present may preclude development of useful information.

15.2.2. The Microanalysis Unit is limited by the instrumentation and experience of the analyst. There are some materials that are better equipped to be analyzed by instrumentation that the laboratory has not procured (e.g. water based lotions often are more suited to analysis by liquid chromatography).

15.2.3. Known and questioned samples shall never be packaged together and all packaging should be sealed to prevent leakage of small items of evidence into or out of the packaging container. Precautions shall be taken to prevent cross-contamination between known and questioned samples.

15.3. Related Information:

15.3.1. Appendix 1 Worksheets
15.3.2. Appendix 2 Abbreviations
15.3.3. Appendix 3 Definitions
15.3.4. Test Method for Physical Comparisons

15.4. Instruments:

15.4.1. Stereomicroscope – a microscope which gives a “three dimensional view” capable of examining samples up to a magnification of approximately 70x. Stereomicroscopical observations provide initial and discriminating information for selecting the examination and analysis protocol.

15.4.2. Calipers – an instrument capable of accurately and precisely measuring the thickness of different samples. It can be used to measure the thickness of materials when both edges are present.

15.4.3. Polarized Light Microscope (PLM) - a microscope which can be used to evaluate the properties of samples. Characterization and identification of material can be

accomplished with the use of PLM.

15.4.4. UV Box – an instrument designed to allow for the viewing of specimens in short and long wavelengths of light. It can be used to look at the fluorescence of a material.

15.4.5. Scanning Electron Microscope with Energy Dispersive Spectroscopy (SEM/EDS) – an analytical instrument capable of chemically analyzing a sample. It can be utilized for the characterization of the inorganic constituents of a material.

15.4.6. X-ray Fluorescence (XRF) – an analytical instrument capable of chemically analyzing a sample. It can be utilized for the characterization of the inorganic constituents of a material down to the trace elements that are present.

15.4.7. Densitometer – an instrument for determining the density of a solid or liquid. It can be used to determine the density of a material.

15.4.8. Fourier Transform Infrared (FTIR) Spectrometer– an analytical instrument capable of analyzing the molecular vibrations of the chemical components of a sample. Organic and some inorganic constituents may be evaluated with the use of infrared spectroscopy. The components of individual particles can be evaluated with the Microscope FTIR.

15.4.9. Pyrolysis-Gas Chromatography Mass Spectrometry (Py-GC/MS) – an instrument capable of thermally breaking down, separating, and then analyzing a sample.

15.4.10. Gas chromatograph/Mass spectrometer (GC/MS) – An instrument that separates a compound into a pure sample so that each component can be identified. The resulting chromatogram shows how much of each singular ion is present, and this is used for identification of unknown liquids.

15.4.11. Microspectrophotometer – an analytical instrument capable of analyzing a sample. It can be utilized for the characterization of color and fluorescence of the material as well as its properties under ultraviolet light.

15.4.12. Microtome – an instrument for cutting thin cross sections of specimens. It can be used to make cross sections of materials.

15.4.13. Comparison Microscope - a microscope capable of simultaneous microscopic examinations of two different items.

15.4.14. Hot stage Microscope - a microscope capable of determining the melting point of a material.

15.5. Reagents/Materials:

15.5.1. Microscope slides
15.5.2. Microscope cover slips

15.5.3. Suitable solvents
15.5.4. Mounting medium
15.5.5. Cargille refractive index liquids (1.3 to 1.8)
15.5.6. Hotplate
15.5.7. Scalpel or razor blade
15.5.8. Low e^- microscope slide
15.5.9. SEM stubs
15.5.10. Roller knife.
15.5.11. Disposable syringes
15.5.12. Disposable gloves
15.5.13. Laboratory coat
15.5.14. pH test strips
15.5.15. Water finding paper
15.5.16. Oven
15.5.17. Thermometer
15.5.18. Spoon/watch glass
15.5.19. Beakers
15.5.20. Test tubes
15.5.21. Tweezers
15.5.22. Probes
15.5.23. Dropper bottles

15.6. Hazards/Safety:

15.6.1. During the initial evidence examination (when the original item is open) proper laboratory attire shall be a lab coat and gloves. If someone is to observe the initial examination of primary evidence they shall also wear a lab coat and gloves. People may be in the examination area, during the normal course of business, without a lab coat and gloves if they are not performing a primary examination. Evidence suspected as or marked as “Biohazard” shall be handled with gloved hands during the entire examination process.

15.6.2. If an item is also submitted for DNA analysis, then the scientist needs to take special precautions to prevent contamination of the item. Even if DNA sampling occurs first, the forensic scientist shall still take precautions during examination for potential additional DNA analysis in the future. During the examination the forensic scientist shall also wear a disposable face mask. Gloves shall be changed after each item. Other personnel present in the vicinity of the sampling area shall refrain from conversing with the forensic scientist while this evidence is open.

15.6.3. If an item is also submitted for fingerprint analysis, the forensic scientist shall take special precautions to preserve fingerprints during the examination process. If the forensic scientist examines the item before fingerprint processing the following precautions shall be taken: the forensic scientist shall double glove (i.e. cloth gloves with disposable gloves on the outside) and handling of the items shall be kept to a minimum. Necessary precautions shall be used to minimize

additional fingerprints, or obliterating existing fingerprints.

15.6.4. General laboratory practices shall be employed when handling preparatory materials (i.e. sharps, liquid nitrogen).

15.6.5. General laboratory practices shall be employed when handling chemicals. The related MSDS's should be consulted.

15.6.6. Some general laboratory practices are described in the Indiana State Police Laboratory Safety Manual, Chemical Hygiene Plan, Bloodborne Pathogen Exposure Control Plan, and MSDS's.

15.6.7. Unknown substances (i.e. poisons, toxins, and explosives, asbestos) present unidentified and unforeseen dangers, and therefore shall be treated with extreme caution.

15.6.7.1. Use caution when acidifying unknown materials, as cyanide gas is very poisonous.

15.7. Reference Materials/Controls/Calibration Checks:

15.7.1. Controls or reference materials are often not submitted with evidence. If the questioned specimen is a suspected tampered consumer product, a similar store bought item will prove useful as a control.

15.7.2. Microscopy tools (e.g. scissors, tweezers, probe) shall be cleaned between samples.

15.7.3. Questioned and known samples shall be analyzed under similar conditions.

15.7.4. The Py-GC/MS shall be performance checked as described in Appendix 7.

15.7.5. The SEM Instrument shall be performance checked as described in Appendix 5.

15.7.6. The FTIR Instrument shall be performance checked as described in Appendix 4.

15.7.7. The melting point microscope shall be performance checked as described in Appendix 11.

15.7.8. The Microspectrophotometer Instrument shall be performance checked as described in Appendix 10.

15.7.9. The Polarized Light Microscope shall be performance checked as described in Appendix 14.

15.7.10. The GC/MS shall be performance checked as described in Appendix 6.

15.7.11. In the event that performance checks are found to be unsatisfactory, the instrument shall be taken out-of-service and measures taken to restore the instrument to proper working order.

15.7.11.1. Any instrument that is out-of-service shall be visibly marked.

15.7.11.2. When an instrument is taken out-of-service for maintenance and/or repair, performance checks shall be performed prior to resuming casework on that instrument.

15.7.11.3. An infrequently used instrument may be placed in an "Inactive" status and the normal verification procedures may be suspended. Normal quality control procedures shall be resumed prior to use in casework analysis.

15.8. Procedures/Instructions:
This section provides an overview of suggested flow of analytical techniques to be utilized for the analysis of physical evidence for general chemical analysis. The exact selection and order of methods depends upon the sample size and/or condition along with the information derived from the analysis and testing of the material. For examinations where a possible general chemical is indicated on the request, it shall be evaluated appropriately using this information. Analysis of the specimen will be determined case by case due to the wide range of samples encountered in foreign substance casework. Using the case history and type of sample submitted as a guide, the examiner shall decide which analytical methods are appropriate.

15.8.1. Initial Evaluation and Handling Due to variation in general chemical examinations, each case shall be evaluated to develop appropriate procedures concerning the sampling size, collection, preservation, and order of examinations.

15.8.1.1. Different forensic disciplines may be called upon to examine the same item of evidence. The order in which the examinations shall be conducted needs to be resolved on a case-by-case basis. The order of examinations shall be selected and conducted so as to preserve the most transient evidence and provide the greatest discrimination and most valuable information. The Forensic Scientist shall make the submitting agency aware of the effects that some disciplines' processing and examinations may have upon other specific examination requests. If another discipline is chosen before the general chemical examination, obtaining an unadulterated representative sample shall be considered.

15.8.1.2. When the amount of the physical evidence specimen present for comparison purposes is adequate in size—as deemed by the forensic scientist — analysis may include several different analytical techniques. Nondestructive methods shall be exhausted before subjecting the sample to any destructive tests.

15.8.1.3. If the testing is a comparison, and if the initial tests are not exclusionary, the examination shall proceed with additional tests that are selected on the basis of their potential for use in discriminating the questioned and known samples. The size, condition, structural complexity, and type of material present will dictate the selected analytical scheme.

15.8.1.4. Sampling technique(s) shall be determined based on the samples characteristics prior to analysis. If it appears to be of uniform consistency, size, color, and texture, then testing of any portion of this sample is adequate.

15.8.1.4.1. Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics. Preliminary examination shall include its general appearance macroscopically and under a stereomicroscope, including any physical characteristics.

15.8.1.5. The item of evidence shall be preserved in a manner that does not interfere with future testing. All procedures shall be conducted in such a manner to ensure that no cross-contamination occurs. The item shall be imaged or described prior to conducting any destructive analyses in order to provide documentation of original condition.

15.8.1.5.1. When appropriate it may also prove useful to contact the manufacturer of the consumer product for information about product processing, ingredients, and packaging.

15.8.1.6. The initial examination may be the final step in an analysis if no questioned material is located, or only exclusionary samples are identified in comparison to the known sample.

15.8.2. Physical Characteristic Evaluation
Macroscopic and stereomicroscopical observations (e.g., colors, thicknesses, and texture) for solid materials provide initial and very discriminating information for comparisons. Physical characterization of material specimens is the initial step of a comprehensive forensic analysis. The physical properties of liquids shall be evaluated as well. Visual characteristics are the quickest, most discriminating, and least invasive examinations.

15.8.2.1. The stereomicroscopical characteristics are sometimes apparent without sample preparation. For a more definitive evaluation, a manual or microtome cross section can employed.

15.8.2.2. General Characteristics of Solid Material The following characteristics shall be documented in the case record when applicable:

15.8.2.2.1. The general form of the material (e.g. solid, powder, stain, tablet, or smear).

15.8.2.2.2. The amount of the material present.

15.8.2.2.3. The homogeneity of the material.

15.8.2.2.4. If more than one type of material is present they all should be characterized.

15.8.2.2.5. The color and texture of each of the material(s).

15.8.2.2.6. The hardness of the material(s).

15.8.2.2.7. The shape of the material (e.g. cubic).

15.8.2.2.8. The number of layers present (if applicable).

15.8.2.3. General Characteristics of Liquids The following characteristics shall be documented in the case record when applicable:

15.8.2.3.1. Amount of the liquid present. If in limited quantities the testing may need to be done with special considerations (e.g. under the microscope).

15.8.2.3.2. Color of the liquid. The clarity or transparency of the liquid shall be noted as well.

15.8.2.3.3. The number of layers present or any liquid interface.

15.8.2.3.3.1. The miscibility of the liquid.

15.8.2.3.3.2. If the item is a liquid, check for sediments and suspensions. Foaming upon shaking may indicate soap or detergent.

15.8.2.3.4. Viscosity of the liquid. This can often be evaluated in the submitted vial that the item was submitted in.

15.8.2.3.5. Test the pH of liquid sample and if possible compare to the pH of a control. If the pH is unusual, test the item for acids or bases. The most common acids and bases are hydrochloric acid and sodium hydroxide.

15.8.2.3.6. If the liquid is organic in nature. If the liquid is flammable the spoon test as described in the fire debris test method can be employed.

15.8.3. Spot Tests or Micro-chemical Tests Spot and micro-chemical tests are used in a variety of examinations including substance identifications; however, most tests

are presumptive and are to be considered only one part of an overall identification scheme. The degree to which a spot test may be used for an identification method depends on its specificity. This procedure lists a number of the more commonly used spot and micro-chemical tests for chemical analysis. Each test observation and analytical result shall determine the future course of analysis.

15.8.3.1. Any spot test or micro-chemical test method in a peer reviewed journal or academic text shall be considered valid and appropriate for use provided that reference materials and blanks are tested prior to use in casework.

15.8.3.1.1. Literature references for methods not described in this test method shall be kept in the case record.

15.8.3.2. Reagents are described for each test. Each reagent prepared shall be recorded in the reagent log and tested with known reference material prior to use. As the reagents are infrequently used, all reagents shall be tested with known standards prior to additional testing on case samples.

15.8.3.2.1. Positive and negative controls shall be run on all spot & micro-chemical tests and the results recorded in the case notes. Additional negative controls may be useful in order to determine what may interfere with the test.

15.8.3.3. Diphenylamine test for oxidizers including nitrates and chlorates

15.8.3.3.1. Dissolve 0.5mg of diphenylamine in 5mL concentrated sulfuric acid.

15.8.3.3.2. Add a small amount of the unknown to a spot well.

15.8.3.3.3. Add two drops of diphenylamine solution.

15.8.3.3.4. A dark blue color indicates the presence of oxidizers.

15.8.3.4. Hydrochloric acid test for bicarbonates, carbonates, hypochlorites, nitrates, nitrites and cyanides

15.8.3.4.1. Acidify test sample with drops of hydrochloric acid.

15.8.3.4.2. Gas evolution indicates bicarbonates, carbonates, cyanides, hypochlorites (bleach), nitrates, or nitrites. Use caution, as cyanide gas is very poisonous.

15.8.4. Polarized Light Microscope Evaluation

15.8.4.1. Microscopic examinations may lead to identification of the unknown. Starch, building materials, plant material, and other substances can be identified using the microscope. Morphological features which are unique are very

significant.

15.8.5. Instrumental Analysis

15.8.5.1. Instrumental analysis offers a method to characterize solids and liquids both organically and inorganically. The initial step of the instrumental analysis shall normally follow a set protocol using instrument parameters that are similar to one used in one of the sub-discipline test methods or in a published testing protocol.

15.8.5.1.1. The instrument parameters shall be noted in the case record.

15.8.5.1.2. Testing following guidelines from other sources shall be referenced and included in the case record.

15.9. Records: Written descriptions, sketches, photography, or other imaging methods shall be used to document each sample's characteristics.

15.9.1. Preliminary examination of physical evidence for a general chemical analysis shall include its general appearance macroscopically and under a stereomicroscope, including any adhering matter.

15.9.2. The case record shall include the instrumental data. All hard copies shall include a unique sample designation, the operator's name/initials, and the date of analysis. Case notes shall also include a description of the evidence analyzed, the method of sample preparation, the analytical instrumentation used, and its operating parameters.

15.10. Interpretations of Results:

15.10.1. Typically, a physical and chemical examination involves the comparison of samples to determine if they could share a common origin. The goal is to determine if any significant differences exist between the samples. The physical and chemical characteristics are evaluated to associate the questioned and known sample. If a significant difference in a characteristic exists the samples are excluded as having a common origin. If the examination doesn't reveal any significant differences it is concluded that the samples could share a common origin.

15.10.2. A general chemical examination can be conducted without a comparison sample. In these examinations an attempt is made to classify or identify a solid material or liquid. In these cases the overall substance is normally evaluated with little regard to trace quantities of other materials that may be present in limited quantity.

15.11. Report Writing:

15.11.1. If significant differences are found in the physical or chemical characteristics between a questioned and a known item, then the report shall state that the items are not consistent and did not come from the same source.

15.11.2. If no significant differences are found in the physical and chemical characteristics, then the report shall state that the items are consistent and could have come from the same source or another source with these same characteristics.

15.11.3. In some circumstances the material can't be fully evaluated. This can be due to the nature of the material, lack of expertise on the forensic scientist, or lack of instrumentation. In this circumstance it shall be stated in the report.

15.11.4. An unknown material may be classified or identified based on the physical and chemical properties.

15.11.5. The instruments used in the analysis will be stated on the certificate of analysis.

15.12. References:

15.12.1. SWGMAT Trace Recovery Guidelines [Online] (October 1999). Available: http://www.fbi.gov/hq/lab/fsc/backissu/oct1999/index.htm

15.12.2. Chamot, Emil and Mason, Clyde. *Handbook of Chemical Microscopy.* 2nd Edition. Chicago: McCrone Research Institute, 1989

15.12.3. Fiegl and Anger. *Spot Tests in Inorganic Analysis.* 6th Edition. Cambridge: Elsevier Publishing Co., 1972

15.12.4. Jungreis, Ervin. *Spot Test Analysis-Clinical, Environmental, Forensic, and Geochemical Applications.* New York: John Wiley & Sons, 1985

15.12.5. McCrone and Delly. *The Particle Atlas.,* 2nd Edition. Ann Arbor: Ann Arbor Science, 1973

APPENDIX 1 WORKSHEETS

1. **Glass Technical Review Sheet**
2. **Paint Technical Review Sheet**
3. **Fiber Technical Review Sheet**
4. **Tape Technical Review Sheet**
5. **Ignitable Liquid Residue Review Sheet**
6. **Lamp Technical Review Sheet**
7. **Clandestine Laboratory Technical Review Sheet**
8. **Unknown Technical Review Sheet**
9. **GRIM III Maintenance/Repair Log Sheet**
10. **GRIM III Quality Control Log Sheet**
11. **XRF Maintenance/Repair Log Sheet**
12. **XRF Quality Control Log Sheet**
13. **SEM Maintenance/Repair Log Sheet**
14. **SEM Quality Control Log Sheet**
15. **FTIR Maintenance/Repair Log Sheet**
16. **FTIR Quality Control Log Sheet**
17. **Densitometer Quality Control Log Sheet**
18. **Digital Caliper Quality Control Log Sheet**
19. **Refractive Index – GRIM** 3 **Worksheet**

APPENDIX 2 ABBREVIATIONS

1. **ASTM – AMERICAN SOCIETY FOR TESTING AND MATERIALS**
2. **ATR - ATTENUATED TOTAL REFLECTION**
3. **BOPP - BIAXIALLY ORIENTATED POLYPROPYLENE**
4. **FTIR - FOURIER TRANSFORM INFRARED SPECTROMETRY**
5. **GC/MS – GAS CHROMATOGRAPHY MASS SPECTROMETER**
6. **GRIM – GLASS REFRACTIVE INDEX MEASUREMENT INSTRUMENT**
7. **Lsec – LIVE SECONDS**
8. **ml - MILLILITER**
9. **MOPP - MONOAXIALLY ORIENTED POLYPROPYLENE**
10. **MSDS - MATERIAL SAFETY DATA SHEET**
11. **OEM – ORIGINAL EQUIPMENT MANUFACTURER**
12. **PDQ – PAINT DATABASE QUERY**
13. **PLM – POLARIZED LIGHT MICROSCOPY**
14. **PSA – PRESSURE SENSITIVE ADHESIVE**
15. **Py-GC/MS – PYROLYSIS GAS CHROMATOGRAPHY MASS SPECTROMETER**
16. **QAM – QUALITY ASSURANCE MANUAL**
17. **RCMP – ROYAL CANADIAN MOUNTED POLICE**
18. **SEM - SCANNING ELECTRON MICROSCOPY**
19. **SEM/EDS – SCANNING ELECTRON MICROSCOPY ENERGY DISPERSIVE SPECTROSCOPY**
20. **SWGMAT – SCIENTIFIC WORKING GROUP FOR MATERIAL ANALYSIS**
21. **XRF – X-RAY FLOURESCENCE**
22. **PFTBA - PERFLUOROTRIBUTYLAMINE**

APPENDIX 3 DEFINITIONS

1. **ABSORBANCE, *A*** The logarithm to the base 10 of the reciprocal of the transmittance, (T). $A = \log_{10}(1/T) = -\log_{10}T$.
2. **ABSORPTION BAND** A region of the absorption spectrum in which the absorbance passes through a maximum.
3. **ABSORPTION SPECTRUM** A plot or other representation of a function of absorbance against any function of wavelength.
4. **ABSORPTIVITY** An absorbance divided by the product of the sample pathlength (b) and the concentration of the absorbing substance (c). The units of b and c shall be specified. a = A/bc.
5. **ACCELERANT** Any substance, usually a liquid, which is used to increase, and sometimes direct the spread of fire.
6. **ADHESIVE** A material that will hold two or more objects together solely by intimate surface contact.
7. **ADDITIVES** Materials that are added in small quantity to improve properties of items such as paint and tapes. Additives may include substances such as dryers, corrosion inhibitors, catalysts, ultraviolet absorbers, and plasticizers. Additives increase overall volume, impart color, or provide other desired properties.
8. **ADSORPTION** The attraction between the surface atoms of a solid and an external molecule by intermolecular forces.
9. **AGE SAG** downward stretching of horizontal filaments caused by the force of gravity on incandescent filaments.
10. **AMPLITUDE** The height of a wave.
11. **ANALYTICAL BLANK** A sample containing all reagents and materials in the proportions used to prepare a sample, processed in the same manner without the sample itself.
12. **ANALYTICAL SAMPLE** The portion of a sample that is actually used for testing.
13. **ANNEALING** The process of reducing residual strain in glass by controlled heating and cooling.
14. **ANISOTROPIC** A material that exhibits properties with different values when measured along axes in different directions.
15. **APERTURE** An opening in an optical system that controls the amount of light passing through a system.
16. **ARCING** Flow of current between two conductors without any physical contact.
17. **ARSON** The willful, malicious, or fraudulent burning of a person's property.
18. **ATTENUATED TOTAL REFLECTION, (ATR)** A method of spectrophotometric analysis based on the reflection of energy at the interface of two media which have different refractive indices and are in intimate contact with each other; also known as Internal Reflection Spectroscopy (IRS).
19. **AUTOGENOUS IGNITION TEMPERATURE** The temperature at which a combustion reaction becomes self-sustaining.
20. **BACKGROUND** Electrical noise caused by anything other than the substance for which the analysis is being made.
21. **BACKGROUND X-RAYS** (Bremsstrahlung, braking radiation, continuous spectrum): Nonspecific X-ray radiation with a continuous energy range from zero up to the beam voltage. Background radiation results from the deceleration of beam electrons in the

atomic Coulombic field. A typical X-ray spectrum consists of both a continuous background and peaks from characteristic X-rays.

22. **BACKING** A thin flexible material to which adhesive is applied.
23. **BACKSCATTERED ELECTRONS (BE)** Primary beam electrons that are scattered from the sample after undergoing few inelastic interactions. The probability of backscattering is proportional to the atomic number.
24. **BACKSIZING** A layer applied to the top side of the backing. Its purpose is to coat and fill a porous surfaced backing with a material that is inert to the adhesive formulation to be used.
25. **BEAM CONDENSER** A series of mirrors that focus the infrared beam to a small area in the sample compartment to permit the examination of smaller samples than would otherwise be possible.
26. **BEAM SPLITTER** An optical component that partially reflects and partially transmits radiation from the source in such a manner as to direct part to a fixed mirror and the other part to a moving mirror.
27. **BECKE LINE** The bright halo near the boundary of a fiber that moves with respect to that boundary as the microscope is focused through best focus.
28. **BECKE LINE METHOD** A method for determining the refractive index of a fiber relative to its mountant by noting the direction in which the Becke line moves when the focus is changed. The Becke line will always move toward the higher refractive index medium (fiber or mountant) when focus is raised and will move toward the lower refractive index medium when focus is lowered.
29. **BIAXIALLY ORIENTATED POLYPROPYLENE (BOPP)** An oriented polypropylene film in which the polymer has been stretched in both the machine direction and cross direction in the manufacturing process. Tapes with such films cannot be torn by hand.
30. **BINDER** A nonvolatile portion of the paint which serves to bind or cement the pigment particles together.
31. **BIREFRINGENCE** The numerical difference in principle refractive indices for a substance. Given by the formula: $n_{||} - n_{\perp}$. Birefringence can be calculated by determining the retardation (r) and thickness (T) at a particular point in a fiber and by using the formula: B = r (nm)/1,000 T (mm).
32. **BLEEDING** A defect in which pigment from a lower coat of paint diffuses into an upper coat and discolors the latter.
33. **BLISTERING (BUBBLING)** The occurrence of air or solvent vapors as small bubbles in a paint film.
34. **BLOOMING** A powder-like deposit which sometimes forms on the surface of a paint film.
35. **BRAIDED** A laid rope manufactured by twisting fibers together into yarns, yarns into strands, and strands into rope. The lay of the rope is the spiral twist of the strands. This rope resembles plaited rope in construction except that it is made up of a great number of yarns instead of strands. Braided rope is balanced by having an equal number of left-hand and right-hand turns. This is actually two ropes, one inside the other. The outer rope is called the cover or sheath and the inner one, the core; both are braided.
36. **BRITTLE FRACTURE** A fracture caused by vibration to an aged brittle filament (after constant heating and cooling the filament can become crystalline). The fracture will be square or with sharp projections at the ends.
37. **BULK ANALYSIS** A type of scanning electron microscopy analysis that determines the representative elemental composition of a material. The area of analysis is as large as possible and may be achieved by a single large area raster or the summed results from multiple smaller area rasters.

38. **BURNOUT(NORMAL/NATURAL)** When the tungsten evaporates from the filament making areas for a buildup in electrical resistance which increases to the melting point of tungsten causing the filament to part. Usually resulting in a ball at one end of the filament and a point on the other end.
39. **C-LINE** 656 nm wavelength (A Fraunhofer line in the spectrum of the sun due to hydrogen emission.)
40. **CALENDERING** The use of a multi-roll device to apply pressure sensitive adhesive at 100% solids to various backings by heat and pressure to produce adhesive tape.
41. **CAPILLARY COLUMN** A columnar assembly of a thin film on the inner periphery and an unobstructed and open lumen running the entire length of the column, which acts as the stationary phase or plate of the chromatograph.
42. **CELLOPHANE** Form of regenerated cellulose. A thin transparent film manufactured from wood pulp. Used as a backing material in tape products.
43. **CELLULOSE ACETATE** A transparent film that is used for tape backings. A matte surface version is used for write-on tapes. It is more moisture-resistant than cellophane.
44. **CELLULOSIC FIBER** Fiber composed of polymers formed from glucose.
45. **CETANE NUMBER** Used to express the ignition characteristics of diesel fuel.
46. **CHALKING** The degradation of a paint film by gradual erosion of the binder. White powdery formation on surfaces of exterior paints.
47. **CHARACTERISTIC X-RAYS** X-ray emission resulting from de-excitation of an atom following inner shell ionization. The energy of the X-rays is related to the atomic number of the atom, providing the basis for energy dispersive X-ray spectroscopy. A typical X-ray spectrum consists of both a continuous background and peaks from characteristic X-rays.
48. **CHARGING** Negative charge accumulation on either a nonconductive sample or a sample that is not properly grounded. This effect may interfere with image formation and X-ray analysis because of beam deflection. It can usually be eliminated by the application of a conductive coating or by the use of a low vacuum system.
49. **CHECKING** Hairline cracks in a coating.
50. **CHROMATOGRAPHY** A method of analysis in which substances are separated by their differential migration rates.
51. **CLASS** A group of items that share properties or characteristics.
52. **CLASS CHARACTERISTICS** Traits that define a group of items collectively.
53. **CLASSIFICATION** The placement of a specimen into a particular product-use or manufacture source category based upon the comparison of measured attributes with a database of known attributes for each category.
54. **COATING** A generic term for paint, lacquer, enamel, or other liquid material that is converted to a solid, protective, or decorative film after application.
55. **COLD SHOCK** A fracture caused by an impact to a non-incandescent filament. The fracture will be square or with sharp projections at the ends.
56. **COLOR** A property of light dependent upon the frequencies of light waves striking the retina.
57. **COLORIMETRY** An analytical method for measuring the color intensity of a substance.
58. **COMBUSTION** A relatively slow oxidation, usually involving carbonaceous substances. Requires fuel, heat, and oxygen.
59. **COMBUSTIBLE LIQUID** A liquid having a flash point above 100 degrees Celsius.
60. **COMPARISON MICROSCOPE** A system of two microscopes positioned side by side and connected via an optical bridge in which specimens are examined simultaneously in either transmitted or reflected light.

61. **COMPENSATOR** Any variety of optical devices that can be placed in the light path of a polarizing microscope to introduce fixed or variable retardation comparable with that exhibited by the fiber. The retardation and sign of elongation of the fiber is then determined. Compensators can employ a fixed mineral plate of constant or varying thickness or a mineral plate that is rotated to alter the thickness presented to the optical path (and retardation introduced) by a set amount.
62. **COMPENSATOR, BEREK** A compensator typically containing a plate of calcite or quartz, which can be rotated by means of a calibrated drum to introduce variable retardation up to about ten orders.
63. **COMPENSATOR, FULL WAVE** A compensator using a plate of gypsum, selenite, or quartz, which introduces a fixed retardation between 530-550 nm (approximately the retardation of the first order red color on the Michel-Lévy chart).
64. **COMPENSATOR, SENARMONT** A quarter-wave plate inserted above the specimen in the parallel 0" position with a rotating calibrated analyzer. Measures low retardation and requires the use of monochromatic light.
65. **COMPENSATOR, QUARTER WAVE** A compensator, usually with a mica plate, which introduces a fixed retardation between 125-150 nm.
66. **COMPENSATOR, QUARTZ WEDGE** A wedge, cut from quartz, having continuously variable retardation extending over several orders of interference colors (usually 3-7).
67. **CONCENTRIC FRACTURE** A fracture of a pane of glass which is concentric to the breaking force (distinguished from a radial fracture). It is also called a spiral, or tangential fracture.
68. **CONTACT** The part of a lamp that makes the electrical connection between the lamp and the electrical socket.
69. **CONCHOIDAL MARKS** Shell-like markings on the broken edge of a piece of glass which are perpendicular to one surface of the glass and asymptotically parallel to the other surface. This type of fracture is irregularly curved and usually a striated surface.
70. **CONDUCTION** Transfer of heat from one body to another through direct physical contact.
71. **CONE (CRATER)** A funnel-shaped area of damage caused by a high-velocity impact.
72. **CONFLAGRATION** Large destructive fire.
73. **CONVECTION** Transfer of heat by a circulating medium, usually a gas or a liquid.
74. **CORDAGE** A comprehensive word for any line, whatever its size or material, which has no special purpose.
75. **CORE** A fiber or fibers running lengthwise through the center of cordage.
76. **CORTEX** The main structural component of hair consisting of elongated and fusiform (spindle-shaped) cells. The cortex contains pigment grains, air spaces called cortical fusi, and structures called ovoid bodies.
77. **CRACKING** Splitting of a paint film. Usually occurs as straight lines which penetrate the entire film thickness.
78. **CRATERING (FISHEYES)** Small round depressions in a paint film which may or may not expose the underlying surface.
79. **CRAZING** The formation of surface cracks, often as a fine network, which do not penetrate to the underlying surface. Crazing is sometimes caused by the softening effect of solvents from successive paint coats.
80. **CREPED** Paper that has small folds in it giving it high stretch and conformability. Used in masking tape (saturated paper tape).
81. **CRIMP** The waviness of a fiber.

82. **CRITICAL ANGLE** The angle of incidence for which the refracted ray emerges tangent to the surface.
83. **CROSS DIRECTION** The direction of the tape that runs across the width of the tape.
84. **CROSS OVER MARKS** Oblique flattened areas along silk fibers caused by the overlapping of extruded silk fibers before they have dried.
85. **CROWN** The raised portion of a strand in a twisted cordage.
86. **CUT** A separation of fabric defined by an evenness of damage margin.
87. **CUTICLE** The layer of scales composing the outer surface of a hair shaft. Cuticular scales are normally classified into three basic types: coronal (crown-like), spinous (petal-like), and imbricate (flattened).
88. **D-LINE** 589 nm wavelength (A Fraunhofer line in the spectrum of the sun due to sodium.)
89. **DEAD TIME** In x-ray spectrometry is the amount of time that a detector is receiving a signal that is not being counted.
90. **DELUSTERANT** An agent used to alter the light reflected from a fiber causing a dulling effect.
91. **DENSITY** The ratio of the weight of a substance to its volume
92. **DEUTERATED TRIGLYCINE SULPHATE (DTGS) DETECTOR** A thermal detector that operates at room temperature but lacks the sensitivity for use with microscope accessories.
93. **DICHROISM** The property of exhibiting different colors, especially two different colors, when viewed in polarized light along different axes.
94. **DIESEL FUEL** Petroleum distillates used in diesel engines.
95. **DISCRIMINATE** To distinguish between two samples on the basis of significant differences; to differentiate.
96. **DISCRIMINATING POWER** The ability of an analytical procedure to distinguish between two items of different origin.
97. **DISLOCATIONS** Characteristics in natural fibers (e.g., flax, ramie, jute, and hemp) where distinct features in the shape of X's, I's, and V's are present along the fiber cell wall. These features are often useful for identification.
98. **DISPERSION** The difference between the refractive indices of a substance in different wavelengths of light: For minerals: $n_F - n_C$ or F – C, For glass: $(n_D - 1) / (n_F - n_C)$ or (D - 1) / (F - C) where n = Refractive Index, and C, D, and F are the respective Fraunhofer line wavelengths.
99. **DISPERSION COATING** A type of paint in which the binder molecules are present as colloidal particles.
100. **DISPERSION CURVE** The graph of the refractive index of a substance in different wavelengths of light. Obtained on glass by varying the wavelength of the light passing through the glass and its immersion liquid, and by varying the temperature of the immersion liquid to obtain matches at several points (Emmon's double variation method)
101. **DISPERSION STAINING** A procedure involving central or annular stops in the objective back focal plane to induce colored images of transparent particles mounted in liquids with indices matching the particle at a wavelength in the visible range.
102. **DOUBLE-PASS TRANSMISSION SPECTRA** Spectra that results from incident radiation passing through the sample, reflecting off the substrate, and passing through the sample a second time.
103. **DRIER** A material that promotes or accelerates the drying, curing, or hardening of oxidizable coating vehicles. The principal driers are metal soaps of a monocarboxylic acid.

104. **DRYING OILS** Naturally occurring triglycerides which form films principally by air oxidation. The same oils may be used as feedstocks for varnishes, alkyd resins, epoxy ester resins, oil modified urethane resins, and some plasticizers.
105. **DUCT TAPE** Fabric-reinforced tape used for air duct installation or for general utility applications
106. **DYE EXTRACTION** The removal of the dye from a fiber by incubating it in an appropriate solvent.
107. **DYES** Soluble substances that add color to textiles. Dyes are classified into groups that have similar chemical characteristics (e.g., aniline, acid, and azo). They are incorporated into the fiber by chemical reaction, absorption, or dispersion.
108. **ELASTOMER** A material that can be deformed, but when the forces are removed will return to its original form. Serves as the base material for PSAs.
109. **ELECTRICAL TAPE** PVC-backed tape with specific dielectric properties designed for electrical applications
110. **ELONGATION** When a filament is hot (incandescent) and a force is applied to the bulb, the filament can stretch. This abnormality suggests that the filament was incandescent at the time.
111. **ENAMEL** The term enamel does not intimate the chemical nature of the coating, but implies a pigmented coating which dries to a hard gloss. Increasingly, the term has come to mean a cross-linked thermosetting resin.
112. **ENDOTHERMIC REACTION** Requires more energy than what is given off (cools).
113. **ESCAPE PEAK** A peak resulting from incomplete deposition of the energy of an X-ray entering the energy dispersive X-ray spectrometer detector. This peak is produced when an incoming X-ray excites a silicon atom within the detector crystal, and the resulting Si K-α fluorescence X-ray exits the detector crystal. It occurs at the principal peak energy minus the energy of the Si K-α fluorescence X-ray (1.74KeV). The escape peak intensity is about 1-2 percent of the parent peak.
114. **ETCHING (GLASS)** A surface design or pattern produced by the application of a corrosive acid.
115. **ETCHING (LAMPS)** The marking or distortion of glass when an incandescent filament comes In contact with the glass bulb.
116. **EXCITATION FILTER** A filter used in fluorescence microscopy that transmits specific bands or wavelengths of energy capable of inducing visible fluorescence in various substrates.
117. **EXTENDER** A low cost product used to modify the gloss, texture, viscosity, other properties, and to reduce the cost of the finished product (used in adhesives and paint).
118. **EXTINCTION ANGLE** As it applies to tape, the angle between the machine edge of a clear oriented polymer tape film under crossed polars and the point of extinction (appears dark under crossed-polars).
119. **EXTRANEOUS MATERIAL** (contaminant, foreign material): Material originating from a source other than the specimen.
120. **EXOTHERMIC REACTION** Energy is given off (heat).
121. **F-LINE** 486 nm wavelength (A Fraunhofer line in the spectrum of the sun due to hydrogen.)
122. **FAR-INFRARED** The infrared region of the electromagnetic spectrum with wavelength range from approximately 25 to 1000µm (wavenumber range 400 to 10 cm^{-1}).
123. **FIBER** The smallest element of material forming the individual fibers of rope.
124. **FILAMENT(LAMP)** The part of the lamp that light is emitted from. It is normally made from tungsten which heats as electricity passes through it.

125. **FILAMENT TAPE** A fiber-reinforced tape in which the reinforcing fibers are only in the warp direction. Also referred to as strapping tape
126. **FILL YARNS** Fibers in the scrim fabric of reinforced tape that run crosswise, perpendicular to the warp direction. Also called weft yarns.
127. **FILLER/EXTENDER** An inorganic material that is added to a tape to modify a physical property or reduce cost.
128. **FINAL APERTURE** The last beam-restricting orifice in an electron optical column. The orifice diameter influences the beam current and depth of focus.
129. **FIRE** Rapid oxidation with the evolution of light and heat.
130. **FLATBACK** Smooth paper backing sometimes used in masking tapes.
131. **FLAME (FIRE) POINT** Temperature at which a liquid produces enough vapor to sustain a flame; slightly higher than the flash point temperature.
132. **FLAMMABLE LIQUID** A liquid having a flash point below 100 degrees Celsius.
133. **FLAMMABLE RANGE** Range of values, expressed as a percentage of the gaseous fuel to air mixture, which will support combustion (between lower and upper flammable ranges).
134. **FLASH POINT** Lowest temperature at which a liquid produces sufficient vapor to cause a flash when ignited.
135. **FLOATING (MOTTLE)** The tendency of pigment particles in a wet paint film to separate from one another and concentrate in particular areas.
136. **FLOODING** Incomplete color pigment dispersion in the base paint. Produces a streaked, non-uniform color when applied.
137. **FLUORESCENCE** The emission of electromagnetic radiation, especially of visible light, stimulated in a substance by the absorption of incident radiation of a shorter wavelength (usually ultraviolet) and persisting only as long as the stimulating radiation is continued.
138. **FOURIER TRANSFORM (FT)** The mathematical process which is used to convert an amplitude-time-spectrum to an amplitude-frequency spectrum, or *vice versa*. In FTIR spectrometry, retardation is directly proportional to time; therefore, FT is commonly used to convert an amplitude-retardation spectrum to an amplitude-wavenumber spectrum, and *vice versa*.
139. **FOURIER TRANSFORM INFRARED SPECTROMETRY (FTIR)** A form of infrared spectrometry in which an interferogram is obtained; this interferogram is then subjected to a Fourier transform to obtain an amplitude-wavenumber (or wavelength) spectrum.
140. **FRAUNHOFER LINES** A set of several hundred dark lines appearing against the bright background of the continuous solar spectrum and produced by absorption of light by the cooler gases in the sun's outer atmosphere at frequencies corresponding to the atomic transition frequencies of these gases. (Named for Joseph von Fraunhofer, Bavarian physicist, 1787 - 1826.)
141. **FREQUENCY** The number of wavecrests which pass by per second. $F = \nu/\lambda$, where F is frequency, ν is the velocity of light, and λ is the wavelength of the light.
142. **FROSTING** A finely grained, slightly roughened surface texture for glass.
143. **FUEL OILS** Consist largely or entirely of the residue from the distillation of the more asphaltic crude oils; used as a fuel for marine vessels and locomotives and for heating purposes.
144. **FUSED GLASS** Glass which has come in contact with an incandescent filament and has melted onto the filament.
145. **GAS CHROMATOGRAPH** A commonly used laboratory instrument used to separate the components of a mixture.

146. **GASOLINE** A blended volatile liquid fuel used in spark ignition engines; a mixture of low to medium boiling hydrocarbons.
147. **GENERIC CLASS** A group of fibers having similar (but not identical) chemical composition. A generic name applies to all members of a group and is not protected by trademark registration. Generic names for manufactured fibers include, for example, rayon, nylon, and polyester. Generic names used in the United States for manufactured fibers were established as part of the Textile Fiber Products Identification Act enacted by Congress in 1954 (12).
148. **GLASS** An amorphous solid, usually a silicate, usually transparent or translucent – a supercooled liquid. It is ordinarily homogeneous.
149. **GLASS BULB** This is the glass globe or envelope which encases the filament and support poles of a lamp.
150. **GLOB** The ball shaped end of a broken lamp filament that results from normal burnout.
151. **GLOWING COMBUSTION (SMOLDERING)** Combustion at the surface of a solid fuel in the absence of heat sufficient to cause Pyrolysis; combustion without the presence of a flame.
152. **HACKLE MARKS** Marks on the broken edge of a piece of glass which are perpendicular to the conchoidal fracture marks.
153. **HAMMERTONE FINISH** A finish which makes the surface look as though it has been peened.
154. **HARTMAN GRAPH PAPER** Graph paper constructed so that dispersion curves of liquids and perfectly annealed glasses are straight lines.
155. **HEAT OF COMBUSTION** The excess energy (heat and often light) liberated from a reaction.
156. **HIGH INTENSITY DISCHARGE (HID) LAMP** A lamp where light is emitted due to a plasma discharge.
157. **HOT BREAK** The parting of a lamp filament due to the stretching caused by an impact. The ends of the filament are tapered or beaded.
158. **HOT SHOCK** A term to describe the possible abnormality when an incandescent filament is exposed to an external force such as an accident.
159. **IGNITION TEMPERATURE** The minimum temperature to which a fuel shall be heated in air to ignite independently of the heating source; the temperature necessary for a solid fuel to decompose and produce flammable vapor.
160. **INCANDESCENT** When a filament is hot enough to glow or emit light.
161. **INDIVIDUAL** A unique item that is identified as itself to the exclusion of all other items.
162. **INDIVIDUAL CHARACTERISTICS** Traits that define and identify an item as unique and exclusive of all other items.
163. **INERT GAS** A gas that is added to a sealed bulb. It is usually nitrogen or a halogen (for quartz halogen bulbs).
164. **INFRARED** The region of the electromagnetic spectrum with wavelength range from approximately 0.78 to 1000µm (wavenumber range 12,800 to 10 cm^{-1}).
165. **INFRARED SPECTROSCOPY (IR)** the study and interpretation of spectra within the infrared region of the electromagnetic spectrum.
166. **INORGANIC FIBERS** A class of fibers of natural mineral origin (e.g., chrysotile asbestos) and manmade mineral origin (e.g., fiberglass).
167. **INTERACTION VOLUME** The sample volume in which the electron beam loses most of its energy. It is generally thought of as the volume in which detectable X-rays are produced.

The actual volume varies depending upon beam voltage, average atomic number, and density of the sample.

168. **INTERFERENCE COLORS** Colors produced by the interference of two out-of-phase rays of white light when a birefringent material is observed at a nonextinction position between crossed polars. The retardation at a particular point in a birefringent fiber is determined by comparing the observed interference color to the Michel-Lévy chart.
169. **INTERFEROGRAM** A plot of the detector output as a function of retardation.
170. **INTERFEROMETER** A device used to split a beam of radiant energy into two paths, generate an optical path difference between the beams, and recombine them in order to produce repetitive interference maxima and minima as the optical retardation is varied.
171. **INTERNAL REFLECTION ELEMENT** A high refractive index material (e.g., ZnSe, Ge, or diamond) used as a substrate for the sample to internally reflect the IR electromagnetic radiation.
172. **INTERNAL REFLECTION SPECTROSCOPY (IRS)** See Attenuated Total Reflection (ATR)
173. **INTERNAL STANDARD** An element either inherent in or added to samples and calibration standards at a known concentration. It is used to correct for differences in sensitivity between samples or among samples and standards.
174. **ISOTROPIC** A material that exhibits properties of identical value when measured along axes in different directions.
175. **ISOTROPY** The property of having the same refractive index regardless of the direction of vibration of light passing through the material. An isotropic particle will exhibit extinction at all orientations between crossed polars.
176. **KEROSENE** A petroleum distillate product in the carbon range C_9 to $C_{16.}$ used for illumination and heating.
177. **KLM REFERENCE LINES** The energies associated with the transitions of the K, L, and M shell electrons. Each element will have a different series of KLM reference lines.
178. **KNIT** A fabric produced by interlooping one or more ends of yarn or comparable material.
179. **KNOWN SAMPLE** A sample of established origin.
180. **LACQUER** Fast-drying coatings, clear or pigmented, that dry by evaporation of the solvent rather than by oxidation or polymerization.
181. **LARGE FILAMENT** The larger diameter filament in a two filament bulb.
182. **LATEX** A suspension of a pigment in a water-based emulsion of any of several resins, for example, acrylic polymers, vinyl polymers, or styrene-butadiene polymers.
183. **LAY** The direction, either left-handed or right-handed, of twist in the strands of rope; also the nature (tight, medium, or loose) of that twist.
184. **LIGHT EMITTING DIODE (LED) LAMP** A lamp where a semiconductor is designed to emit light with electroluminescence.
185. **LIGHT MICROSCOPE** A microscope that employs light in the visible or near-visible portion of the electromagnetic spectrum.
186. **LIGNIN** The majority noncarbohydrate portion of wood. It is an amorphous polymeric substance that cements cellulosic fibers together. The principal constituent of woody cell walls.
187. **LIMIT OF DETECTION** The concentration equivalent of a signal that is sufficiently above the level of the analytical blank to be replicately measured.
188. **LIVE TIME** The time over which the energy dispersive X-ray spectroscopy electronics are available to accept and process incoming X-rays. Live time is often expressed as a percentage of real time.

189. **LONG-WAVE UV ILLUMINATION** In the wavelength range from 400 nm – 315 nm with peak wavelength energy at 366 nm
190. **LOW E-GLASS** Glass that is coated with an IR reflective surface. Such glass is suitable for use as a sample support when performing IR reflection techniques.
191. **LPG** Liquified petroleum gas.
192. **LUMEN** The cavity or central canal present in many natural fibers (e.g., cotton, flax, ramie, jute, and hemp). Its presence and structure are often a useful aid in identification.
193. **LUSTER** The gloss or shine possessed by a fiber, resulting from its reflection of light. The luster of manufactured fibers is often modified by use of a delustering pigment.
194. **MACHINE DIRECTION** The direction of the tape that runs the length of the tape.
195. **MANUFACTURED FIBER** A class name for various families of fibers produced from fiber-forming substances, which are synthesized polymers, modified or transformed natural polymers, and glass.
196. **MANUFACTURING FLAW** A feature that appears to be an abnormality, however, it occurs at the time of manufacturing.
197. **MASKING TAPE** Paper-backed tape having a creped, usually beige or buff-colored backing.
198. **MASS SPECTROMETER** A laboratory instrument used to detect and identify the components that have separated.
199. **MATCH POINT** Any combination of temperature and wavelength, at which two media have indistinguishable refractive indices. At match point, the glass will exhibit minimum contrast and visibility.
200. **MECHANICAL BREAKAGE** The breakage of a lamp filament caused by a foreign object.
201. **MEDULLA** The central portion of a hair composed of a series of discrete cells or an amorphous spongy mass. It may be air-filled and, if so, will appear opaque or black using transmitted light or white using reflected light. In animal hair, several types have been defined: uniserial or multiserial ladder, cellular or vacuolated, and lattice.
202. **MERCURY CADMIUM TELLURIDE (MCT) DETECTOR** A quantum detector that utilizes a semi-conducting material and requires cooling with liquid nitrogen to operate. This type of detector is commonly used in microscope accessories due to its sensitivity.
203. **METALLIC PAINT** Paint which contains reflective metallic particles, usually in the form of tiny flakes (often aluminum).
204. **METAMERIC PAIR** Two colors that appear the same under one illumination but different under another illumination.
205. **MICHEL-LEVY CHART** A chart relating thickness, birefringence, and retardation so that any one of these variables can be determined for an anisotropic fiber when the other two are known.
206. **MICROSCOPICAL** Concerning a microscope or the use of a microscope.
207. **MID-INFRARED** The infrared region of the electromagnetic spectrum with wavelength range from approximately 2.5 to 25µm (wavenumber range 4000 to 400 c^{m-1}).
208. **MIGRATION** The movement over a period of time of an ingredient from one layer to another. This often occurs in PVC tapes where plasticizer in the PVC backing “migrates” into the adhesive.
209. **MIRROR REGION** The smooth portion of the crack surface that exhibits a mirror-like reflection when viewed using low power magnification.
210. **MODIFICATION RATIO** A geometrical parameter used in the characterization of noncircular fiber cross sections. The modification ratio is the ratio in size between the outside diameter of the fiber and the diameter of the core. It is also called aspect ratio.

211. **MOLD MARKS** The transfer impressions present on the surface of a molded object resulting from direct contact between the mold and the material being molded. (e.g. a line on a glass jar).
212. **MONOAXIALLY ORIENTED POLYPROPYLENE (MOPP)** An oriented polypropylene film in which the polymer has been stretched in one direction only in the manufacturing process. Tapes with such films can be torn by hand.
213. **MOUNTING MEDIUM** A liquid, polymer, or resin used to mount specimens for microscopical examinations.
214. **NANOMETER (NM)** A unit of linear measurement in the Metric System equal to 10^{-6} meters (0.000001, one one millionth of a meter).
215. **NATURAL FIBERS** A class name of fibers of vegetable origin (e.g., cotton, flax, and ramie), animal origin (e.g., silk, wool, and specialty furs), or of mineral origin (e.g., asbestos).
216. **NICK** Small cut or notch, sometimes at the end of a cut.
217. **NOMINAL WIDTH** The design width of the tape, usually in terms of round numbers. Measured width can vary from nominal width due to stretching or weathering.
218. **OCTANE NUMBER** Used to express the ignition characteristics of gasoline.
219. **ORANGE PEEL** An irregularity in the surface of a paint film resulting from the inability of the wet film to "level out" after being applied. Orange peel occurs as a characteristically uneven or grainy surface to the eye, but usually feels smooth to the touch.
220. **OXIDATION** The loss of electrons by an atom, ion, or molecule, leading to an increase in oxidation number.
221. **OXIDATION (LAMP)** A discoloration that results on a filament due to oxidation. Occurs when the lamp seal is broken and oxygen is allowed to enter the bulb. The filament shall have an electric current running through it or be hot for oxidation to occur.
222. **OXIDIZER** Chemical that supplies oxygen for a reaction.
223. **PACKAGING TAPE** a) Pressure-sensitive tape consisting of an oriented polymer with a brown or clear adhesive layer, or b) Paper-backed tape, which has a moistenable adhesive.
224. **PAINT** Commonly known as a pigmented coating. A material, which when applied as a liquid to a surface, forms a solid film for the purpose of decoration and/or protection. Generally, a paint contains a binder, solvent, and pigment.
225. **PARTICLE ANALYSIS** An analytical method intended to determine the elemental composition of a single particle such as a pigment particle in an adhesive. Usually performed with a static (non-scanning) electron beam.
226. **PHASE CONTRAST MICROSCOPE** An interference microscope in which contrast is enhanced by altering the optical path of the diffracted ray with respect to the direct ray. This is accomplished by the action of a phase-shifting element, which results in retarding one of the rays relative to the other.
227. **PHYSICAL END MATCH** A one-of-a-kind fit between two pieces of torn or cut ends.
228. **PIGMENT(PAINT)** A finely ground, inorganic or organic, insoluble, and dispersed particle. Besides color, a pigment may provide many of the essential properties of paint such as opacity, hardness, durability, and corrosion resistance. The term pigment also includes extenders.
229. **PIGMENT (FIBER)** A finely divided insoluble material used to deluster or color fibers (e.g., titanium dioxide and iron oxide).
230. **PILLING** Small balls of fibers on fabric caused by friction.
231. **PITCH** The number of crowns per inch of the same strand.

232. **PLAITED** A type of rope construction. An eight-strand rope is made up of four right-hand and four left-hand turned strands, laid in pairs. This type of construction resists rotation and is used for flag halyards, clothes lines, etc.
233. **PLANAR ARRAY** Ends of fibers or yarns line up in the same plane.
234. **PLANE POLARIZED LIGHT** Light that is vibrating in one plane.
235. **PLASTICIZER** Material added to plastics to impart flexibility by creating spaces between the polymer chains and lowering the inter- and intra-chain attractive forces, allowing freer movement of the chains. Used in pressure sensitive backings (particularly PVC) as well as some adhesives to lower glass-transition temperatures and allow use at sub-ambient temperatures.
236. **PLEOCHROISM** The property of exhibiting different colors, especially three different colors, when viewed in polarized light along different axes.
237. **PLY** The number of single yarns twisted together to form a plied cord. An individual yarn in a plied yarn or cord.
238. **POLARIZED LIGHT** Light in which the vibration of the electric or magnetic field is confined to one plane. Ordinary light consists of a mixture of waves vibrating in all directions perpendicular to its line of propagation.
239. **POLARIZED LIGHT MICROSCOPE (PLM)** A microscope equipped with two polarizing filters, one below the stage (the polarizer) and one above the stage (the analyzer).
240. **POPPING** A paint defect characterized by raised bumps in the surface. Caused by solvent vapor forming within the paint after it has begun to "skin-over”.
241. **PRESSURE SENSITIVE ADHESIVE (PSA)** Consists of a polymeric base usually with appropriate plasticizers and tackifiers. It can form an adhesive bond with no physical or chemical change, and with no more than slight pressure.
242. **PRESSURE SENSITIVE TAPE** Consists of a flexible backing and PSA, which when applied to a surface, bonds immediately at room temperature with slight pressure. The bond can be broken (usually) without damage to the surface and without leaving a residue.
243. **PRIMER** A type of paint applied to a surface to increase its compatibility for the top coat or to improve the corrosion resistance of the substrate.
244. **PRIME COAT** A coating of adhesive-like material between the tape adhesive and backing that serves as a bonding agent.
245. **Privileged direction (of a polarizer)** The direction of vibration to which light emerging from a polarizer has been restricted.
246. **PULSE PROCESSOR TIME CONSTANT** Operator-selected value for pulse-processing time. A higher value (longer time) results in a more accurate determination of the detector amplifier pulse height (better spectral resolution). A lower value results in a higher count rate but with reduced spectral resolution.
247. **PUNCTURE** Holes produced by a blunt instrument used in thrusting action.
248. **PYROLYSIS** Exposure to heat causing decomposition of solids into gaseous products.
249. **QUESTIONED SAMPLE** A sample whose original source is unknown.
250. **RADIAL FRACTURE** A fracture of a pane of glass which extends radially away from the breaking force.
251. **RADIATION** Transfer of heat from one body to another by heat waves or rays through the intervening air or space existing between the two bodies.
252. **RASTER** The rectangular pattern scanned by the electron beam on a sample. The raster dimensions change inversely with magnification.
253. **REFLECTIVE PAINT** Paint which contains reflective particles, sometimes in the form of glass beads or reflective particles in the form of tiny flakes.

254. **REFRACTION** The change in direction observed when light passes obliquely from one medium to another in which it travels with a different velocity.
255. **REFRACTIVE INDEX** The ratio of the velocity of light in a vacuum to the velocity in some medium.
256. **REINFORCEMENT** Cloth, scrim, glass filaments, or plastic filaments added to tape for stability and strength.
257. **RELATIVE REFRACTIVE INDEX** The estimate of the refractive index of a substance (e.g. fiber) in relation to the index of its surrounding medium.
258. **RELATIVE STANDARD DEVIATION** The standard deviation divided by the mean.
259. **RELEASE COAT** A coating applied to the backing on the side opposite the adhesive that provides ease of unwind and prevents delamination or tearing.
260. **RESOLUTION** The ability to visually separate two things.
261. **RETARDATION** The actual distance of one of the doubly refracted rays behind the other as they emerge from an anisotropic substance. The amount of retardation is dependent on the difference between the two refractive indices and the thickness of the material.
262. **RIB MARKS** Conchoidal marks.
263. **ROPE** Cordage over .4 inch in diameter.
264. **SAMPLE** (representative sample): A representative portion of the specimen selected and prepared for analysis that is believed to exhibit all of the elemental characteristics of the parent specimen.
265. **SCANNING ELECTRON MICROSCOPY (SEM)** A type of electron microscope in which a focused electron beam is scanned in a raster on a solid sample surface. The strength of resulting emissions of signals varies according to sample characteristics such as composition or topography. These signals directly modulate the intensity of the display cathode ray tube. The electron beam of the scanning electron microscope and the display cathode ray tube are scanned synchronously, resulting in a two-dimensional image of the sample. By popular usage, the term scanning electron microscopy may also include the analytical techniques energy dispersive X-ray spectroscopy and wavelength dispersive X-ray spectroscopy.
266. **SCRIM** A loosely-woven gauze-type cloth added to duct tape for reinforcement and strength
267. **SCRIM COUNT** The dimensional count of the scrim, in terms of threads per inch, expressed as warp count by fill count
268. **SEAL (LAMP)** A condition which protects the filament from oxidation. It makes the bulb airtight.
269. **SEALER** A coating used to increase the compatibility of a prime coat for a top coat.
270. **SECONDARY ELECTRONS (SE)** Low-energy electrons produced from the interaction of beam electrons and conduction band electrons of atoms within the interaction volume. They are produced throughout the interaction volume, but only those near the surface have enough energy to escape. The secondary electron signal is typically used to form topographic images.
271. **SELVAGE** The narrow edge of woven fabric that runs parallel to the warp. It is made with stronger yarns in a tighter construction than the body of the fabric to prevent raveling.
272. **SENSITIVITY** The measure of an analytical response to a unit of the sample.
273. **SHORT-WAVE UV ILLUMINATION** In the wavelength range from 280 nm- 100 nm with the peak wavelength energy at 254 nm.
274. **SIGN OF ELONGATION** A reference to the orientation of the refractive indices in an anisotropic substance as it relates to the elongated direction of a substance. If the slow

wave (higher refractive index) is in the elongated direction it has a positive sign of elongation. If the fast wave is in the elongated direction it has a negative sign of elongation.

275. **SIGNIFICANT DIFFERENCE** A difference between two samples that indicates that the two samples do not have common origin.
276. **SMALL FILAMENT** The smaller diameter filament in a two filament bulb.
277. **SNELL'S LAW** For monochromatic light and for a given pair of substances *a* and *b* on opposite sides of a surface of separation, the ratio of the sine of the angle Φ_a (between the ray in substance *a* and the normal) and the sine of angle Φ_b (between the ray in substance *b* and the normal) is a constant, *i.e.*, $\sin \Phi_a / \sin \Phi_b$ = constant.
278. **SNIPPETS** Short segments of yarn created if a knit fabric is cut at an angle to the thread direction.
279. **SOLVENT** Organic liquids of various types having the function of dissolving another material (solid or liquid).
280. **SPECIMEN** Material submitted for examination. Samples are removed from a specimen for analysis.
281. **SPECTRAL ARTIFACTS** Spectral peaks other than characteristic peaks, produced during the energy dispersive X-ray spectroscopy detection process. Examples include escape peaks and sum peaks.
282. **SPECTRAL RESOLUTION** A measure of the ability to distinguish between adjacent peaks in an X-ray spectrum. It is usually determined by measuring peak width at half the maximum value of the peak height or full-width-half-maximum.
283. **SPECTROMETER** An instrument for the measuring of some function of spectral power, or other physical quantity, with respect to spectral position within a spectral range.
284. **SPECTROSCOPY** The study of the theory and interpretation of spectra generated by any phenomenon, such as electromagnetic waves or particles, ordered in accordance with the magnitude of a common physical property (wavelength, frequency, or mass).
285. **SPECTRUM** An arrangement of the component parts of any phenomenon, such as electromagnetic waves or particles, ordered in accordance with the magnitude of a common physical property.
286. **SPHERULITES** Spheres composed of needles or rods all oriented perpendicular to the outer surface or a plane section through such a sphere. A common form of polymer crystallization from melts or concentrated solutions.
287. **SPIRAL FRACTURE** Concentric fracture.
288. **SPONTANEOUS COMBUSTION** The result of a natural heat-producing process (bacteria, chemical oxidation) in poorly ventilated containers.
289. **STAIN** A solution of a dye or a suspension of a pigment in a vehicle designed to impart a color to a wood surface rather than to form a protective coating.
290. **STAPLE** Graded fibers or chopped filaments for rope and threads (rope and threads made from staple has a fibrous or fuzzy surface due to all the ends).
291. **STEREOMICROSCOPE** A microscope containing two separate optical systems, one for each eye, giving a stereoscopic view of a specimen.
292. **STRAND** Threads or yarns twisted together in the opposite direction to that of the yarn or thread itself; a major element in the complete rope or thread. Rope or thread made with strands--not braided--is "laid line". A single fiber, filament, or monofilament.
293. **STRAPPING TAPE** See filament tape.

294. **SUBGENERIC CLASS** A group of fibers within a generic class that shares the same polymer composition. Subgeneric names include, for example, nylon 6, nylon 6,6, and poly(ethylene terephthalate).
295. **SUM PEAK** A peak resulting from the simultaneous detection of two photons. This is manifested as a peak at the combined energy of line(s) for the specific element(s) involved.
296. **SUPPORT POLE** The part of a lamp in which the filament is attached to and allows for the flow of electricity through the bulb.
297. **SURFACE DYE** A colorant bound to the surface of a fiber.
298. **SYNTHETIC FIBER** A class of manufactured polymeric fibers that are synthesized from chemical compounds (e.g., nylon and polyester).
299. **SYSTEM DEAD TIME** The time during which the energy dispersive X-ray spectrometer is not able to process X-rays. Dead time is typically expressed as a percentage of real time.
300. **SYSTEM PEAKS** (stray radiation) Peaks that may occur in the X-ray spectrum resulting from interaction of the electron beam or fluorescent radiation with components of the scanning electron microscope itself.
301. **TACK** Property of an adhesive achieved by the addition of a low molecular weight organic component that allows the elastomer to form a bond immediately with a surface under low pressure.
302. **TACKIFIER** Material added to the adhesive base polymer to impart tack.
303. **TAKE-OFF ANGLE** Angle between the specimen surface and the detector axis.
304. **TANGENTIAL FRACTURE** Concentric fracture.
305. **TEAR** A separation of fabric determined by unevenness (random breakage) of fibers and threads at the damage margin.
306. **TECHNICAL FIBER** A bundle of natural fibers composed of individual elongated cells that can be physically or chemically separated and examined microscopically for identifying characteristics (e.g., hemp, jute, and sisal).
307. **TEXTURIZED YARN** A crimped feature in reinforcement fibers designed to give bulk.
308. **THERMOPLASTIC FIBER** A synthetic fiber that will soften or melt at high temperatures and harden again when cooled.
309. **THERMOPLASTIC POLYMER** A resin which polymerizes without the necessity of heat. If the resin is heated below its decomposition temperature, it softens and hardens again upon cooling; hence, the term "thermoplastic".
310. **THERMOSETTING POLYMER** A resin which can be made to form cross-linkages when baked.
311. **THICKNESS (TAPE)** Distance from one surface of either a tape, backing, or adhesive to the other, usually expressed in mils or thousandths of an inch.
312. **THICKNESS (FIBER)** The optical path through the fiber used for the calculation of birefringence, typically measured in micrometers.
313. **TRANSMITTANCE, (T)** ratio of the energy of the radiation transmitted by the sample to the background, usually expressed as a percentage.
314. **TWIST** The direction of twist in yarns is indicated by the capital letters S and Z. Yarn has an S-twist if when it is held vertically, the spirals around its central axis slope in the same direction as the middle portion of the letter S, and Z-twist if they slope in the same direction as the middle portion of the letter Z.
315. **ULTRAVIOLET (UV) LIGHT** The range of invisible electromagnetic radiation having wavelengths from about 4 nanometers (on the border of the x-ray region), to about 380 nanometers (just below violet in the visible spectrum). The short UV wavelengths lie in the

range 280 - 300 nanometers (UVB); the long UV wavelengths are in the range 320 - 400 nm (UVA).

316. **VARNISH** A homogeneous solution of drying oils and resins in organic solvents. The resins may be naturally occurring (for example, rosin or dammar), or synthetic (for example, products of phenol and formaldehyde).
317. **VEHICLE** The portion of a surface coating other than the pigment, the purpose of which is to enable the pigment to be distributed over the surface. The vehicle includes solvents, binders, and other additives. In general discussion, the term vehicle is frequently used to indicate the oil or resin which forms a continuous film and binds the pigment to the substrate.
318. **VISIBLE SPECTRUM** The part of the electromagnetic spectrum between 400 nm (blue) and 700 nm (red).
319. **WALLNER LINES** Ridges or rib marks.
320. **WARP YARNS** Yarns in scrim fabric of reinforced tape that run lengthwise (in the machine direction)
321. **WAVELENGTH** The distance, measured along the line of propagation, between two points that are in phase on adjacent waves.
322. **WAVENUMBER** The number of waves per unit length, in a vacuum, usually given in reciprocal centimeters cm^{-1}.
323. **WEAR AND TEAR** Damage caused to garments in the course of everyday wear, such as matting of yarns and pilling.
324. **WEAVE** Fabric Formed by interlacing two sets of yarns at right angles.
325. **WEFT YARNS** See fill yarns.
326. **X-RAY FLUORESCENCE** A specific wavelength of light (x-ray region) that can cause some materials to be fluorescent. The fluorescence can be used to evaluate a material.
327. **WOVEN FABRIC** Generally used to refer to fabric composed of two sets of yarns, warp, and weft (filling), that is formed by weaving, which is the interlacing of these sets of yarns.
328. **WRINKLING** A paint defect in which the film surface skins over and then absorbs liquid within the film. The absorption results in swelling of the surface skin with the formation of a wrinkled texture.
329. **YARN** For the purposes of this document, yarns refer to lengths of fiber reinforcement: twisted staple fibers or filament fibers.

APPENDIX 4 – Bruker FTIR Quality Control

Make and Models

FTIR – Bruker Tensor 27 (Serial Number: T27.0416.03)
FTIR Microscope – Bruker Hyperion 2000 (Serial Number: 263)
Bruker OPUS Software Version 6.5 Build 6, 5, 92 (20080204)

Quality Control Measures

Every day that the Bruker FTIR is used, a performance test shall be run to ensure that it is operating properly. The "validation instrument test program" shall be used. This test includes a signal to noise test, 100% line test, peak amplitude test, source test, X-axis test, and a Y-axis test. All shall register 'Ok" to pass. A hard copy of the results shall be kept and the date and forensic scientist initials shall be recorded in the instrument log.

Method

The software OPUS 6.5 is opened. Instrument status is opened and a PQ test is run. This test is a macro program specially designed by Bruker to run a QC test on the instrument.

APPENDIX 5 – SEM/EDS Quality Control

Make and Models

JEOL JSM-6360LV (Serial Number: MP18310021)
JEOL SEM Control User Interface Version 6.52A

Oxford EDS Inca Xsight 7582 (Serial Number: A3384-5350-TV3-2053)
Oxford Inca Suite Version 4.04

Quality Control Measures

Every day that the SEM/EDS is used, a performance test shall be run to ensure that it is operating properly. This test includes a daily elemental analysis of a copper tape. The $K_{\alpha\beta}$ and L_{α} lines shall be present and the K_{α} peak shall be 8.04 keV (±0.01 keV). The cursor shall be placed on the K_{α} peak and then the spectra printed. A copy of the spectra shall be initialed and the placed in the instrument log binder. The date of analysis and forensic scientist's initials shall be recorded in the instrument log.

Method

A piece of a copper tape (approximately a 1 cm x 1 cm square) is placed on a carbon stub. The copper tape is run at 20keV and a 10 mm working distance. A spectrum is produced, and the resulting peaks are identified and keV is verified.

APPENDIX 6 – GC/MS Quality Control

Make and Models

Agilent 6890N GC/5973 MSD (Serial Number: CN10426086/US41710361)
Agilent MSD Chemstation Software G1701 EA D.01.02.16

Agilent 6890 N GC/5975B MSD (Serial Number: CN10644032/US62724858)
Agilent MSD Chemstation Software G1701 EA D.03.00.611

Agilent 7890A GC/5975C MSD (Serial Number: CN10929004/US92022952)
Agilent MSD Chemstation Software G1701 EA E.02.00.493

Quality Control Measures

The ASTME1618 test mixture shall be run every month to ensure that the instrument is operating properly. The chromatogram shall have the major constituents at similar peak ratios to the previous run. A hard copy of the chromatogram shall be placed in the QC binder. The instrument shall be autotuned every week and a hard copy of the autotune results shall be placed in the QC binder with the results of the autotune. If an autotune cannot be achieved the instrument shall be taken out of service.

Additionally, each time the instrument is used for casework, a gasoline reference shall be run at the beginning of the run. If multiple samples are run there shall be a gasoline reference run at the beginning and end of the run. The chromatogram(s) shall have the major constituents at similar peak ratios to previous runs. A hard copy of the gasoline reference chromatogram(s) shall be kept with each case record. Blanks shall be run between samples.

Method

Agilent MSD ChemStation software is opened to run the autotune and tune evaluation. These tests are specifically designed by Agilent to do a performance check on the instrument.

APPENDIX 7 – Py-GC/MS Quality Control

Make and Models

Agilent 6890 N GC/5975B MSD with CDS 5150 Pyroprobe (Serial Number: CN10644032/US62724858)
Agilent MSD Chemstation Software G1701 EA D.03.00.611

Quality Control Measures

Quality Control Measures for Operation with the CDS 5150 Pyroprobe: For every case in which the pyrolysis GC/MS is used in the pyrolysis mode, a paint reference material shall be run to ensure that the instrument is working properly. The chromatogram shall have the major constituents at similar retention times and peak ratios to the previous run. A hard copy of the chromatogram shall be kept in the QC binder. Additionally, the instrument shall be autotuned each week and a hard copy of the autotune results shall be kept in the QC binder. Blanks shall be run between samples.

Quality Control Measures for Operation without the CDS 5150 Pyroprobe: See Appendix 6.

Method

Agilent MSD ChemStation software is opened to run the autotune and the tune evaluation. These tests are specifically designed by Agilent to do a performance check on the instrument.

APPENDIX 8 – XRF Quality Control

Make and Models

EDAX Eagle III Micro-XRF (Serial Number: 738363)
Vision 32 XRF Software for Eagle III u-probe (Software Version 5. 00978B)

Quality Control Measures

When the XRF is used for casework, the instrument shall be calibrated and a performance check shall be run to ensure that it is operating properly. The instrument shall be calibrated with a traceable standard material. The performance check shall include either an aluminum/copper reference material or a glass reference material. A hard copy of the performance check results shall be placed in the QC binder. The analyst's initials and date of calibration and performance check shall be recorded on the QC log.

Method

The instrument calibration shall follow the procedure that is outlined in the software. The standard used shall be 226 AD. A copy of the calibration will be put in the XRF QC binder.

When the XRF is used for glass casework, a performance check shall be run to ensure that the instrument is operating properly. The performance check shall be conducted with a glass reference material. The standard NIST SRM 1831, Soda-Lime sheet glass is the preferred glass reference material. The spectra shall have all of the elemental constituents at similar KeV and peak ratios to the previous run.

When the XRF is used for metal or unknown casework, a performance check shall be run to ensure that the instrument is operating properly. The performance check shall be conducted with a metal reference material. The aluminum/copper 2024 is the preferred reference material. The spectra shall have all of the elemental constituents at similar KeV and peak rations to previous runs.

APPENDIX 9 – GRIM Quality Control

Make and Models

Foster/Freeman GRIM III (Serial Number: 6121)
Foster/Freeman Glass for GRIM III Software Version 2.3.121

Quality Control Measures

Every day that GRIM is used a test shall be run to ensure that it is operating properly before the refractive indices (R.I.'s) are measured for each case. The data and results shall be recorded along with the date and analyst initials in the QC binder.

Method

The test shall be conducted using the NBS 45 glass reference material and a 589 nm bandwidth light filter (D Line). At least ten refractive indices shall be obtained to establish an acceptable range of data. The mean of the R.I.'s shall be between 1.51780-1.51814 to pass the quality control. The result (pass/fail) shall be recorded in the QC binder.

APPENDIX 10 – Microspectrophotometer Quality Control

Make and Models

Craic QDI 1000 (Serial Number: 1000B-040807 UB)
Thermo Galatic GRAMS/AI (Software Version 7,02)

Quality Control Measures

Every day the microspectrophotometer is used, a test shall be run to ensure that it is operating properly. This test includes a wavelength calibration test and a photometric test. Both tests shall register "passed". A hard copy of the results shall be kept for record and date of analysis and analyst's initials shall be recorded in the QC binder.

Method

For the wavelength calibration test, a Holmium Oxide filter is placed on top of the polarizer and a wavelength measurement is taken by the instrument. This is followed by a Didymium filter. The instrument compares measured wavelength values to target wavelength values and calculates the difference. If the difference is within the maximum allowed difference, then the test passes.

For the photometric test, a ND0.1 filter is placed on top of the polarizer and an absorbance measurement is taken by the instrument. This is followed by a ND0.5 filter and a ND1.0 filter. The instrument compares measured absorbance values to target absorbance values and calculates the difference. If the difference is within the maximum allowed difference, then the test passes.

The wavelength and photometric calibration pass/fail tests were specifically designed by Craic to run a QC check on the instrument.

APPENDIX 11 – Melting Point Microscope Quality Control

Make and Models

Polarized Light Microscope - Olympus BX51TF (Serial Number: 7M23928)
Hotstage – Linkham LTS 350 (Serial Number: 0804-11005-0255)
Temperature Programmer – Linkham TMS94 (Serial Number: 0804-001-2041

Quality Control Measures

A test shall be run every day that the hot stage is used to ensure that it is operating properly. This consists of a melting point test of Nylon (NBS/CTS Fiber Sample 85A0306). The melting point shall be recorded with a date of analysis, analyst's initials and a note of any variation in instrument performance in the QC binder.

Method

A portion of a nylon fiber from NBS/CTS Fiber Sample 85A0306 is placed on a glass slide and covered with a cover slip. The hot stage is opened and the slide is placed in the slide holder. The hot stage is closed and the PLM is focused on the fiber. The temperature programmer is set to the maximum temperature desired and the rate at which the temperature ramps up. A melting point range is determined, and this range shall be between 266 – 275 °C.

APPENDIX 12 – Densitometer Quality Control

Make and Models

Mettler Paar DMA40 (Serial Number: 40149 A)

Quality Control Measures

When the densitometer is used, a test shall be run to ensure that it is operating properly. The test shall be conducted with a solvent with a known density. The standard shall be comparable to the established value. The data/results shall be recorded along with the date, time, and analyst initials in the QC binder.

Method

A known solvent shall be injected into the Mettler Paar DMA 40 Densitometer to test the instrument. Based on ambient conditions, the density of the known liquid and a questioned liquid can then be calculated. The density value shall be comparable to established literature value.

APPENDIX 13 - Caliper Quality Control

Make and Models

Fowler & NSK Max-Cal Electronic Digital Calipers (Serial Number: 272091)
Mitutoyo Gauge Block Set (Serial Number: 9909592)

Quality Control Measures

A test shall be run the first day that the calipers are used in each case to ensure that it is operating properly and is valid for 30 days. The measurements for each gauge block shall be recorded with a date of analysis, analyst's initials and a note of any variation in instrument performance in the QC binder.

Method

The NIST traceable gauge blocks (.0625in, .100in, .125in, .200in, .250in, .300in, .500in, 1.000in, and 2.000in) shall be measured. The actual measurement shall be within +/- 0.001in of the standards to pass the calibration test.

APPENDIX 14 – PLM Quality Control

Make and Models

Comparison PLM – Olympus BX51TF (Serial Numbers 4K11242, 5F13598)
PLM – Olympus BX50F4 (Serial Number 8A04730)
PLM – Olympus BX60F3 (Serial Number 6D00681)

Quality Control Measures

Every year the microscopes are in service, a performance test measurement shall be performed to ensure that the lens optics are functioning properly and that proper magnification factors are established for the appropriate objective lenses. This test consists of a yearly measurement with a microscope micrometer. A copy of the results shall be maintained with a date of measurement and forensic scientist's initials in the microscope QC log binder.

Method

Arrange stage micrometer and ocular scale in a parallel orientation. Determine the number of ocular scale divisions (OSD) equal to an even number of stage micrometer divisions (SMD). This ratio of SMD/OSD shall provide a magnification factor for the Ocular Scale paired with that particular objective.

Example

Stage Micrometer: "Objective Micrometer" 2mm = 200 x 10µm (0.01mm) divisions
Ocular Scale: Olympus WH10X/22 (100 divisions)

Example: Using a 4x objective, 130 stage micrometer divisions (SMD) equaled 52 ocular scale divisions (OSD). 130 SMD x 10µm per division = 1300µm. The magnification ratio shall be 1300µm/52 or 25µm per ocular scale division.

APPENDIX 15 – Ohm Meter Quality Control

Make and Models

Micronta FET Analog (Identification - ISP M 3155)

Quality Control Measures

A test shall be run every day that the Ohm meter is used to ensure that it is operating properly.

Method

The Ohm meter shall be turned on and the dial switched to Ohms. The electrode probes are to be touched together to check the continuity to see it the meter is reacting properly. This shall be documented in each case record that the Ohm meter is utilized.

APPENDIX 16 – PDQ Quality Control

Make and Models

PDQ Software

Quality Control Measures

Every PDQ update shall be performance checked upon receipt and prior to use in casework.

Method

The software check shall be the one that is provided to the Indiana State Police by the Royal Canadian Mounted Police (RCMP). The performance check shall be documented in the PDQ QC log book along with the date and analyst's initials. The procedure and expected results shall follow the directions of the software check as received from the RCMP.

APPENDIX 17 – Bruker FTIR Preventive Maintenance

Make and Models

FTIR – Bruker Tensor 27 (Serial Number: T27.0416.03)
FTIR Microscope – Bruker Hyperion 2000 (Serial Number: 263)
Bruker OPUS Software Version 6.5 Build 6, 5, 92 (20080204)

Preventive Maintenance Measures

The FTIR shall be serviced as needed. When the FTIR cannot pass the QC as prescribed in Appendix 4 it shall be serviced by a qualified technician. The service shall be documented in the maintenance log for the instrument. Before being placed back into service, the instrument shall pass the QC in Appendix 4.

The desiccant in the instrument shall be dried as necessary. The need for drying is indicated by a red light on the instrument.

Method

The desiccant shall be removed from the instrument and placed in a beaker in a heated oven to dry out the desiccant.

APPENDIX 18 – SEM/EDS Preventive Maintenance

Make and Models

JEOL JSM-6360LV (Serial Number: MP18310021)
JEOL SEM Control User Interface Version 6.52A

Oxford EDS Inca Xsight 7582 (Serial Number: A3384-5350-TV3-2053)
Oxford Inca Suite Version 4.04

Preventive Maintenance Measures

The SEM shall be serviced as needed. When the SEM cannot pass the QC as prescribed in Appendix 5 it shall be serviced by a qualified technician. The service shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 5.

The source filament shall be replaced when it burns out. This shall be recorded in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 5.

The water in the chiller shall be replaced as needed. This shall be recorded in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 5.

Method

The filament and water change shall follow the procedures as outlined in the instrument manual.

APPENDIX 19 – GC/MS Preventive Maintenance

Make and Models

GC/5973 MSD (Serial Number: CN10426086/US41710361)
Agilent MSD Chemstation Software G1701 EA D.01.02.16

Agilent 7890A GC/5975C MSD (Serial Number: CN10929004/US92022952)
Agilent MSD Chemstation Software G1701 EA E.02.00.493

Preventive Maintenance Measures

The GC/MS shall be serviced as needed. When the GC/MS cannot pass the QC as prescribed in Appendix 6 it shall be serviced by a qualified technician. The service shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 6.

The instrument shall be cleaned as needed. This shall be recorded in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 6.

The filament, column, and seals shall be replaced as needed. This shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 6.

The column insert, auto-sampler needle and septum shall be replaced as needed. This shall be documented in the maintenance log for the instrument.

Method

The preventive maintenance conducted shall follow the procedures as specified in the instrument manual.

APPENDIX 20 – Py-GC/MS Preventive Maintenance

Make and Models

Agilent 6890 N GC/5975B MSD with CDS 5000 Pyroprobe (Serial Number: CN10644032/US62724858)
Agilent MSD Chemstation Software G1701 EA D.03.00.611

Preventive Maintenance Measures

The GC/MS shall be serviced as needed. When the GC/MS cannot pass the QC as prescribed in Appendix 7 it shall be serviced by a qualified technician. The service shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 7.

The instrument shall be cleaned as needed. This shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 7.

The filament, column, and seals shall be replaced as needed. This shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 7.

The column insert, needle, quartz tube, and septum shall be replaced as needed. This shall be documented in the maintenance log for the instrument.

Method

The preventive maintenance conducted shall follow the procedures as specified in the instrument manual.

APPENDIX 21 – XRF
Preventive Maintenance

Make and Models

EDAX Eagle III Micro-XRF (Serial Number: 738363)
Vision 32 XRF Software for Eagle III u-probe (Software Version 5. 00978B)

Preventive Maintenance Measures

The XRF shall be serviced as needed. When the XRF cannot pass the QC as prescribed in Appendix 8 it shall be serviced by a qualified technician. The service shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 8.

APPENDIX 22 – GRIM
Preventive Maintenance

Make and Models

Foster/Freeman GRIM III (Serial Number: 6121)
Foster/Freeman Glass for GRIM III Software Version 2.3.121

Preventive Maintenance Measures

The GRIM shall be serviced as needed. When the GRIM cannot pass the QC as prescribed in Appendix 9 it shall be serviced. The service may include recalibration of the instrument. The service shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 9.

The microscope and hot stage shall be aligned and cleaned as needed.

APPENDIX 23 – Microspectrophotometer Preventive Maintenance

Make and Models

Craic QDI 1000 (Serial Number: 1000B-040807 UB)
Thermo Galatic GRAMS/AI (Software Version 7,02)

Preventive Maintenance Measures

The microspectrophotometer shall be serviced as needed. When the microspectrophotometer cannot pass the QC as prescribed in Appendix 10 it shall be serviced by a qualified technician. The service shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 10.

The filters shall be cleaned as needed with appropriate solvents as specified in the instrument manual.

APPENDIX 24 – Melting Point Microscope Preventive Maintenance

Make and Models

Polarized Light Microscope - Olympus BX51TF (Serial Number: 7M23928)
Hotstage – Linkham LTS 350 (Serial Number: 0804-11005-0255)
Temperature Programmer – Linkham TMS94 (Serial Number: 0804-001-2041

Preventive Maintenance Measures

The microscope and hot stage shall be serviced as needed. When the instrument cannot pass the QC as prescribed in Appendix 11 it shall be serviced by a qualified technician. The service shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 11.

The microscope and hot stage shall be aligned and cleaned as needed..

APPENDIX 25 – Densitometer Preventive Maintenance

Make and Models

Mettler Paar DMA40 (Serial Number: 40149 A)

Preventive Maintenance Measures

The densitometer shall be serviced as needed. When the densitometer cannot pass the QC as prescribed in Appendix 12 it shall be serviced by a qualified technician. The service shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 12.

APPENDIX 26 – Caliper Preventive Maintenance

Make and Models

Fowler & NSK Max-Cal Electronic Digital Caliper (Serial Number: 272091)
Mitutoyo Gauge Block Set (Serial Number: 9909592)

Preventive Maintenance Measures

The caliper does not normally need to be serviced. When the caliper cannot pass the QC as prescribed in Appendix 13 it shall be evaluated to determine if it is repairable. There is a battery to change as needed. If it is serviced the repair shall be documented in the maintenance log for the instrument. Before being placed back into service the instrument shall pass the QC in Appendix 13.

APPENDIX 27 – Microscope Preventive Maintenance

Make and Models

Item	Description	Manufacturer	Model #	S.N.
Boom Stereomicroscope	Microscope	Leica	Leica MZ6	
Boom Stereomicroscope	Microscope (.7-3x)	Leica	StereoZoomSZ-4	
Stereomicroscope	Stereomicroscope	Olympus	SZX7	6C12321
Stereomicroscope	Stereomicroscope	Olympus	SZX-7	6C12136
Stereomicroscope	Stereomicroscope	Olympus	SZX-12	4H18127
Stereomicroscope	Stereomicroscope	Olympus	SZX-12	4M12732
Stereomicroscope	Stereomicroscope	Olympus	SZX-12	5K07651
Stereomicroscope	Stereomicroscope	Olympus	SZX-12	4H05319
Stereomicroscope	Stereomicroscope	Olympus	SZX-12	5E1590
Stereomicroscope	Stereomicroscope	Olympus	SZX-12	5L16209
Polarized Light Microscope	Microscope	Olympus	BX50F4	8A04730
Polarized Light Microscope	Microscope	Olympus	BX60F3	6D00681
Comparison Microscope	Right Microscope	Leitz	Laborlux 512794	58447
Comparison Microscope	Left Microscope	Leitz	Laborlux 512794	58729
Comparison PL Microscope	Right Comparison PLM	Olympus	BX51TF	4K11242
Comparison PL Microscope	Left Comparison PLM	Olympus	BX51TF	5F13598

Preventive Maintenance Measures

The microscopes shall be aligned and cleaned on an annual basis.

If a microscope needs service it shall be serviced by a qualified technician. The service shall be documented in the maintenance log. Before being placed back into service it shall be aligned and cleaned.

APPENDIX 28 – Ohm Meter Preventive Maintenance

Make and Models

Micronta FET Analog (Identification - ISP M 3155)

Preventive Maintenance Measures

The Ohm meter shall be serviced as needed. When the Ohm meter cannot pass the QC as prescribed in Appendix 15 it shall be evaluated to determine if it is repairable. Before being placed back into service the instrument shall pass the QC in Appendix 15.

The batteries of the Ohm meter shall be changed when the battery check fails.

APPENDIX 29 – Microanalysis Unit Reference Standards

Reference Standards

The Microanalysis Unit currently has the following reference standards:

1. 226 AD traceable standard (used for the calibration of the XRF)

2. GRIM Calibration glasses (B1-B12)

3. Caliper metal blocks

APPENDIX 30 – Microanalysis Unit Reference Materials

Reference Materials

The current reference materials include tape, safe insulation, ignitable liquids, fibers, and lamps. Different reference materials are also used in the performance tests on the instruments (PFTBA (Perfluorotributylamine), ASTM E1618 Test Mix, Microspectrophotometer filters, Cargille oils, NIST 1826, and chemicals).

Identifier

Each reference material shall be uniquely cataloged.

Use of Reference Materials

The Microanalysis Unit retains reference materials for use as training materials and for reference of different types of evidence. Some reference materials are used in the performance checks on the instruments. The reference material shall not be used for identification.

Storage

Reference materials shall be stored in a location where they do not affect evidence samples. e.g. ignitable liquid residues shall not be stored in a location where physical evidence from cases are stored. Reference materials shall be stored in a restricted access area within the Microanalysis Unit.

Handling

The reference material shall be handled in a manner which does not cross contaminate other reference material or the evidence samples. The reference material shall not come in contact with the actual evidence samples.

Transportation

The reference materials are not transported out of the Indianapolis Regional Laboratory.

APPENDIX 31 – Microanalysis Unit Training Materials

Training Materials

Training materials are items that often have a limited lifetime in the Laboratory and/or are used to demonstrate or study a particular phenomenon.

Identifier

Each material does not need to be uniquely cataloged.

Use of Training Materials

The Microanalysis Unit has training materials to assist in the training of new analysts and to use as tour demonstration pieces. In some instances, the training material could be used in conjunction with casework. (e.g. fabric to make test cuts from a knife in a cut/tear analysis, or background material for pyrolysis in a ignitable liquid residue case). If training samples are used in conjunction with casework, the testing shall be documented in the case notes. The actual materials tested do not need to be saved and returned with the evidence.

Storage

Training materials shall be stored in a location where they do not affect evidence samples. e.g. ignitable liquid residues shall not be stored in a location where physical evidence from cases are stored. Training materials are not required to be in a restricted area within the Microanalysis Unit.

Handling

The training material shall be handled in a manner which does not cross contaminate reference material or the evidence samples.

Transportation

The training materials can be transported out of the Indianapolis Regional Laboratory.

APPENDIX 32 – Thermo Nicolet FTIR Quality Control

Make and Models

FTIR – Thermo Nicolet iZ10 (Serial Number: ALM 1200149)
FTIR Microscope – Thermo Nicolet iN10 (Serial Number: ALI 1200710)
OMNIC 9 Software (Version 9.1)

Quality Control Measures

Every day that the Thermo FTIR is used for casework, a test will be run to ensure that it is operating properly. The test will be the instrument validation test program that is on the instrument. This test for the Bench/Transmission analysis with OMNIC includes six components that are Energy ratio, Noise level, Wavenumber accuracy, Optical resolution, Repeatability, and Detector linearity. This test for microscope analysis with PICTA includes 10 components that are Transmission Energy Ratio, Transmission Noise level, Transmission Wavenumber accuracy, Transmission Optical Resolution, Transmission Repeatability, Reflection Energy ratio, Reflection Noise ratio, Reflection Wavenumber accuracy, Reflection Optical resolution, and Reflection repeatability. The date of analysis and forensic scientist's initials shall be recorded in the instrument log.

Method

The Omnic software is opened. Instrument status is opened and a performance test is run. This test is a macro program specially designed by to run a QC test on the instrument.

APPENDIX 33 – Thermo Nicolet FTIR Preventive Maintenance

Make and Models

FTIR – Thermo Nicolet iZ10 (Serial Number: ALM 1200149)
FTIR Microscope – Thermo Nicolet iN10 (Serial Number: ALI 1200710)
OMNIC 9 Software (Version 9.1)

Preventive Maintenance Measures

The FTIR shall be serviced as needed. When the FTIR cannot pass the QC as prescribed in Appendix 32 it shall be serviced by a qualified technician. The service shall be documented in the maintenance log for the instrument. Before being placed back into service, the instrument shall pass the QC in Appendix 32.

The desiccant in the instrument shall be dried as necessary. The need for drying is indicated by a red light on the instrument.

Method

The desiccant shall be removed from the instrument and placed in a beaker in a heated oven to dry out the desiccant.

Appendix 34 - MICROANALYSIS UNIT WASTE DISPOSAL PROCEDURES

Non-hazardous Chemical Waste

Not all waste materials and chemicals in a laboratory are hazardous waste. For Indiana State Police Laboratory Division, these include paper and plastic trash, empty containers, broken glass, non-hazardous liquid and solid wastes, and color/spot testing waste.

Chemicals from color/spot testing and clean-up of a spot plate may be disposed of through flushing into a sink drain connected to a sanitary sewer with at least twenty (20) volumes of water for each volume of waste.

GC/MS vials containing small amounts of solvent are routinely returned with evidence to the contributor, and are not handled as waste.

Empty glass containers and broken glass are collected in “broken glass” boxes. Empty containers as defined by EPA and IDEM are described in the Laboratory Waste Management Program. After the “broken glass” boxes are full, they shall be sealed for final disposal in “normal” trash.

Empty aerosol cans may be disposed of in “normal” trash. To be considered empty, aerosol cans shall contain <u>no propellant and no product</u>, and shall be at atmospheric pressure.

Non-hazardous liquid and solid wastes may be processed for disposal down a sink drain or in “normal” trash as outlined in the Laboratory Waste Management Program. A list of non-hazardous chemicals suitable for drain or trash disposal is included as an appendix in the Laboratory Waste Management Program. You may dispose these types of solid chemicals in normal trash if the containers are tightly capped and of good integrity.

If you are unsure whether or not you should dispose of a material as a non-hazardous waste, then it should be handled as a hazardous chemical for waste disposal.

Acid and Base Disposal

Acidic and alkaline (basic) chemical wastes are classified as hazardous waste if the pH is less than or equal to 2 or greater than or equal to 12.5. If the acid or alkaline waste ONLY has characteristics of corrosivity and is not a listed waste, it may be neutralized to within a pH range of 5 to 9 before disposal to a sanitary sewer. Neutralization can be incorporated in the analysis procedure.

Neutralized acid and alkaline waste shall be flushed with at least twenty (20) volumes of water for each volume of waste.

Acid and Base Neutralization Procedures

These procedures explain the disposal of concentrated solutions of acids, such as hydrochloric, nitric and sulfuric acid, and bases such as ammonium hydroxide and sodium hydroxide.

Caution: vapors and heat are generated during neutralization.

You are not required to neutralize any wastes yourself. If you choose to neutralize and dispose of these materials as non-hazardous wastes, the following steps should be used:

- Perform all steps slowly.

- Keep containers cool while neutralizing.

- **Acid neutralization:** While stirring, add acids to large amounts of a cold solution of aqueous base (examples: sodium carbonate, calcium hydroxide, 0.45 N sodium hydroxide or 8 M sodium hydroxide).

- **Base neutralization:** First add the base to a large vessel containing cold water. Slowly add acid (examples: 1 M solution of Hydrochloric Acid or 0.5 N Sulfuric Acid).

- Neutralize concentrated acid and base solutions to within a pH range of 5 to 9, and then flush them into the sanitary sewer with at least 20 volumes of water for each volume of waste.

- **If necessary, allow the contents to react for at least twenty-four hours to obtain a stable pH and to dissipate the any heat associated with the neutralization reaction. The container should not be hot and the contents should not be smoking.**

Hazardous Chemical Waste

Laboratory personnel are not responsible for final classification of waste chemicals for hazardous waste manifests, yet shall be generally aware of waste classification criteria to determine if a chemical is hazardous or non-hazardous for disposal. The classifications for hazardous wastes are: F-list, K-list, U-list, P-list, and characteristic wastes. Information is included in the Laboratory Waste Management Program to classify potential hazardous chemical wastes.

F-list waste – These are non-specified source waste. This includes all spent solvent mixture/blends containing, before use, a total of 10% or more (by volume) of one or more of the solvents listed in F001, F002, F003, F004, and F005

K-list waste - **this list does not apply to Indiana State Police Laboratories**. It includes certain waste from specific industries, such as petroleum refining or pesticide manufacturing.

U- and P-list waste - (discarded and unused commercial chemical products) U and P list waste include specific commercial chemical products in an unused or "virgin" form.

Virgin chemicals – a chemical that has not been previously used or consumed, or subjected to processing other than for its original production.

P-list chemicals are classified as acutely hazardous waste, and are subject to a 1 kg limit for accumulation quantity.

Characteristic hazardous waste groups are classified by characteristics of ignitability, corrosivity, reactivity, and toxicity.

Expired or Unused Chemicals

Hazardous chemicals that are past their expiration date for analytical use or are no longer needed for analysis are to be slated for hazardous waste disposal. These containers do not need to be emptied or contents transferred to another container. The original container shall be marked as "hazardous waste." Any dates (i.e. received date, expiration date) shall be covered up or obliterated. A new date shall be marked on the container, which is the date the container is placed in storage for hazardous waste disposal.

Microanalysis Unit procedures for collection, storage and disposal of hazardous waste

The proper way to collect and store hazardous waste is through use of hazardous waste containers in a Satellite Hazardous Waste Accumulation area until full. Transfer the full container to a Central Hazardous Waste Accumulation area for disposal by a contracted chemical waste disposal vendor.

Examples of satellite hazardous waste collection containers for use in the Microanalysis Unit are:

1. Halogenated waste including bromoform, chloroform, bromoform mixtures, etc.

2. Flammable wastes including pentane, methanol, acetone, pentane, hexane, petroleum ether, toluene, flammable organic chemical mixtures, etc.

3. Oxidizers including potassium dichromate and potassium permanganate

Organic chemicals used as a rinse in cleaning glassware shall be collected as either chlorinated wastes or flammable waste.

> Examples:
> Chloroform rinse of glassware shall be collected as chlorinated hazardous wastes.
> Methanol or Acetone rinse of glassware shall be collected as flammable hazardous wastes.

Solvents used in extractions that are not evaporated to recover evidence materials for further analysis shall be processed as hazardous waste.

GC/MS vials containing hazardous chemical solvent that are not returned as evidence shall be collected as hazardous waste.

Chemicals in color tests if conducted in a test tube shall be processed as hazardous waste.

Full or partially empty aerosol cans shall be collected for disposal as hazardous waste in a satellite container labeled as "Aerosol Cans” for hazardous waste disposal. IDEM and EPA regulate all partially empty spray cans as hazardous waste, because they may still contain chlorinated solvents, flammable material or toxic substances. **Do Not** discard partially empty spray cans in the trash. **Do Not** puncture any aerosol cans.

Dilution of hazardous chemical wastes and disposal in the sink drain is **<u>not</u>** the proper way to dispose of hazardous waste.

If a spill occurs, the chemical in the spill and the materials used to clean up the spill are considered to have the same hazard classification. Spill clean-up materials are not to be thrown in the "normal" trash. These materials are to be properly disposed of as hazardous waste. Procedures in the Laboratory Chemical Spill Management Program shall be used for spill clean-up and disposal.

Hazardous Waste Containers

1. Each laboratory and/or Unit shall supply their own containers
2. For liquid wastes, the amber 4 liter solvent bottles are preferred because they are non-recyclable and are compatible with most types of waste.
3. All containers shall be in good condition and compatible with their waste contents. The original container the chemical came in is usually the best container for chemical waste.
4. All containers shall have securely fitting lids or caps.
5. Funnels shall be removed and not left in waste containers.
6. Hazardous waste container shall be marked "hazardous waste."
7. A log of chemical waste contents, quantities, dates wastes were added and initials of waste generator shall be listed on the label or an attached tag.
8. Containers shall be stored with a closed lid or cap.

Liquid Hazardous Waste Containers

1. Leave 10% headspace (volume left at top of container) in case of expansion due to temperature.
2. Do not pour hot liquids into hazardous waste bottles.
3. Do not combine or comingle incompatible wastes (i.e. acids and bases)
4. Provide secondary containment.
5. Any container with a capacity of less than or equal to 4 liters shall have secondary containment.

Solid Hazardous Waste Containers

1. The original container is generally the "best" waste container for solid hazardous waste.
2. If original containers are not available – double bag the material and place in a sturdy cardboard box for support.
3. Do not use Biohazard bags.

4. Bags used should be trash bags.

Satellite Hazardous Waste Accumulation Storage

1. Hazardous waste regulations require that the generator accumulate hazardous chemical waste in containers at or near the point of generation where waste initially accumulates until full and which is under the control of the operator who generated the waste.
2. Under no circumstances shall waste be stored down the hall and/or out of your control.
3. Provide secondary containment, as necessary to contain spills.

Central Hazardous Waste Accumulation Storage

1. The Central Hazardous Waste Accumulation Storage area for Indianapolis Laboratory is located in the dock area and accessed by contacting the Dock Manager.
2. Full hazardous waste containers shall be moved to the Central Hazardous Waste Accumulation Storage area within three days after being filled to capacity.
3. Full hazardous waste containers shall be labeled with appropriate label and marked with the accumulation date. This is the date the satellite waste container was completely filled with the hazardous waste and transferred to the Central Hazardous Waste Storage area. This date is not the date the collection of hazardous waste began in the satellite container.
4. All waste containers shall have securely fitting lids or caps.
5. Provide secondary containment, as necessary to contain spills during transport to the storage area.

Secondary Containment to Minimize Spills of Hazardous Wastes

1. Secondary containment should be used to minimize the potential for breakage, spillage and the comingling of incompatible materials (i.e. acids and bases).

2. Plastic trays, pans, or tubs may be used.

3. Without exception, secondary containment is required for the following:
   A. All glass containers of liquid hazardous waste stored on the floor.
   B. All containers with capacity less than or equal to 4 liters of liquid hazardous waste, regardless of storage location.

4. Hazardous materials containers should be segregated by hazard class and stored in separate cabinets, trays or pans.

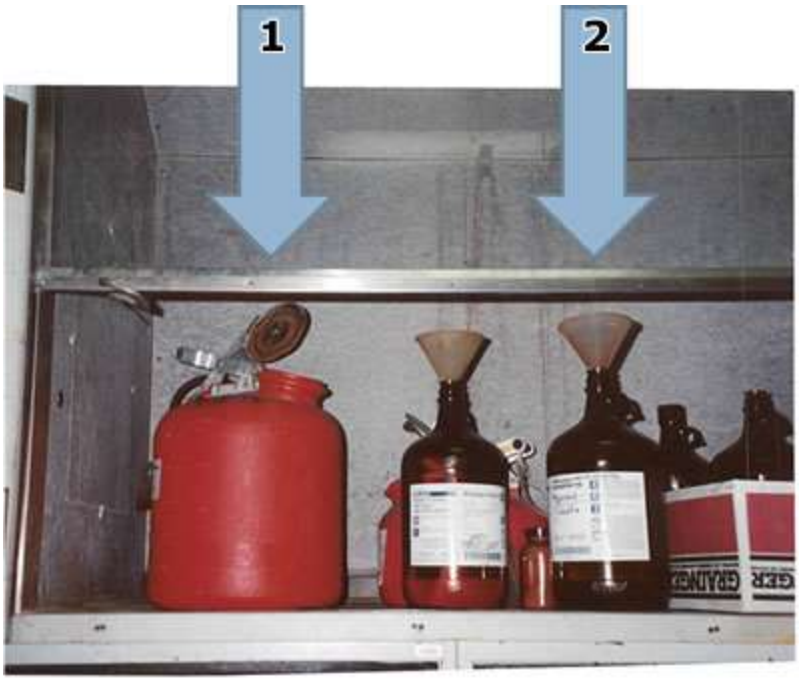
Example – Leaks with Spill Contained in Tray

Lids or Caps on Hazardous Waste Containers

1. Lids shall be securely in-place except when material is being removed or added to the container.
2. A funnel resting on the mouth of a bottle does not constitute a lid
3. Lids on waste containers shall be on tight (Note: Be sure that gas producing reactions have worked to completion before transferring the material to a hazardous waste container).
4. A closed container, when tipped over, won't leak!

Example - Improper Lids [Open Containers]

1. Lid open when not in use. **2. A funnel is <u>not</u> a lid.**



Labels on Hazardous Waste Containers

1. If a chemical container is reused, the original label shall be defaced, removed or completely covered.

2. EPA and IDEM regulations require that the name of waste chemicals be clearly identified on the label or attached tag.

3. Chemical formulas and abbreviations such as H_2SO_4, HCl, NaOH, HOAc, and MeOH are **not** accepted by EPA and IDEM. Use the chemical name such as sulfuric acid, hydrochloric acid, sodium hydroxide, acetic acid and methanol.

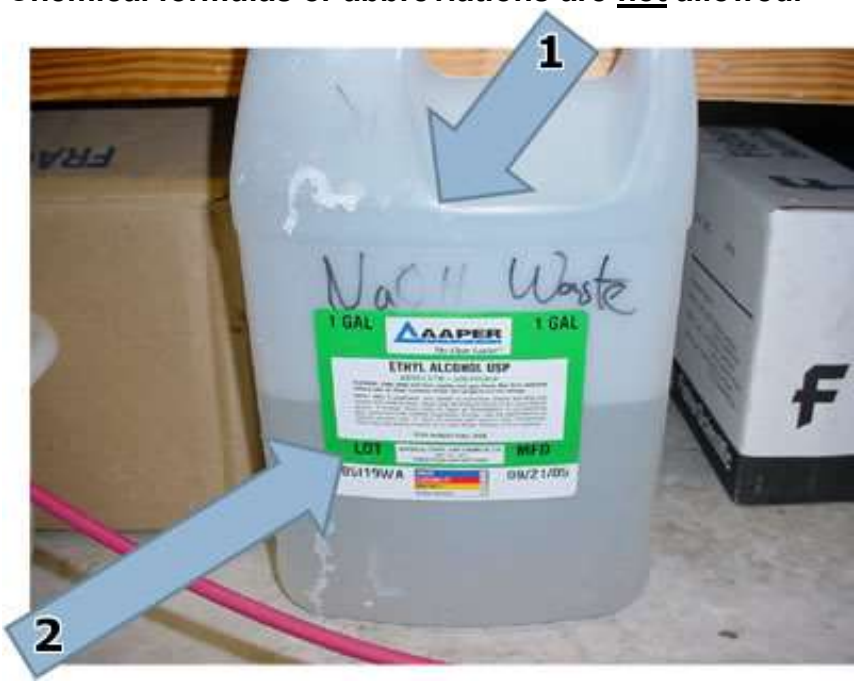
4. Hazardous waste regulations require the words "Hazardous Waste", or words which clearly identify the contents such as "Acetone Waste", be on each waste container.

5. The satellite container label shall have an area where the accumulation date (the date that the container is full – **not** the date that collection began in the container) can be documented.

Example – Proper Label

Example - Improper Label

1. **Chemical formulas or abbreviations are <u>not</u> allowed.**



2. **If you re-use a container for collecting hazardous waste, you shall deface, cover, or remove the original label.**