

## **General Approach for Paint and Coating Casework**

### **1 Introduction**

The most common types of coating evidence analyzed in crime laboratories are automotive and architectural (e.g., house, do-it-yourself (DIY)) paints. Cosmetic nail polish, tool, industrial, and non-automotive vehicle paints are also encountered.

Forensic paint examinations involve either (1) determining if a material is a coating/paint, (2) comparing two (or more) coating samples to determine if they can be differentiated, or (3) developing a probable source of an original equipment manufacturer (OEM) automotive coating.

### **2 Scope**

This procedure describes general guidelines Chemistry Unit caseworking personnel who analyze paint and coating evidence submitted to the FBI Laboratory. Separate, detailed standard operating procedures exist that cover sample processing and acquisition of both physical and chemical compositional data on paint and coating evidence.

### **3 Equipment/Materials/Reagents**

Not applicable.

### **4 Standards and Controls**

Not applicable.

### **5 Collection and Preservation Considerations**

The proper selection, collection, preservation, and packaging of paint evidence are of paramount importance in a forensic examination. The potential for a physical match between the broken edges of a known and questioned sample should be considered before selecting a collection method. Care must be taken to keep these edges intact.

Sources of questioned paint specimens include manageable objects such as clothing, tools, or bicycles, and less manageable objects such as motor vehicles, walls, sidewalks, roadways, etc. Whenever possible, items with potential paint transfer or smeared paint transfer should be submitted for examination in their entirety. Items of clothing should be packaged separately in paper bags. When paint evidence is suspected on an item such as a person, floor, or roadway, every effort should be made to manually remove it from that object. If the investigation involves a motor vehicle, the recommended practice is to place brown kraft paper under the area where paint transfer is suspected and then section, scrape, or cut the area of interest from the vehicle.

This paper should be saved and submitted along with the collected paint sample.

In order to account for any possible variation in layer structure across a painted surface, “control” (or known) samples should be taken from an area close to (but not within) any damaged area. If no damage is obvious, controls should be taken from several areas of the suspected substrate. In the case of motor vehicles, various layer systems and paint chemistries can be present on a single vehicle depending on factors such as different substrates (sheet metal or composite materials), horizontal versus vertical surfaces, stone chip susceptibility, and assembly plant spot repair. Aftermarket refinishes/repairs and weathering can also influence the paint system. “Control” specimens should comprise all of the layers of paint down to the parent substrate. This can be accomplished by a number of methods: sectioning an area of the painted surface, cutting a paint sample from the parent substrate using a clean blade or knife, lifting or prying loosely attached chips, or dislodging by gently impacting the opposite side of the painted surface.

Paint chips should be confined between two glass microscope slides, contained in pharmacy folded paper, or packaged in covered containers (e.g., pillboxes, glass or plastic vials). Plastic bags, cotton, and envelopes should not be used as the initial packaging for paint specimens. Other items, such as tools or sections of automotive parts, should be safely packaged (i.e., to minimize injury or compromising of the packaging due to sharp edges) separately and precautions taken to minimize the potential for dislodging the suspected paint transfer during transport to the FBI Laboratory.

## **6 Consideration for Other Forensic Examinations**

Process clothing for trace evidence and/or paint transfer prior to any fabric impression or DNA examinations. Regardless of which unit receives the evidence first, Paints and Polymers (P&P) personnel will then visually and/or microscopically examine the items and any associated debris for paint evidence.

If latent fingerprint and/or toolmark examinations are requested on an item (e.g., a tool), the item should be examined for paint evidence first. The paint can be removed from the item with tweezers or a relatively soft, pliable material such as wood or Teflon™. Metal probes and blades must not be used on the working end of a tool as they can alter the surface, thereby interfering with subsequent toolmark examinations. Ensure appropriate laboratory precautions are observed when working with latent print evidence.

## **7 Procedures**

Conduct a critical review of the contributor’s request and the item(s) received. As applicable, recommend additional examinations that could be probative and determine the logical sequence for the requested forensic examinations.

In a paint characterization, analyze the material in question using some or all of the methods outlined in Figure 1.

In a paint comparison examination, establish if any differences are detected between two (or more) samples after subjecting them to the same rigorous analytical testing. Figure 1 shows a flowchart that outlines the scheme for a paint comparison examination. A P&P Standard Operating Procedure (SOP) describes each analytical technique depicted in Figure 1 in detail.

If the paint sample is a factory-applied, original equipment manufacturer's (OEM) automotive finish, several reference collections and databases exist to aid in developing possible make, model, and model year information about the source vehicle. Figure 2 is a flowchart that outlines the analytical scheme for an automotive make-model-year search. A P&P SOP describes each analytical technique depicted in Figure 2 in detail.

## **8 Sample Selection**

Due to the wide variety of examination requests, numbers of samples submitted, and conditions of the samples submitted, P&P examiner discretion will determine the appropriate sample(s) to examine on a case-by-case basis. For indistinguishable samples, as determined by a discretionary number of analytical examinations, an option is to take an individual sample, assign a new item identifier (e.g., Item1-1), specifically list the item in the item inventory, and discuss it independently in the report of examination. Record the decision criteria used for determining the sample(s) selected in the case notes. If the complexity of the case warrants discussion of the sample(s) selection with another P&P examiner, also record this discussion in the case notes.

## **9 Calculations**

Not applicable.

## **10 Measurement Uncertainty**

Not applicable.

## **11 Limitations**

- a. Sample size and condition can preclude conducting certain examinations, including color assessment and layer structure.
- b. If the sample is a smear, layers can commingle and hinder attempts to isolate each for analysis.
- c. A factory-applied, OEM automotive finish is required for make-model-year determinations.

- d. Sourcing an evidentiary paint specimen to a single make, model, and year may not be feasible because several different vehicles can be manufactured at a single assembly plant and/or the same color of paint can be utilized on different vehicle models over a period of time.

## 12 Safety

Take standard precautions for the handling and disposal of chemicals and sharps. Use universal precautions when handling potentially biohazardous materials. Refer to the most current revision of the *FBI Laboratory Safety Manual* and appropriate Safety Data Sheets for further details.

## 13 References

ASTM E1610, Standard Guide for Forensic Paint Analysis and Comparison. ASTM International, West Conshohocken, PA

Castle, D.A. The forensic examination of paint. *Surface Coatings Int.*, 1992; 75(7): 247-252.

*FBI Laboratory Safety Manual*

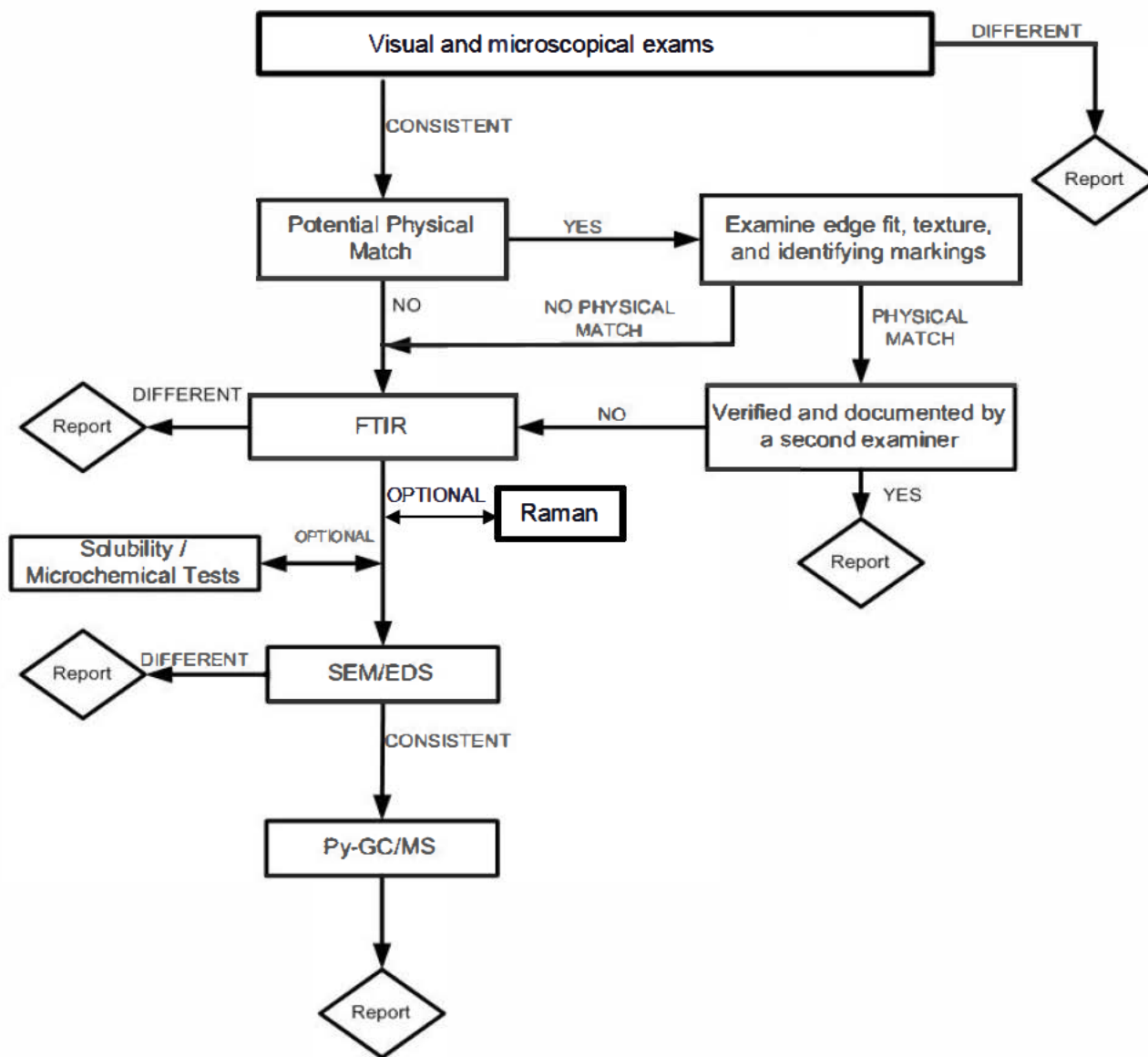
Learner, T.J.S. *Analysis of Modern Paints*. The Getty Conservation Institute, Research in Conservation series, Los Angeles: 2004.

Neilsen, H.K. Forensic analysis of coatings. *J. Coatings Tech.*, 1984; 56(718): 21-32.

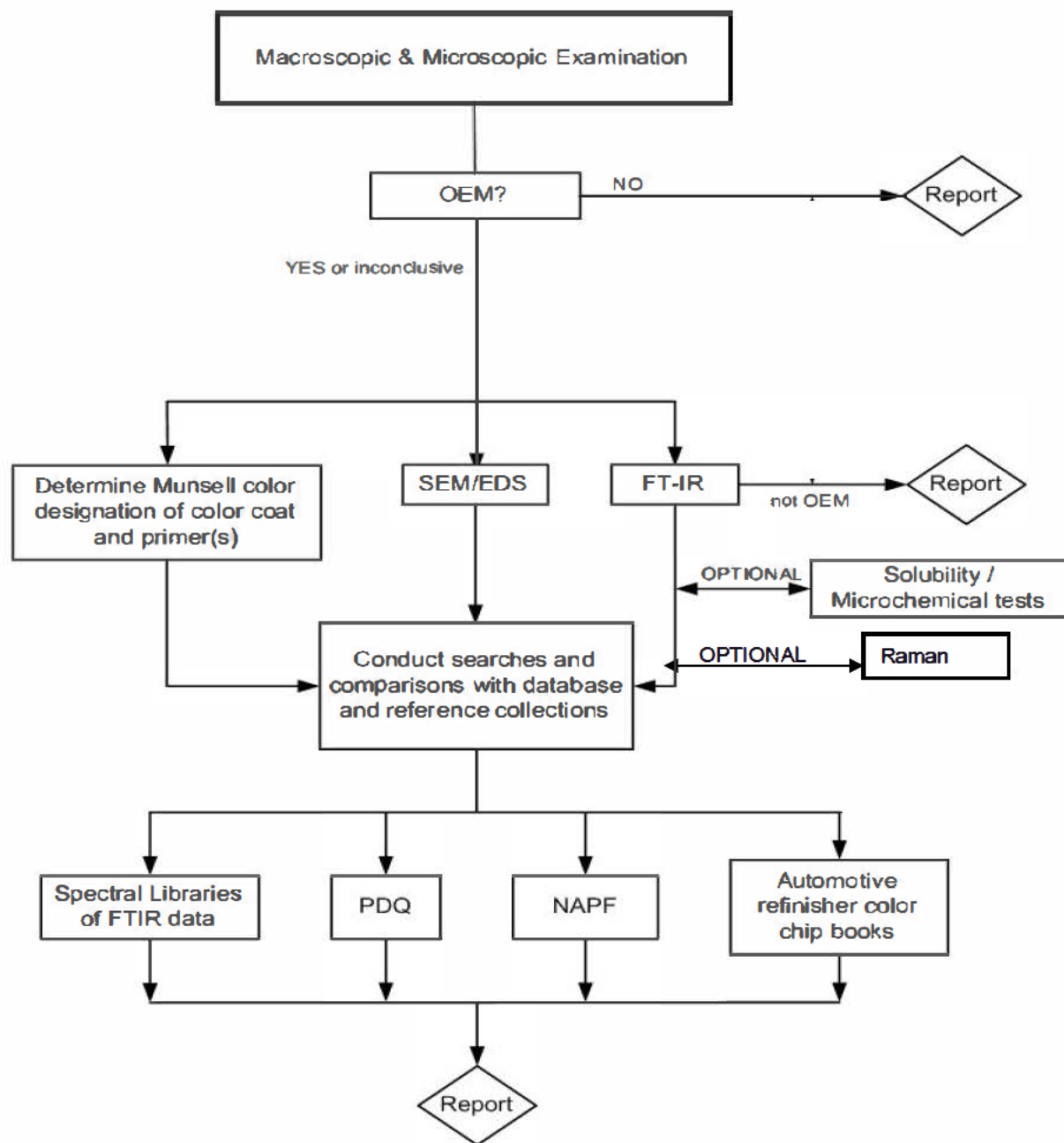
Ryland, S.G. Infrared microspectroscopy of forensic paint evidence. Chapter 6 in *Practical Guide to Infrared Microspectroscopy*. (ed. H.J. Humecki) NY: Marcel Dekker, Inc., 1995.

Ryland, S.G. and Suzuki, E.M. Analysis of paint evidence. Chapter 5 in *Forensic Chemistry Handbook*. (ed. L.F. Kobilinsky) NJ: John Wiley and Sons, Inc. 2012.

**Figure 1: Analytical Scheme for a Paint Comparison Examination**



**Figure 2: Analytical Scheme for Make-Model-Year Search of Automotive Paint**



Rev. #	Issue Date	History
0	06/21/06	New document that replaces previous document also titled <i>General Approach for Paint and Coating Casework</i> .
1	09/30/09	Added a section to describe the sampling plan and added ASTM reference. Revised Figures 1 and 2.
2	03/14/12	Changed “sampling” to “sample selection” in Section 10. Updated references in section 15.
3	02/03/14	Changed “must” to “should” in Section 7 in describing ideal control paint sample, updated item designator example in Section 10 to reflect new QA policies, and made minor grammatical changes.
4	09/18/18	Updated Section 1, Introduction and revised Scope to describe who document applies to; Removed Calibration and Types of Paint Evidence sections as they did not describe procedural content and renumbered. Updated Sections 5, 6, 7 and 8 for clarity. Updated references and added Raman to Figures 1 and 2.

### **Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 09/17/2018

Chemistry Unit Chief:

Date: 09/17/2018

### **QA Approval**

Quality Manager:

Date: 09/17/2018

## **General Approach for Polymeric Casework**

### **1 Introduction**

Polymeric materials, such as adhesives and plastics, can be found in all types of materials including automotive parts, packaging, consumer products, and apparel.

Forensic polymer examinations involve either (1) determining whether a material is a polymer, and if so, its chemical composition, (2) comparing two (or more) samples to determine if they can be differentiated, or (3) developing a probable source or end use.

### **2 Scope**

This procedure describes general guidelines for Chemistry Unit caseworking personnel who analyze polymeric evidence submitted to the FBI Laboratory. Separate, detailed standard operating procedures exist that cover sample processing and acquisition of both physical and chemical compositional data on polymeric evidence.

### **3 Equipment/Materials/Reagents**

Not applicable.

### **4 Standards and Controls**

Not applicable.

### **5 Collection and Preservation Considerations**

Oftentimes when a motor vehicle has been involved in a traffic accident, fragments of plastic such as lens covers can be left at the scene. These pieces can be physically reconstructed with the remnants of the fixture left on the car. Therefore, the potential for physical matches between the broken edges of specimens should be considered before selecting a collection method and care should be taken to keep these edges intact. Pieces of suspected automotive parts should be secured in boxes or in sealable plastic bags. Damaged automotive parts from a suspect vehicle should be removed and packaged separately.

Where possible, items with potential polymeric transfer or smeared polymeric transfer should be submitted in their entirety. Items of clothing should be packaged separately in paper bags. When polymeric evidence is suspected on an immovable item (e.g., roadway, flooring, wall), every



effort should be made to manually collect it from that surface or substrate. If the investigation involves a motor vehicle or other large item, the recommended practice is to cut or section the area where the transfer is suspected.

Small pieces of polymeric material should be contained in pharmacy folded paper, sealable plastic bags, or packaged in covered containers (e.g., pillboxes, glass or plastic vials, film canisters). Cotton, paper bags, and envelopes should not be used for packaging polymeric specimens. Other items, such as tools or sections of automotive parts, should be packaged separately and precautions taken to minimize the potential of dislodging the suspected polymeric transfer during transport to the laboratory.

## **6 Considerations for Other Forensic Examinations**

Process clothing for trace evidence and/or polymeric transfer prior to any fabric impression or DNA examinations. Fragments or pieces of plastic should be processed for latent fingerprints or DNA prior to any polymeric examinations.

Regardless of which unit receives the items first, Paints and Polymers (P&P) personnel will then visually and/or microscopically examine the items and any associated debris for polymeric evidence.

If latent fingerprint and/or toolmark examinations are requested on an item (e.g., a tool), the item should be examined for polymeric evidence first. The suspected polymeric material can be removed from the item with tweezers or a relatively soft, pliable material such as wood or Teflon™. Metal probes and blades must not be used on the working end of a tool as they can alter the surface and interfere with subsequent toolmark examinations. Ensure appropriate laboratory precautions are observed when working with latent print evidence.

## **7 Procedures**

Conduct a critical review of the contributor's request and the item(s) received. As applicable, recommend additional examinations that could be probative and determine the logical sequence for the requested forensic examinations.

In a comparison examination, establish if any differences are detected between two (or more) samples after subjecting them to the same rigorous analytical testing. Figure 1 shows a flowchart that outlines the scheme for a comparison examination of polymeric materials. Each analytical technique depicted in Figure 1 is described in detail in a P&P Standard Operating Procedure (SOP).

With a sourcing request, first examine the material in question for manufacturer markings (e.g., part numbers, descriptors) that would provide useful classification or identification (sourcing) information. If no such markings are found, analyze the material using some or all of the

methods outlined in Figure 2. Based on the results of the analyses, determine whether the material is polymeric in nature, and if so, attempt to identify the polymer composition and/or end use(s). Use the numerous commercially available reference materials and instrumental reference libraries maintained in the FBI Laboratory to obtain compositional information.

## 8 Sample Selection

Due to the wide variety of examination requests, numbers of samples submitted, and conditions of the samples submitted, P&P examiner discretion will determine the appropriate sample(s) to examine on a case-by-case basis. For indistinguishable samples, as determined by a discretionary number of analytical examinations, an option is to take an individual sample, assign a new item identifier (e.g., Item1-1), specifically list the item in the item inventory, and discuss it independently in the report of examination. Record the decision criteria used for determining the sample(s) selected in the case notes. If the complexity of the case warrants discussion of the sample(s) selection plan with another P&P examiner, also record this discussion in the case notes.

## 9 Calculations

Not applicable.

## 10 Measurement Uncertainty

Not applicable.

## 11 Limitations

- a. Sample size and condition can preclude conducting certain examinations.
- b. Sourcing capabilities of common synthetic polymeric materials is limited. This is directly related to the abundance of such materials in the marketplace and the number of end uses for many types of polymeric material.

## 12 Safety

Take standard precautions for the handling and disposal of chemicals and sharps. Use universal precautions when handling potentially biohazardous materials. Refer to the most current revision of the *FBI Laboratory Safety Manual* and appropriate Safety Data Sheet(s) for further details.

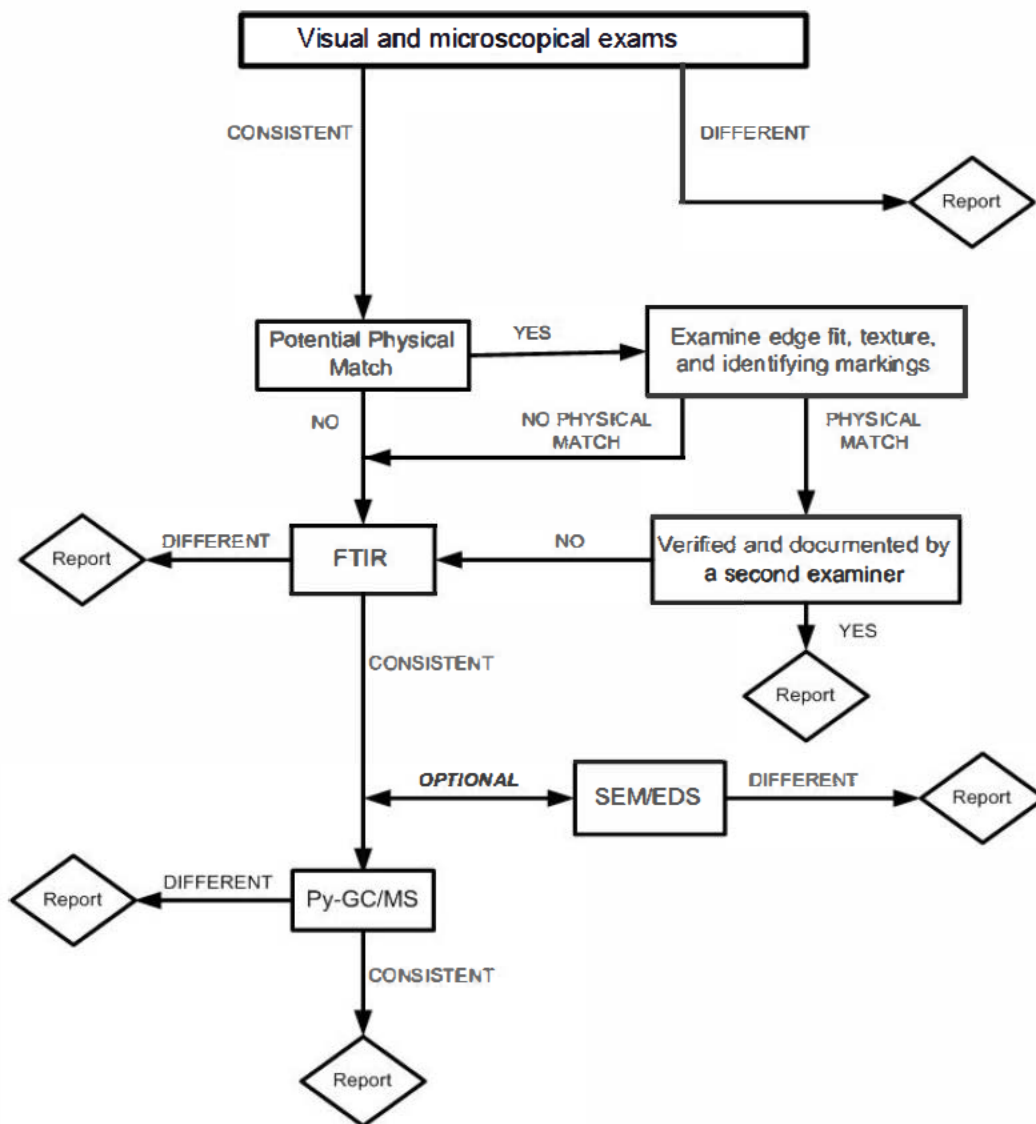
### 13 References

Alger, M.S.M. *Polymer Science Dictionary*. NY: Elsevier Science, 1989

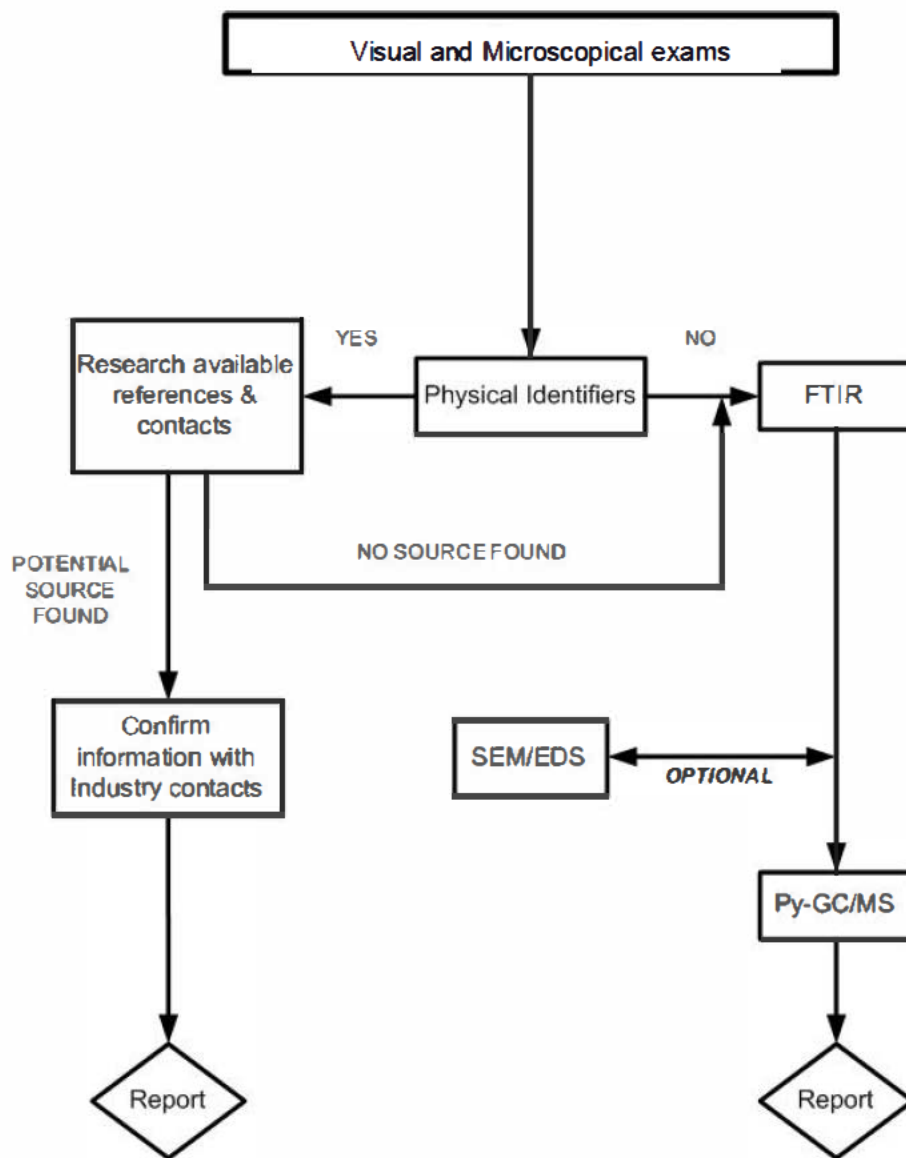
*FBI Laboratory Safety Manual*

Seymour, R.B., Carraher, Jr., C.E. *Polymer Chemistry: An Introduction*, 2d ed. NY: Marcel Dekker, 1988.

**Figure 1: Analytical Scheme for Examination and Comparison of Polymeric Materials**



**Figure 2: Analytical Scheme for Identification of Polymeric Materials**



Rev. #	Issue Date	History
0	06/21/06	New document that replaces previous document also titled <i>General Approach to Polymeric Casework</i> .
1	09/30/09	Added a section to describe the sampling plan. Revised Figure 1.
2	03/14/12	Clarified section 9 in regards to an identification request. Changed “sampling” to “sample selection” in section 10.
3	02/03/14	Updated item designator example in Section 10 to reflect new QA policies and made minor grammatical changes.
4	09/18/18	Updated Section 1, Introduction and revised scope to describe who document applies to; Removed Calibration and Types of Polymeric Evidence sections and renumbered. Updated Sections 5, 6, 7, 8 and 11 for clarity.

### **Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 09/17/2018

Chemistry Unit Chief:

Date: 09/17/2018

### **QA Approval**

Quality Manager:

Date: 09/17/2018

## **General Approach for Tape Casework**

### **1 Introduction**

The most common types of tape evidence received in the FBI Laboratory are duct tape, vinyl electrical tape, and packaging tape. Masking tape and clear office tape are also received but are usually analyzed first by the Questioned Documents Unit (QDU).

Forensic tape examinations involve either (1) determining if a material is a pressure sensitive adhesive to include its type or chemical composition (e.g., styrene-butadiene), (2) comparing two (or more) tape samples to determine if they can be differentiated, or (3) developing a probable source (e.g., manufacturer, brand, point of sale).

### **2 Scope**

This procedure describes general guidelines for Chemistry Unit caseworking personnel who analyze tape evidence submitted to the FBI Laboratory. Separate, detailed standard operating procedures exist that cover sample processing and acquisition of both physical and chemical compositional data on tape evidence.

### **3 Equipment/Materials/Reagents**

Not applicable.

### **4 Standards and Controls**

Not applicable.

### **5 Collection and Preservation Considerations**

When tape is used, pieces are torn or cut from the source roll; a physical reconstruction (end match) is possible between pieces and/or with the free end of the source roll of tape. Therefore, the potential for an end match between torn or cut tape specimens should be considered before selecting a collection method. Care must be taken to preserve the condition of all tape ends.

Whenever feasible, tape should be submitted to the FBI Laboratory still adhered to the original substrate. This will minimize the potential for loss of valuable trace evidence, latent fingerprints, or contact impressions. If it is not reasonable to submit the substrate to which the tape is adhered, it can be manually removed and placed adhesive side down on a clean, colorless piece of plastic sheeting (e.g., transparency film, Kapak<sup>®</sup> tubular rollstock). The tape should not be distorted or torn during this removal process. If the tape is cut during removal, it is imperative to document and initial each cut. A method that produces a unique pattern (e.g., use of pinking

shears) should be employed. Once adhered to plastic sheeting, tape specimens can be packaged in cardboard boxes, paper bags, manila envelopes, or sealable plastic bags. Partially-consumed rolls of tape should be packaged separately.

## **6 Considerations for Other Forensic Examinations**

Some tape examinations can be destructive to materials or latent prints adhered to the tape surfaces. Therefore, in general, analysis of the tape construction and chemical composition should be the last examinations conducted.

## **7 Procedures**

Conduct a critical review of the contributor's request and the item(s) received. As applicable, recommend additional examinations that could be probative and determine the logical sequence for the requested forensic examinations.

In a comparison examination, establish if any differences are detected between two (or more) samples after subjecting them to the same rigorous analytical testing. Figure 1 outlines the analytical approach for comparison examinations of duct tape and other types of tapes. Each analytical technique depicted in Figure 1 is described in detail in a Paints and Polymers Standard Operating Procedure (P&P SOP). If a fabric, fiber, or glass filament reinforcement is present within the tape construction, these analyses are conducted and reported by forensic examiners in other units.

First, examine the specimen(s) in question for manufacturer markings (e.g., product information printed on either the tape backing or the roll core) that would provide useful sourcing information. If no such markings are found, analyze the material using some or all of the methods outlined in Figure 2. Compare the results to known reference samples maintained in the FBI Laboratory to yield information regarding the product grade or manufacturer. Sourcing of tape based solely on analytical examination is limited to duct tape specimens. For sourcing examinations of duct tape, it is up to the discretion of the P&P examiner as to whether fiber examination of the fabric reinforcement will be requested.

## **8 Sample Selection**

Due to the wide variety of examination requests, numbers of samples submitted, and conditions of the samples submitted, P&P examiner discretion will determine the appropriate sample(s) to examine on a case-by-case basis. For indistinguishable samples, as determined by a discretionary number of analytical examinations, an option is to take an individual sample assign a new item identifier (e.g., Item1-1), specifically list the item in the item inventory, and discuss it independently in the report of examination. Record the decision criteria used for determining the sample(s) selected in the case notes. If the complexity of the case warrants discussion of the sample(s) selection with another P&P examiner, also record this discussion in the case notes.



## 9 Calculations

Not applicable.

## 10 Measurement Uncertainty

Not applicable.

## 11 Limitations

- a. Sample size, type, and condition can preclude conducting certain examinations.
- b. Sourcing capabilities of certain types of tape is limited. This is directly related to the number of manufacturers and distributors of a particular type of tape.

## 12 Safety

Take standard precautions for the handling and disposal of chemicals and sharps. Use universal precautions when handling potentially biohazardous materials. Refer to the most current revision of the *FBI Laboratory Safety Manual* and appropriate Safety Data Sheet(s) for further details.

## 13 References

*FBI Laboratory Safety Manual*

Hobbs, A. Use of a database for significance assessment and sourcing of duct tapes. In: *Proceedings of the American Academy of Forensic Sciences Annual Meeting*, Seattle, WA, February 20-25, 2006, 102.

Johnston, J. *Pressure Sensitive Adhesive Tapes*. Northbrook, IL:Pressure Sensitive Tape Council, 2000.

Kee, T.G. The characterization of PVC adhesive tape. *Proceedings of the International Symposium on the Analysis and Identification of Polymers*, FBI Academy, Quantico, VA, July 31- August 2, 1984, 77-85.

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- August 2, 1984, 77-85.

Maynard, P., et al. Adhesive tape analysis: establishing the evidential value of specific techniques. *J. Forensic Sci.* 2001; 46(2): 280-287.

Merrill, R., Bartick, E.G. an approach to the forensic analysis of black plastic tape. Unpublished manuscript, 1992.

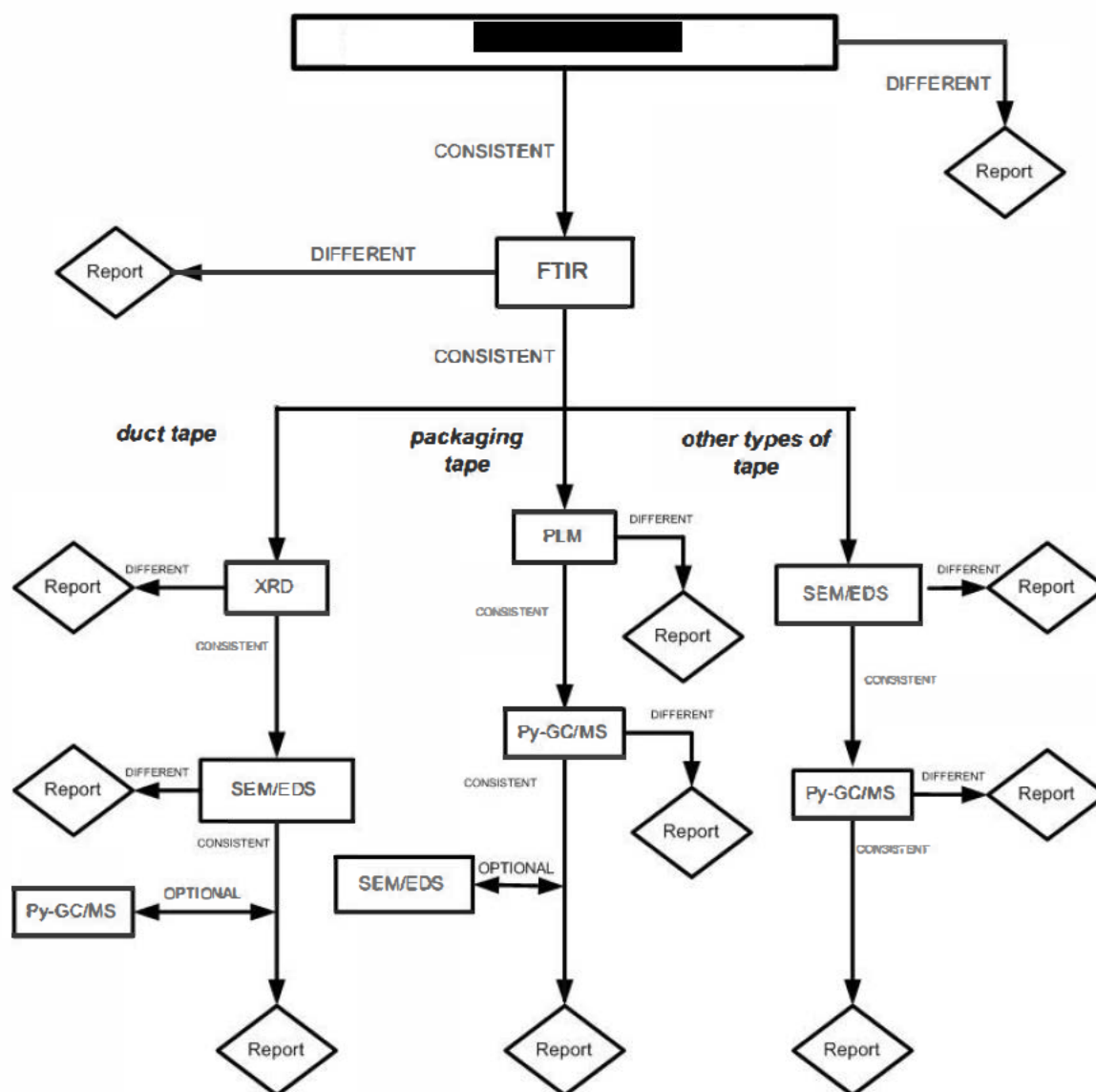
Satas, D. Ed. *Handbook of Pressure Sensitive Adhesive Technology*, 2d ed., NY: Van Nostrand Reinhold, 1989.

Seymour, R.B., Carraher, Jr., C.E. *Polymer Chemistry: An Introduction*, 2d ed. NY: Marcel Dekker, 1988.

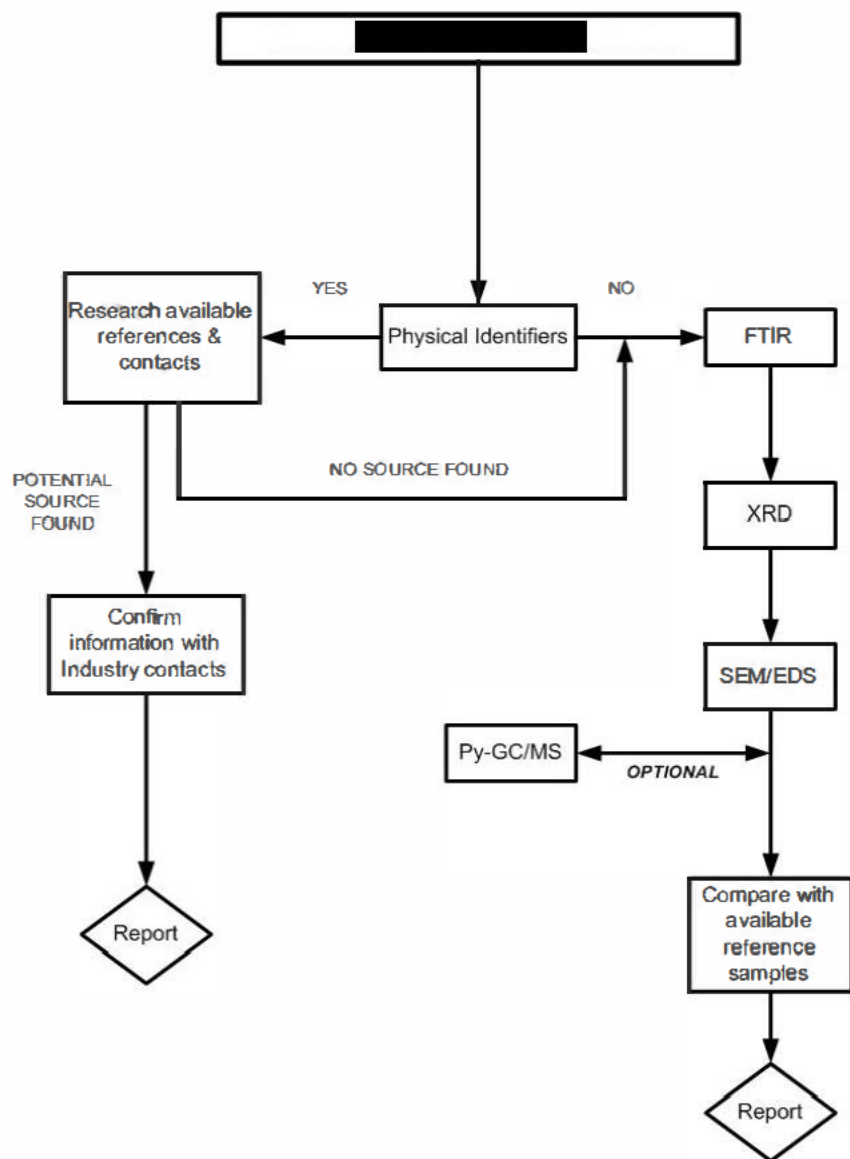
Smith, J. The forensic value of duct tape comparisons. *Midwestern Association of Forensic Scientists, Inc. Newsletter*, 27(1), January 1998, 28-33.

Scientific Working Group for Materials Analysis (SWGMA). Guideline for the Forensic Examination of Pressure Sensitive Tapes. *Forensic Science Communications*, 2008; 10(4).

**Figure 1: Analytical Scheme for Examination and Comparison of Tapes**



**Figure 2: Analytical Scheme for Identification of Tape Specimens**



Rev. #	Issue Date	History
0	06/21/06	New document that replaces previous document also titled <i>General Approach for Tape Casework</i> .
1	09/30/09	Added a section to describe the sampling plan and updated references. Revised Figures 1 and 2.
2	03/14/12	Changed “sampling” to “sample selection” in section 10. Deleted a reference in section 15.
3	02/03/14	Updated item designator example in Section 10 to reflect new QA policies and made changes to Figures 1 and 2.
4	09/18/18	Updated Section 1, Introduction and revised Scope to describe who document applies to; Removed Calibration and Types of Tape Evidence sections and renumbered. Updated Sections 5, 6, 7 and 8 for clarity.

### **Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 09/17/2018

Chemistry Unit Chief:

Date: 09/17/2018

### **QA Approval**

Quality Manager:

Date: 09/17/2018

## Paints and Polymers General Approach to Report Writing

### 1 Scope

Reports issued by Paints and Polymers examiners summarize analytical findings and provide interpretation of paint/polymer results. Due to the wide variety of requests and evidence received, this standard operating procedure is only a general guideline for report writing. It will not always be possible to write a report using only the examples given here. It is acceptable to use other wording as long as the results of the examinations are accurately communicated, a summary of the methodology used to reach the results is included, any known limitations are addressed, and the wording is approved by a second qualified paint/polymer examiner during the technical review process. Additionally, any wording must comply with the *FBI Approved Standards for Scientific Testimony and Report Language for the Paints and Polymers Discipline* (P&P ASSTR).

This document applies to Chemistry Unit (CU) personnel that are authorized to author *Laboratory Reports* that pertain to Paints and Polymers materials.

### 2 Equipment/Materials/Reagents

Not applicable.

### 3 Standards and Controls

Not applicable.

### 4 Sampling

Not applicable.

### 5 Procedure

- a. Prepare and format the *Laboratory Report* in accordance with requirements set forth in the *FBI Laboratory Operations Manual*. Prepare a **Results of Examinations** section, an **Interpretation** section as applicable, and a **Remarks** section. Any substantive changes to the *Laboratory Report* that occur during technical review will be recorded.
- b. The **Results of Examinations** section will be used to communicate the results of the Paints and Polymers examinations and a summary of the methodology used, and will include the

requirements set forth in the *FBI Laboratory Operations Manual*. This section may also include a description of the items received, a statement regarding sampling (as appropriate), or any other information to assist in communicating the results (e.g., any pertinent limitations of the samples and/or the results that are not included in an **Interpretation** section). The below list contains guidance for additional information that is included in the **Results of Examinations** section for Paints and Polymers reports. Appendix 1 contains example text/scenarios for the **Results of Examinations** section.

- A conclusion statement for comparisons which describes the examiner's opinion as to whether the items could be associated/discriminated.
  - A category for the opinion is assigned to provide context for the conclusion within a scale framework. This category will align the results to the **Interpretation** Section.
  - An explanation as to why the assigned category was chosen.
- c. The **Interpretation** section will be used to communicate any limitations not described in the **Results of Examination** section as well as to provide further guidance that may aid the reader in understanding the examiner's opinion as to the significance of the reported results. This section will contain a *Characterization* Scale when reporting the chemical composition or manufacturing information of a material. A *Comparison* Scale is included when describing the stated conclusions in a comparative examination. These scales are included to provide context to the reported results. Appendices 2 and 3 contain example text that will appear under the heading of **Interpretation** in reports where a scale has been used.
- d. The **Remarks** section will include the requirements set forth in the *FBI Laboratory Operations Manual*. The following information may also be included in the **Remarks** section when applicable.
- A listing of the evidence received but not examined.
  - Guidance to properly collect, mark, and preserve paint/polymer specimens in the future.
  - Other information to assist the reader that does not belong in another section of the report.
- e. If cross-transfer is reported, text similar to the following may also be included: "From the above results, a cross transfer of material between A and B appears to have occurred. These results add additional weight to the association."
- f. If multiple evidentiary materials appear to have transferred from one source (e.g., vehicle, victim) to another, text similar to the following may also be included: "From the above results, transfer of multiple materials appears to have occurred from A to B. These results may add additional weight to the association."
- g. Copies of issued reports will be maintained for reference in a central, designated area.

## 6 Calculations

Not applicable.

## 7 Measurement Uncertainty

Not applicable.

## 8 Limitations

Not every scenario can be anticipated. This document serves as a general guideline only.

## 9 Safety

Not applicable.

## 10 References

*FBI Laboratory Operations Manual*

<http://projects.nfstc.org/trace/2009/presentations/4-bommarito-report.pdf>

*Standard Practice for Interpretation and Report Writing in Forensic Comparisons of Trace Materials*, Materials (Trace) Subcommittee, Chemistry Scientific Analysis Committee, Organization of Scientific Analysis Committees (OSAC), draft dated December 2019; available at <http://www.nist.gov/topics/forensic-science/materials-trace-subcommittee>.



Rev. #	Issue Date	History
0	06/08/10	New document.
1	03/14/12	Example text for cross-transfer and multiple transfers moved from section 7, part 2, to section 7, parts 5 and 6, and the remainder renumbered accordingly. The “supporting documentation” statement was removed from section 7, part 4. Section 7, old part 6, deleted. Added reference to section 12.
2	02/03/14	Updated item designator example in Appendix 1 to reflect new QA policies and made minor grammatical changes.
3	08/03/15	Removed sections no longer required by <i>Practices for Writing Standard Operating Procedures</i> and re-numbered sections accordingly; changed “Levels” to “Types” throughout the entire document, combined <i>Levels IV</i> and <i>V</i> in Appendices 1 and 2 to now read as <i>Type IV</i> , and modified verbiage in Appendix 2 to align more closely with Trace/Materials OSAC subcommittee language; resultant minor terminology changes to coincide with same in Section 6 of the Procedure, step #6, and updated references.
4	02/27/18	Removed “subunit” throughout. Added “limitations are addressed” to Section 1. Edited Scope per LOM changes. Removed Sections that pertain only to technical procedures and renumbered remaining sections. Reordered Section 3 (previously 6) to describe Results, Interpretation, and Remarks in the order these would appear in a report. Added language in Section 6 to describe results involving identifications and classifications of materials as well as reporting no transfer observed or transfer in one direction only. Updated references. Changed Appendix 1 to include examples of identification and classification and no transfer observed language and deleted analytical techniques wording; updated Appendix 2 to Interpretation Scale for characterization; added Appendix 3 to be the Interpretation scale for comparisons and edited wording.
5	01/15/20	Moved content of Introduction section to Scope section and re-numbered sections accordingly; added <i>Inconclusive</i> and <i>Negative</i> definitions and examples to the <i>Characterization</i> scale; added sections required by FBI Laboratory Operations Manual; updated references; minor edits throughout to align with other Chemistry Unit documents.

**Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 01/14/2020

Chemistry Unit Chief

Date: 01/14/2020

## **Appendix 1: Examples of Appropriate Wording for the Results of Examinations Section of a Paints and Polymers Report**

### **Characterizations:**

#### *Identification:*

The Item 1 paint chip was *identified* as an original equipment manufacturer (OEM) automotive paint system.

#### *Classification:*

Based on resources available to the FBI Laboratory, the Item 1 paint chip is consistent with originating from a 2006 Hyundai Elantra produced in Ulsan, Korea, with the color code VZ, also known as Spruce Green (*Classification*).

#### *Indication:*

Physical and chemical characterization of the material *indicates* that it is possibly a decal.

#### *Inconclusive:*

Item 1 was visually and stereomicroscopically examined for the presence of glue. Extraneous material was observed and chemically characterized. This material is consistent with a chemical used in both latent print processing as well as in common glue formulations. Therefore, it cannot be determined whether this extraneous material was applied before or during laboratory processing (*Inconclusive*).

#### *Negative:*

The Item 1 through 3 clothing and their associated debris were visually and stereomicroscopically examined for the presence of automotive paint. None was observed (*Negative*).

### **Comparisons:**

#### *Type I:*

The Item 1 paint chip was visually and microscopically examined and compared to the Item 2 paint chip recovered from the suspect vehicle. Based on these examinations, Item 1 fractured from Item 2. This reconstruction demonstrates that Item 1 and Item 2 were once a single item (*Type I Association*).

#### *Type II:*

Item 1 is a six-layered blue metallic automotive paint chip. Visual and microscopical examinations revealed that Item 1 contains a layer structure consistent with Item 2. The layer structures consist of four factory-applied, original equipment manufacturer's (OEM) layers with

additional aftermarket clear and blue layers applied on top. These specimens were further compared chemically.

Based on the examinations conducted, the six layers of paint comprising the Item 1 paint chip are comparable in sequence, color, texture, relative thickness, and chemical composition with the corresponding layers of paint in the Item 2 paint exemplar. Therefore, the Item 1 paint chip originated from a repainted area of the suspect vehicle represented by Item 2, or from another vehicle painted in the same manner (*Type II Association*). This type of association was reached due to the presence of two aftermarket repaint layers on top of four OEM paint layers.

*Type III:*

Item 1 is a black nonmetallic four-layered automotive paint chip. This paint chip was examined and compared to the Item 2 paint exemplar.

Based on the examinations conducted, both Item 1 and Item 2 are factory-applied, original equipment manufacturer's (OEM) automotive finishes. The four layers of paint comprising Item 1 are comparable in sequence, color, texture, relative thickness, and chemical composition to the corresponding layers of paint in Item 2. Accordingly, Item 1 and Item 2 originated from the same vehicle or from different vehicles painted in the same manner (*Type III Association*). This type of association was reached because vehicles produced at the same manufacturing plant as the source of Item 2, which were painted with the same color code and same paint formulations, would also be indistinguishable from the source of Item 2.

*Type IV:*

The Item 1 paint chips recovered from the BMW consist of two factory-applied, original equipment manufacturer's (OEM) layers: a clear coat and a silver metallic layer. These chips are physically consistent with the corresponding layers of the Item 2 silver metallic paint exemplar from the Honda. Further, the corresponding paint layers in Item 1 and Item 2 are comparable in chemical composition. Based on these examinations, the area of the vehicle represented by Item 2 cannot be excluded as the source of the Item 1 paint chips (*Type IV Association*). This type of association was reached due to the limited layer structure of the Item 1 paint chips as well as the prevalence of silver metallic automobiles.

*or*

Visual and microscopical examination of the red smears present on the Item 1 paint chip from the GMC Yukon could not definitively ascertain how many layers were transferred to its surface. However, chemical analyses indicated that these red smears are generally consistent with the aftermarket refinish topcoat on the Item 2 paint exemplar from the Jeep. Therefore, the area of the Jeep represented by Item 2 cannot be excluded as the source of the red smears present on the Item 1 paint chip (*Type IV Association*). This type of association was reached because of the limited sample size of the smear as well as potential variations within the sample.

*Inconclusive:*

A black smear was noted on the Item 1 paint chip and was compared to the Item 2 paint. Due to variations in the composition of the black smear on Item 1, a comprehensive comparison to Item 2 could not be conducted (*Inconclusive*).

*Elimination:*

The Item 1 paint chips were examined and compared to the Item 2 paint. Based on the examinations conducted, the Item 1 paint chips did not originate from the same source as Item 2 (*Elimination*). This conclusion was reached because Item 1 and Item 2 differ in layer structure.

**No transfer or one-way transfer:**

*No transfer observed:*

Such a result can be interpreted in several ways: 1) automotive paints/polymers had no contact with the items, 2) automotive paints/polymers may not have transferred during contact, or 3) automotive paints/polymers that did transfer may have been lost prior to submission to the FBI Laboratory.

*Transfer observed in one direction only:*

Possible reasons for this result (e.g., paint transfer is being reported from Vehicle A to Vehicle B, but not in the opposite direction) are that the transfer on Vehicle A did not result from contact with Vehicle B; paint recovered from Vehicle A may not have transferred during contact with Vehicle B; or the area represented by Item X is not the area of Vehicle B that made contact with Vehicle A.

## **Appendix 2: Appropriate Wording for the Interpretations Section of a Characterization Paints and Polymers Report**

The following categories and their descriptions are meant to provide context to the conclusions reached in this report. Every category may not be applicable in every case nor for every material.

*Identification:* The analytical data provides reliable information to specify a particular chemical or product.

*Classification:* The analytical data does not support an identification of a specific chemical or product but does provide reliable information to include the substance within a class of materials. The phrase “consistent with” may be used in this context.

*Indication:* The analytical data suggests a particular type of material but does not support a classification or identification. The terms “possible” and “similar to” may be used in this context.

*Inconclusive:* No conclusion could be reached.

*Negative:* No material of interest was observed.

### **Appendix 3: Appropriate Wording for the Interpretations Section of a Comparative Paints and Polymers Report**

The following categories and their descriptions are meant to provide context to the conclusions reached in this report. Every category may not be applicable in every case nor for every material.

*Type I Association: Physical/Fracture Match* – The items exhibit physical features that demonstrate they were once part of the same object.

#### *Associations of Evidence with Class Characteristics:*

Class characteristics are physical and/or chemical properties that place an item within a particular group of items. Associations of evidence with class characteristics can have varying degrees of significance. In general, the smaller the size of the group relative to the relevant population, the more significant the association. A class association cannot definitively establish that the items came from the same source.

*Type II: Association with Highly Discriminating Characteristics* – An association in which items could not be differentiated. Therefore, the possibility that the items came from the same source cannot be eliminated. Additionally, the items share unusual characteristics that would not be expected to be encountered in the relevant population.

*Type III: Association with Discriminating Characteristics* – An association in which items could not be differentiated. Therefore, the possibility that the items came from the same source cannot be eliminated. Other items have been manufactured that would also be indistinguishable from the submitted items and could be encountered in the relevant population.

*Type IV: Association with Limitations* – An association in which items could not be differentiated. Therefore, the possibility that the items came from the same source cannot be eliminated. As compared to the categories above, this type of association has decreased evidential value. For example, the items are more commonly encountered in the relevant population, a complete analysis was not performed due to limited characteristics or a limited analytical scheme, or minor variations were observed in the data.

*Inconclusive* – No conclusion could be reached.

*Elimination* – The items exhibit exclusionary differences that demonstrate they did not originate from the same source.

## **Chemistry Unit (CU)**

# **FBI Approved Standards for Scientific Testimony and Report Language for Paints and Polymers Materials**

### **1 Purpose**

This document provides examples of the scientifically-supported conclusions and opinions approved for reporting examination conclusions and offering expert opinion statements during testimony by Paints and Polymers Examiners. It is noted that these examples are not intended to be all inclusive and may be dependent upon the precedent set by the judge or locality in which a testimony is provided. Further, these examples are not intended to serve as precedent for other forensic laboratories and do not imply that statements by other forensic laboratories are incorrect, indefensible, or erroneous.

### **2 Scope**

This document applies to Chemistry Unit employees who prepare an FBI *Laboratory Report* and/or provide testimony related to Paints and Polymers materials. This document does not apply to Chemistry Unit employees who provide fact witness testimony.

### **3 Responsibilities**

**3.1** The Examiner will ensure that a *Laboratory Report* is consistent with the approved language contained within this document as well as the *Paints and Polymers General Approach to Report Writing*.

**3.2** The Examiner will ensure that testimony related to Paints and Polymers examinations is consistent with the statements contained within this document.

**3.3** The Technical and Administrative Reviewers will ensure compliance of Paints and Polymers *Laboratory Reports* with the statements contained within this document as well as the *Paints and Polymers General Approach to Report Writing*.

### **4 Statements Approved for FBI Paints and Polymers Testimony and/or Laboratory Reports**

For more detailed guidance on report writing regarding Paints and Polymers materials, see the *Paints and Polymers General Approach to Report Writing* standard operating procedure (SOP).



- An examiner may report and/or state an association between two or more items based on their physical and/or chemical properties. For the large majority of such cases, these associations are limited to class characteristics and, as such, are not individualizing.
- The examiner may report and/or state the relative strength of the association. The degree to which this association is qualified is stated in the report of examinations using the *Comparison Conclusion Scale* that is defined in the *Paints and Polymers General Approach to Report Writing SOP*.
- The examiner may report and/or state that additional significance may be given to examples of cross-transfer and/or if multiple types of evidence appear to have transferred from one source to another.
- An examiner may report and/or state that an *Elimination* is the determination that two paint/tape/polymer items did not originate from the same source due to sufficient differences in their physical or chemical properties.
- The examiner may report and/or state the limitations of his/her examinations and opinion.
- An examiner may report and/or state the polymeric composition of an item according to the terms described in the *Characterization Conclusion Scale*, as well as the possible common uses of the material.
- An examiner may report and/or state the likely manufacturer of an automotive paint or duct tape based on resources available to the FBI Laboratory (e.g., databases, industry contacts) according to the terms described in the *Characterization Conclusion Scale*.
- An examiner may report and/or state that an *Inconclusive* is the inability to reach a conclusion.
- An examiner may report and/or state the manufacturing process used to produce a paint/tape/polymer item and may explain the variability possible between products.
- An examiner may report and/or state the batch size involved in production, such as how many single rolls can be produced from a jumbo duct tape roll or how many vehicles from an assembly line might contain the same paint layer system.
- An examiner may report and/or state the application process used to paint an item when the physical characteristics permit such an inference.

## 5 Statements Not Approved For FBI Paints and Polymers Testimony and/or Laboratory Reports

- An examiner may not state or imply that the methods used in conducting paint/tape/polymer comparisons have a zero error rate.
- An examiner may not state or imply a statistical weight or degree of certainty in the conclusions that is absolute or numerically calculated.
- An examiner may not state or imply that a conclusion has been reached to within a “reasonable degree of scientific certainty” as this term has no basis in scientific inquiry or research and has been strongly discouraged for use by the National Commission on Forensic Science.
- An examiner will not cite the number of Paints and Polymers examinations performed in the span of a career as a direct measure for the accuracy of the proffered conclusion. (An examiner may cite the number of Paints and Polymers examinations performed within the span of a career for the purpose of establishing, defending, or describing the stated qualifications or experience.)

## 6 Laboratory Report Reviews

The content of a Paints and Polymers *Laboratory Report* will be reviewed per the *FBI Laboratory Quality Assurance Manual*, *FBI Laboratory Operations Manual*, and the *Chemistry Unit Case Record and Review Procedures*, as well as the *Paints and Polymers General Approach to Report Writing* to ensure compliance with the statements in this document.

## 7 Testimony Reviews

Paints and Polymers testimonies will be reviewed following the *FBI Laboratory Operations Manual*. The review will ensure compliance with the statements in this document.

## 8 References

*Paints and Polymers General Approach to Report Writing SOP.*

*Chemistry Unit Case Record and Review Procedures, Chemistry Unit Quality Assurance and Operations Manual.*

ASCLD-LAB-International Supplemental Requirement for the Accreditation of Forensic Science Testing and Calibration Laboratories. American Society of Crime Laboratory Directors/Laboratory Accreditation Board, Garner, NC, 2011.

FBI Laboratory Quality Assurance Manual. Latest Revision.

FBI Laboratory Operations Manual. Latest Revision.

[www.justice.gov/ncfs](http://www.justice.gov/ncfs), NCFS Meeting #9 - March 21-22, 2016

Rev. #	Issue Date:	History:
0	05/23/14	New document.
1	08/31/15	Changed second bullet in Section 4 to reflect the change made to the SOP <i>General Approach to Report Writing in the Paints and Polymers Subunit</i> , where “Level” has been replaced by “Type” and the number of designations has been changed from V to IV.
2	02/27/18	Removed term “subunit” throughout, added <i>Characterization Association Scale</i> and error rate mitigation in Section 4 and deleted specific scenario examples; stated no support for “reasonable degree of scientific certainty” in Section 5; editorial changes throughout to align with latest version of LOM.
3	01/15/20	Deleted section 3.4 since now covered in the LOM; amended statement approved for an <i>Inconclusive</i> result; added an additional statement to the not approved language section (section 5); minor grammatical changes; removed QA approval line

### **Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 01/14/2020

Chemistry Unit Chief

Date: 01/14/2020

## FTIR Analysis of Paints, Tapes, and Polymers

### 1 Scope

This procedure applies to Chemistry Unit caseworking personnel who analyze paints and polymers via (Fourier transform infrared spectroscopy) FTIR. This document describes the sample preparation and suggested instrumental parameters for the FTIR analysis of paints, tapes, and other polymeric materials.

### 2 Equipment/Materials/Reagents

- a. FTIR Spectrometer with operating and search software: Nicolet Nexus 6700 FTIR E.S.P. spectrometer with Omnic software (or equivalent)
- b. Microscope Accessory: Continuum microscope with Mercury Cadmium Telluride (MCT)/A detector (or equivalent)
- c. Attenuated Total Reflectance (ATR) Accessory: SmartOrbit (or equivalent)
- d. Liquid Nitrogen
- e. Polystyrene 1.5 film
- f. Diamond compression cell (Spectra-Tech Inc., Shelton, CT or equivalent)
- g. IR inactive windows (e.g., Potassium Bromide (KBr)) (Spectra-Tech Inc., Shelton, CT or equivalent)
- h. Stereo microscope (~6X to ~100X) with appropriate light source (e.g., an annular ring light, fiber optic light)
- i. Scalpel handle with blades
- j. Roller knife (Spectra-Tech Inc., Shelton, CT or equivalent)
- k. Probes (steel, tungsten, wood or Teflon<sup>TM</sup>)
- l. Glass microscope slides
- m. Tweezers
- n. Hexane (Reagent grade)
- o. Chloroform (Reagent grade)

- p. Disposable wipes (Kimwipes or equivalent)
- q. Compressed-gas duster
- r. Cotton swabs

### 3 Standards and Controls

#### 3.1 Standards

Manufacturer-supplied and commercially available paints, tapes, polymers, adhesives, and sealants are maintained in reference collections within the FBI Laboratory. These materials are used in casework in accordance with the *Procedures for the Use of Reference Materials and Known Materials*.

#### 3.2 Performance Check

Refer to the *Performance Monitoring Protocol (QA/QC) for the Thermo Nicolet FTIRs* for details on the performance checks and necessary supplies to conduct the check and operate the instrument.

### 4 Sample Selection

Refer to the current version of the relevant material's *General Approach* Paints and Polymers Standard Operating Procedure (P&P SOP) (e.g., PPSU 100, PPSU 101, PPSU 102) for guidance on sample(s) selection. Record the sample(s) selected for analysis in the case notes.

### 5 Procedure

Cease comparison examinations whenever a test reveals a difference between the two (or more) samples being compared. Record any factors limiting the analysis, (e.g., sample size, condition) in the case notes.

1. If using the microscope accessory, determine whether the detector has been cooled. If it has not, fill the detector reservoir with liquid Nitrogen.
2. Verify that the daily performance check has been successfully completed and recorded before proceeding to sample analysis.
3. Sample an item from a clean, core area (e.g., cut into the sample and discard the top portion, or clean the surface). If appropriate, dried material (e.g., cured spray paint on the nozzle) can be sampled from the container of an uncured specimen. Alternatively, a portion of an uncured sample (e.g., glues, two-part adhesive systems, liquid paint) can be

mixed, applied to a clean microscope slide or other suitable substrate, and permitted to dry/harden according to the manufacturer's recommendations.

As described below, samples are prepared in a manner suitable for analysis by transmission infrared microscopy. ATR is also noted as an alternative where applicable.

a. Paint:

Prepare either thin peels of individual layers or a cross-section of a multi-layered specimen. Thin peels can be achieved by manually cutting through individual layers with an angled scalpel blade or similar tool. Cross-sections are achieved manually or with a microtome. Compress each thin layer or cross-section with a roller, scalpel blade, between two IR inactive windows, or between the two windows of a diamond compression cell. Alternatively, analyze an area free of visible contaminants directly by ATR.

b. Tape:

- i. Adhesive: Sample a core area, free of any contamination, with a scalpel or similar tool. Smear the adhesive evenly onto an IR inactive window. Alternatively, analyze an area free of visible contaminants directly by ATR.
- ii. Backing: For duct tapes, clean the top side of the backing with a cotton swab or disposable wipe and a suitable solvent (e.g., alcohol). Thoroughly remove the adhesive and reinforcement fabric from an area of the tape with a suitable solvent (e.g., hexane). Proceed to step 5 and analyze both sides of the backing by ATR. For other types of tapes, clean the tape backing with a cotton swab or a disposable wipe and, if necessary, appropriate solvent. Take a thin peel of a core area of the backing with a scalpel or similar tool. Compress with a roller, scalpel blade, between two IR inactive windows, or between the two windows of a diamond compression cell. Alternatively, analyze the backing directly by ATR.
- iii. If warranted, the plasticizer or other organic material(s) can be extracted from the backing of a tape and analyzed. Sample a portion of the backing. Remove any residual adhesive manually with a scalpel or chemically with a solvent (e.g., hexane). Extract the backing with an appropriate solvent (e.g., chloroform for plasticizers in Polyvinyl Chloride (PVC) electrical tapes). Allow the extract to air-dry. Smear the extract evenly onto an IR inactive window.

c. Glues, sealants, elastomers, and plastics:

Sample a core area with a scalpel or similar tool. Compress with a roller, scalpel blade, between two IR inactive windows, or between the two windows of a diamond compression cell. Alternatively, analyze directly by ATR.

4. If using the microscope accessory, place the IR inactive window containing the sample into the appropriate holder and place the window and holder on the microscope stage. Adjust the compensator to accommodate the sample holder. View the sample of interest

through the microscope. Adjust the apertures so that the sample fills the field of view. Collect a spectrum using the instrumental conditions listed below. After analyzing the sample, move to a clear area of the window without making any other adjustments and collect a background spectrum. Proceed to step 6.

5. If using an ATR accessory, clean the crystal surface with a cotton swab or disposable wipe. Collect a background spectrum using the instrumental conditions listed below. For tape adhesives, place the sample area of interest in direct contact with the crystal and collect a spectrum using the instrumental conditions listed below. For cured material (e.g., paints, polymeric films, elastomers), place the sample with the area of interest on the crystal. Turn the compression device until the sample area is in direct contact with the crystal surface. Collect a spectrum using the instrumental conditions listed below.
6. Clean the IR windows or ATR crystal manually with disposable wipes, compressed-gas duster, or an appropriate solvent between samples and after daily use.

## 6 Instrumental Conditions

The following instrumental conditions are a guide for all standards and samples described in this SOP:

Parameter	Microscope	ATR
Detector:	MCT/A	DTGS <sup>1</sup>
Spectral range (cm <sup>-1</sup> ):	4000 - 650	4000 – 550
Beam splitter:	KBr	KBr
Source:	IR	IR
Gain:	Auto	Auto
Resolution (cm <sup>-1</sup> ):	4	4
Minimum number of sample scans:	128	32
Minimum number of background scans:	128	32

## 7 Decision Criteria

- a. Differences between spectra of samples being inter-compared or compared to a known material or a library are considered to be true differences in compositional components if those differences are determined to be meaningful. A meaningful difference is defined as a feature or property of a sample that does not fall within the variation exhibited by the comparison sample, considering the limitations of the sample or technique, and therefore indicates the two samples do not share a common origin. In that regard, an example of a meaningful difference would be the presence of polyurethane in only one of the paints being compared.

<sup>1</sup> Deuterated TriGlycine Sulfate Detector



- b. If meaningful differences are observed between the FTIR spectra of two (or more) samples being compared, then it is concluded that the specimens are different.
- c. If no meaningful differences are observed between the FTIR spectra of two (or more) samples being compared, then it is concluded that they are indistinguishable from one another.
- d. If FTIR is being used to characterize a material type, the FTIR spectrum should compare favorably to a corresponding spectrum of a known material or library spectrum.
- e. Proper sample preparation is critical for FTIR absorption/transmission analysis. The following are some common sample preparation problems encountered in FTIR analysis, which can result in spectral differences that need to be evaluated to determine if they are meaningful.
  - i. Over absorption of a sample is characterized by rounded peaks or peaks that flatten out along the baseline, (i.e., not sharply-defined). To resolve this issue, the specimen should be re-sampled with a smaller sample size or compressed further to achieve a thinner analysis area.
  - ii. Under absorption can occur when insufficient sample is placed in the field of view. To resolve this issue, adjust the aperture opening or repeat sampling to obtain a larger sample size.
  - iii. Interference fringes are often observed in the transmission spectrum of a thin film introduced to the beam on an IR inactive window due to internal reflections of the IR radiation. The interference fringe can alter the relative band intensities or give the appearance of additional peaks. This phenomenon is most commonly observed for clear, colorless films. To reduce this effect, break direct contact between the specimen and the IR inactive window, transfer the specimen to a different IR inactive window, or roughen the surface of the prepared specimen. Alternatively, repeat the sample preparation and analyze the newly prepared sample.

## 8 Calculations

Not applicable.

## 9 Measurement Uncertainty

Not applicable.

## 10 Limitations

- a. The inability to discriminate color and/or texture differences between layers (e.g., adjacent white paint layers) can result in inadequate sampling.
- b. Some useful characteristic IR peaks occur outside of the chosen accessory's detector spectral range (e.g., inorganic pigment absorption bands).
- c. The spectrum of a mixture can be difficult to interpret due to spectral overlap (e.g., calcite and isoprene at  $\sim 1450$  and  $\sim 1375$   $\text{cm}^{-1}$ , tackifier and butadiene at  $\sim 967$   $\text{cm}^{-1}$ ).
- d. The sub-generic class of polymers cannot always be determined by FTIR analysis (e.g., type of acrylic).
- e. Available sample size can limit or preclude analysis by this technique. A cross-section less than 15 microns wide cannot be analyzed.
- f. FTIR analysis is not effective for elucidation of water-based peaks.

## 11 Precautionary Statements

- a. As with any procedure involving trace evidence, ensure actions minimize the potential for loss or contamination of the sample.
- b. The presence of some pigments can cause difficulty with interpretation of the resulting spectrum. For example, carbon black causes an upward slant to the spectrum and organic red pigment(s) often have very sharp peaks in the region of interest for paint components.
- c. The elasticity inherent in some polymers can cause the flattened sample to curl up when the object used to flatten the specimen is removed.

## 12 Safety

Use standard precautions for the handling of potentially biohazardous materials, chemicals, or sharps. Refer to the *FBI Laboratory Safety Manual* and appropriate Safety Data Sheet(s) for further details. Operators should familiarize themselves with the specific User's Guide safety section of the instrument prior to use.

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Rev. #	Issue Date	History
0	06/21/06	New document that replaces previous document also titled <i>FTIR Analysis of Paints, Tapes, and Polymers</i> .
1	09/30/09	Changed the sampling plan guidelines and updated references.
2	03/14/12	Updated equipment in section 4. Added statement to section 7 to refer reader to other PPSU SOPs regarding sample selection. Changed "sampling" to "sample selection" in section 7. Changed monthly comprehensive evaluation to be optional in section 5.2. Added new section 8, part 2 regarding observing instrument and renumbered subsection. Also, deleted reference to monthly QA/QC in section 8, part 3. Changed instrument standby conditions in section 8, part 7. Removed instrument parameters for MCT/B detector in section 9. Updated decision criteria in section 10, parts a, b, and c. Changed wording of precautionary statement in section 14, part a. Updated references in section 16.
3	02/03/14	Specified that instrument specific polystyrene standard is used for performance checks, made minor grammatical changes, specified sampling for cured vs uncured samples in Section 8, part 4, specified instrumental conditions in Section 9 are guidelines, removable variable parameters, and adjusted suggested conditions to reflect normal conditions used, added an example to Section 13b and reworded Sections 13c, clarified 14a, and updated references
4	09/18/18	Deleted Introduction and Principle sections and renumbered. Revised Scope to describe who document applies to. Deleted equipment already described in referenced IOSS SOP, deleted 'Calibration' section and renumbered. Updated remaining sections in the document for clarity including expanding decision criteria, Updated references.

### **Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 09/17/2018

Chemistry Unit Chief:

Date: 09/17/2018

### **QA Approval**

Quality Manager:

Date: 09/17/2018

## **Pyrolysis-Gas Chromatography/Mass Spectrometry Analysis of Paints, Tapes, and Polymers**

### **1 Scope**

This procedure applies to Chemistry Unit caseworking personnel who analyze paints and polymers via pyrolysis-gas chromatography with mass spectrometry (Py-GC/MS). This document describes the sample preparation and suggested instrumental parameters for the Py-GC/MS analysis of paints, tapes, and other polymeric materials.

### **2 Equipment/Materials/Reagents**

- a. Instrumentation - Gas Chromatograph, Mass Selective Detector with EI Source, and Software (Agilent or equivalent)
- b. Autosampler - Pyrolysis Autosampler, accessories, and software (Frontier, or equivalent)
- c. Sample holder: alloyed metal cups (Frontier or equivalent)
- d. Cleaning and preparation apparatus for sample holders (e.g., small butane torch, sample cup inspector, sample cup holder)
- e. Polymeric reference materials (Scientific Polymer Products, Inc. or equivalent)
- f. Stereo-microscope (~ 6 to ~ 50x) with appropriate lighting
- g. Scalpel handle with blades
- h. Wire probe
- i. Tweezers
- j. Glass microscope slides
- k. Analytical microbalance (optional)

### **3 Standards and Controls**

#### **3.1 Standards**

Manufacturer-supplied and commercially available paints, tapes, polymers, adhesives, and sealants are maintained in reference collections within the FBI Laboratory. These materials are

used in casework in accordance with the *Chemistry Unit Procedures for the Use of Reference Materials and Known Materials*.

### 3.2 Performance Checks

Refer to the *Performance Monitoring Protocol (QA/QC) for the Pyrolysis-GC/MS (Py-GC/MS)* for details on the performance checks and necessary supplies to conduct these checks and operate the instrument.

## 4 Sampling

Refer to the current version of the relevant material's *General Approach Paints and Polymers Standard Operating Procedure (P&P SOP)* (e.g., PPSU100, PPSU 101, PPSU 102) for guidance as to how samples are selected for analysis and comparison. Record the sample(s) selected for analysis in the case notes.

## 5 Procedure

- 1) Using a scalpel or similar tool, prepare a clean<sup>1</sup> sample of material large enough (approximately 50 µg) to provide an adequate signal. The amount can vary depending on instrument sensitivity and chemical composition of the material (e.g., amount of inorganic filler, type of binder).
  - a. Separate individual components (e.g., paint layers, backings, adhesives) by taking thin peels with a scalpel.
  - b. Multi-layer samples can be analyzed if it is not possible to separate layers.
  - c. If appropriate, sample dried material (e.g., cured spray paint on the nozzle) from the container of an uncured specimen. Alternatively, a portion of an uncured sample (e.g., glues, two-part adhesive systems, liquid paint) can be mixed, applied to a clean microscope slide or other suitable substrate, and permitted to dry/harden according to the manufacturer's recommendations.
- 2) Place the sample in an appropriate container for analysis. Proper and consistent placement of the sample within the base of the sample cup is critical for complete pyrolysis and reproducibility of data.
- 3) Verify that the daily and monthly performance monitoring procedures have been conducted and appropriately recorded in the relevant case notes. See the instrument SOP

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<sup>1</sup> "Clean" is defined as either 1) an inner core sample; 2) a sample cleaned with an appropriate solvent; or 3) a sample void of contamination from components that exist within the same environment.



entitled Performance Monitoring (QA/QC) Protocol for the Pyrolysis-GC/MS (Py-GC/MS) for specific details.

- 4) Analyze the performance standard, sample(s), and blanks by Py-GC/MS using the suggested instrumental parameters below. At a minimum, one blank is placed before and after each standard, sample, or reference material analyzed.
- 5) If identification of the pyrolyzates is warranted, known standards can be analyzed under the same instrumental conditions used for sample analysis.
- 6) Evaluate the data using the decision criteria below.

## 6 Instrumental Conditions

The following instrumental conditions are a guide for all standards and samples described in this SOP, and as such, are set within the instrument method but are not necessarily exact values. The method used is retained with the case record (e.g., printed and stored in the casenotes).

### Pyrolysis GC/MS(EI):

#### Pyrolysis Autosampler:

Interface Set Point: 300°C  
Furnace Temp: 600°C  
Furnace Hold Time: 0.20 min

#### GC Oven:

Initial Temp: 50°C  
Initial Time: 2 minutes  
Ramp Rate: 13°C/minute  
Final Temp: 325 °C  
Final Hold Time: 15 minutes  
Run Time: 38.154 minutes

#### GC Inlet:

Mode: Split  
Split Ratio: 50:1  
Inlet Temp: 300°C  
Split Flow: 35 mL/min  
Carrier: Helium  
Init Flow: 0.7 mL/min  
Flow Mode: Constant flow  
Septum purge: 3mL/min

#### GC Column:

Type: HP-5 or equivalent  
Length: 40 m  
Diameter: 250 µm  
Film Thickness: 0.25 µm

#### MS Parameters:

Source Temp: 230°C  
Transfer Line Temp: 300 °C  
Scan Mode: Full Scan

Ionization Mode: Electron Impact  
Scan Range: 34-650 m/z

## 7 Decision Criteria

The following criteria are used as a guide in determining the acceptability of the data produced in this procedure. Retention time, peak shape, relative intensity, and the presence/absence of corresponding diagnostic peaks are all evaluated. Examples of diagnostic peaks would be those that aid in the classification or identification of the polymer based on the components present.

### 7.1 Blanks

The blank run before each sample is evaluated for the presence of carry-over peaks from the previous sample. It is also evaluated for system peaks that could contribute to a sample's signal, possibly hindering appropriate evaluation of true peaks that are present. Ideally, the blank preceding the sample should not exhibit any chromatographic peaks greater than the CO<sub>2</sub> response. If extraneous peaks are noted, document that the peak was considered as well as the results of the evaluation.

### 7.2 Samples

- a. For characterizations of samples, evaluate the data (pyrograms and mass spectra) for unknown samples with respect to a known reference material or mass spectral library.
- b. For comparisons of samples, compare pyrograms of two or more specimens side-by-side or using overlays. Compare the retention times and corresponding mass spectral data.
- c. As applicable, assess heterogeneity, sample size, or reproducibility of the pyrolysis process through the analysis of replicates.
- d. The presence of additional peaks could be inherent differences between samples, from contamination or carry-over, or system peaks (e.g., siloxanes). Document that the peak was considered and the possible explanations for why those peaks were present. If the additional peaks are explainable as contamination, carry-over, or system peaks, then they are not exclusionary differences.
- e. Differences between samples being inter-compared are considered to be exclusionary if they indicate dissimilar compositional components or dissimilar concentrations of components that can be indicative of formulation differences. An exclusionary difference is defined as a difference in feature or property between compared items that is substantial enough to conclude that they did not come from the same source. In that regard, an example of an exclusionary difference would be a strong signal for a single phthalate in one sample vs a mixture of phthalates in another sample.

## 8 Calculations

Not applicable.

## 9 Measurement Uncertainty

Not applicable.

## 10 Limitations

- a. Pyrolysis-gas chromatography/mass spectrometry is a destructive analytical technique.
- b. Sample size or condition may preclude examination by this technique.
- c. The information obtained from the analysis of combined layers of a multi-layer paint chip will hinder the ability to discriminate between the individual layers.

## 11 Precautionary Statements

- a. As with any procedure involving trace evidence, ensure actions minimize the potential for loss or contamination of the sample.
- b. When conducting comparative analysis of two or more samples, care must be taken to ensure that each specimen to be compared is present in approximately the same amount. Unequal sampling can result in relative intensity differences within this analytical technique.
- c. In a multi-layer material, care must be taken to ensure that each specimen is free of contribution from unwanted adjacent layers.
- d. Care must be taken during sample preparation to ensure consistency of sample placement within the bottom of the sample vessel.

## 12 Safety

Use standard precautions for the handling of potentially biohazardous materials, chemicals, or sharps. Refer to the FBI Laboratory Safety Manual and appropriate Safety Data Sheet(s) for further details.

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Rev. #	Issue Date	History
4	09/18/18	Deleted Introduction, renumbered, and modified Scope to describe who document applies to. Removed equipment already described in referenced IOSS SOP and deleted 'Calibration' section and renumbered. Updated remaining sections in the document for clarity including expanding decision criteria. Updated references.
5	11/15/19	Editorial changes throughout; Section 5 – Added a procedural step and clarity to other steps Section 6 – Edited instrumental conditions Section 7 – Modified Decision Criteria to describe how blanks and samples are evaluated and documented Section 10 – Edited limitations

**Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 11/14/2019

Chemistry Unit Chief:

Date: 11/14/2019

## SEM Analysis of Paints, Tapes, and Polymers

### 1 Scope

This procedure applies to Chemistry Unit caseworking personnel who analyze paints and polymers via Scanning Electron Microscopy/Energy Dispersive Spectroscopy (SEM/EDS). This document describes the sample preparation and suggested instrumental parameters for the SEM analysis of paints, tapes, and other polymeric materials. In this document, the term SEM refers to the SEM imaging system as well as the Energy Dispersive X-ray Spectrometer (EDS).

### 2 Equipment/Materials/Reagents

- a. Scanning Electron Microscope with Backscattered Electron (BE) detector: JEOL model JSM 6510LV, TESCAN model Vega 3 XMU (or equivalent)
- b. Energy Dispersive X-ray Spectrometer (EDAX Apollo or equivalent)
- c. Spectral Library Identification and Classification Explorer (SLICE) (xk, Incorporated)
- d. Energy Dispersive X-ray processing software (EDAX Genesis or equivalent)
- e. Stereomicroscope (~ 6X to ~ 100X) with appropriate lighting
- f. Glass microscope slides
- g. Compressed gas duster
- h. Acetone (Reagent grade)
- i. Distilled water
- j. Cotton tipped applicators
- k. Double-sided adhesive tape, clear/colorless or carbon
- l. Graphite paint (Ted Pella, Inc. or equivalent)
- m. Embedding molds (Ted Pella, Inc. or equivalent)
- n. Epoxy resin and hardener (Buehler EPO-KWIK<sup>®</sup> or equivalent). For EPO-KWIK<sup>®</sup>, mix 5 parts resin to 1 part hardener and blend gently but thoroughly with a stir stick. The pot life of the mixture is ~5 minutes. The epoxy will cure at room temperature; however, curing in a moderate oven (~65°C) for two or more hours is preferable.
- o. Oven with temperature capability ~ 100°F -150°F (40°C -65°C)
- p. Analytical balance (up to 50 grams)



- q. Microtome (Leica Ultracut UCT or equivalent)
- r. Carbon coater (Cressington 108carbon or equivalent)
- s. Carbon rods (SPI Supplies or equivalent)
- t. Scalpel handle with blades
- u. Pyrolytic carbon planchets (Ernest F. Fullam, Inc. or equivalent)
- v. Adhesive (e.g., Durotak 387-2287 (National Starch) or equivalent)
- w. Wood applicator sticks

### 3 Standards and Controls

#### 3.1 Standards

Manufacturer-supplied and commercially available paints, tapes, and polymers are maintained in reference collections within the FBI Laboratory. These materials are used in casework in accordance with the *Procedures for the Use of Reference Materials and Known Materials*.

#### 3.2 Performance Check

Refer to the *Performance Monitoring Protocol (QA/QC) Scanning Electron Microscope (SEM)-Energy Dispersive X-ray Spectrometer (EDS)* for details on the performance checks and necessary supplies to conduct the check and operate the instrument.

### 4 Sample Selection

Refer to the current version of the relevant material's *General Approach* Paints and Polymers Standard Operating Procedure (P&P SOP) (i.e., PPSU 100, PPSU 101, PPSU 102) for guidelines on sample(s) selection. Record the sample(s) selected for analysis in the case notes.

### 5 Procedure

#### 5.1 Sample Preparation

1. The choice of a specific method for sample preparation will depend on the size and condition of the specimen. As needed, use multiple preparation methods in order to determine all sample characteristics. For an accurate comparison of elemental composition and structure, samples must be prepared in as similar a manner as possible.
2. Samples are first examined with a stereomicroscope. If extraneous (contaminant) materials are present, remove using the tip of a scalpel blade, by taking a series of thin peels, or with a cotton-tipped applicator moistened with water or a suitable solvent. When

extraneous materials cannot be removed, note their location during light microscopy or backscatter electron SEM observations and avoid these areas during subsequent SEM analysis.

3. Attach small samples or shavings directly to a pyrolytic carbon planchet using double-sided tape or a thin adhesive layer. Attach a tape backing to the planchet using its own adhesive. If the backing has been separated from the adhesive or if a cross section of the backing has been prepared, mount these using double-sided tape or a thin adhesive layer. Remove adhesives from the tape backing and spread into a thin layer of uniform thickness directly onto a planchet.
4. A paint smear is composed of commingled particles and fragments. Select particles that are approximately 50  $\mu\text{m}$  individually and attach to a carbon planchet for analysis. It is also possible to lift a collection of deposited particles with a sticky material, such as tape adhesive, and attach them to a carbon planchet. Individually analyze such particles.
5. There are a number of sample preparation methods available to expose individual layers in a multilayered sample. Affix a manual cross section, an intact chip oriented on edge, or thin peels of the individual layers to a carbon planchet with double-sided tape or a thin adhesive layer. Alternatively, expose the individual layers in a "stair step" fashion by cutting through and removing the overlying layer(s). Continue carving until a large, flat surface area of each layer is exposed and then affix the sample to a carbon planchet. Alternatively, embed multilayered samples such as paints and tape backings in a medium that hardens and then expose the cross section by microtomy. This technique is described in further detail in steps 6 and 7.
6. Embedment provides mechanical support for subsequent sample preparation. Begin by attaching the sample to the bottom of a mold with a thin adhesive layer. Position the sample in the mold to reveal the structures of interest when subsequently cross sectioned. A sample identification label can also be placed in the mold. Add the embedding medium, such as two-part liquid epoxy, to the mold slowly so as to prevent air bubble entrapment and allow it to cure.
7. Once embedded, cross-section the samples by hand or by microtomy, to produce a flat block and/or thin cross-sections of the sample. Trim the block face to an area of approximately 2 x 3 mm with a pyramidal shape using a jeweler's saw and/or a razor blade. For microtomy, clamp the block into a holder that is attached to the microtome arm. Adjust parameters such as cutting speed, cutting thickness, and knife angle to optimize the resulting sample. Use the knife to trim the block face first with rough cuts followed by fine cuts. If sections are desired, remove these from the knife face; if a faced block is desired, remove the block from the holder and processed for analysis.
8. Apply a conductive layer (e.g., carbon) to the sample surface of polymeric materials in order to minimize sample charging. Place carbon rods in the electrodes of the vacuum evaporator, place the sample on the base plate, and cycle the evaporator to high vacuum. Current is induced through the carbon rods in order to evaporate the carbon onto the sample. Then, the chamber is pressurized and the sample removed.

9. When analyzing multiple samples, construct a map identifying sample location on or within the sample holder.

## 5.2 Analytical Procedures

1. Structural imaging:
  - a. Light microscopy demonstrates layer structure as well as some structural detail within each layer of a multilayered sample when examining either a thin cross-section or the prepared block.
  - b. Collect a backscatter electron image when elucidating layers and structures, and/or for defining distribution of particulate components.
  - c. When collecting SEM micrographs, include a measuring scale or magnification scale or both. Also include a display to document which signal (e.g., back scattered electron or secondary electron) was used to produce the image.
2. Collection of a “bulk” EDS spectrum permits determination and comparison of average elemental compositions of a material. Collect a bulk spectrum after the raster area of the SEM is selected to yield the largest sample area possible. If it is not possible to select one large area, several small areas are analyzed, and the data from each are summed.
3. Particle analysis is performed when bulk analysis alone is insufficient to discern adequate structural and compositional discrimination of select components (e.g., aluminum flakes, decorative flakes,  $\text{CaCO}_3$ ,  $\text{BaSO}_4$ ). Perform a particle analysis by directing the electron beam of the SEM directly onto the structure of interest, either by increasing the magnification or placing the scan generator in “spot” mode. Generally, the beam current is decreased to minimize the interaction volume.
4. Once an X-ray spectrum is collected, perform a spectral peak identification in order to determine the elements present.
  - a. Spectral peak identification is best achieved through SLICE. The algorithms used for peak identification consider factors such as escape peaks, sum peaks, peak overlap, and X-ray line families.
  - b. Regardless of which automatic element identification application is utilized, peak identification must be confirmed by the examiner by superimposing and scaling KLM reference lines on the spectrum and/or referring to published tables.
  - c. The presence of an element is considered unequivocal only when a distinctive set of lines is produced, or when a single peak occurs at an energy where it cannot be mistaken for another element or artifact. The peak(s) are labeled with the corresponding elemental symbol.

- d. 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 (an)other element(s). When identification is probable, but not unequivocal, the elemental symbol is parenthesized.
5. Direct spectral comparisons can be achieved using SLICE. The compositional similarity of questioned materials to reference materials can also be performed using SLICE.

## 6 Instrumental Conditions

The following operating conditions are meant as general guidelines for starting conditions. Actual requirements can vary as determined by specific analytical needs.

Beam voltage:	20-25 kV
Beam current:	adjusted to yield at least 5000 CPS
Live counting time:	100 - 200 seconds
Amp Time:	6.4 $\mu$ S
Working distance:	13-18 mm
Take off angle:	~35-40°

Generally, changes in the suggested instrumental conditions, listed above, are required under the following circumstances:

- a. The beam voltage is increased when higher energy X-ray line excitation is required.
- b. The beam voltage is decreased when greater spatial resolution is required.
- c. The EDS detector-to-sample distance is reduced to increase X-ray collection efficiency.
- d. The spectral energy display scale is expanded when sufficient detail is not evident.

## 7 Decision Criteria

- a. Differences between samples being inter-compared or compared to a reference material or library are considered to be true differences in compositional components if those differences are determined to be meaningful. A meaningful difference is defined as a feature or property of a sample that does not fall within the variation exhibited by the comparison sample, considering the limitations of the sample or technique, and therefore indicates the two samples do not share a common origin.
- b. These differences can be qualitative or quantitative. Meaningful qualitative differences can consist of the absence of trace amounts of rarely occurring elements or the absence of a significant peak from a more commonly occurring element. Quantitative differences can consist of meaningful ratio differences in the peak heights of major and minor elements.

- c. Spectra are considered *generally similar* if they exhibit slight differences. These differences can be qualitative or quantitative. Permissible qualitative differences in materials considered to be generally similar can consist of the absence of trace amounts of common elements. Permissible quantitative differences can consist of slight ratio differences in the peaks heights of major or minor elements.
- d. Spectra are considered *indistinguishable* if channel-to-channel intensity variations are the only differences observed.

## 8 Calculations

Not applicable.

## 9 Measurement Uncertainty

Not applicable.

## 10 Limitations

The methods described in this guide can have some limitations, including the inability to detect elements in trace concentrations, the need for a conductive coating on the sample, and the discoloration of materials by irradiation.

- a. The information available from a specimen can diminish as its size is reduced and its condition degrades. As specimen size is reduced or the material becomes degraded, it may no longer be representative of the original material.
- b. A disadvantage of embedment is the inability to remove a sample from most embedding materials after analysis.
- c. Although the natural X-ray line width is approximately 2 eV, EDS resolution is generally no better than approximately 140 eV. As a result, overlap of peaks in the EDS spectrum of materials containing several elements can occur. The following are some commonly occurring overlaps encountered in EDS: TiK $\alpha$ /VK $\alpha$ , VK $\beta$ /CrK $\alpha$ , CrK $\beta$ /MnK $\alpha$ , MnK $\beta$ /FeK $\alpha$ , FeK $\beta$ /CoK $\alpha$ , PbM $\alpha$ /SK $\alpha$ /MoL $\alpha$ , BaL $\alpha$ /TiK $\alpha$ , KK $\beta$ /CaK $\alpha$ , ZnL $\alpha$ /NaK $\alpha$ , PK $\alpha$ /ZrL $\alpha$ , and AlK $\alpha$ /BrL $\alpha$ .
- d. Any individual particle or fragment from a heterogeneous material may not be compositionally representative of the bulk and therefore would not be expected to produce spectra similar to the bulk material.

## 11 Precautionary Statements

- a. As with any procedure involving trace evidence, ensure actions minimize the potential for loss or contamination of the sample.
- b. Orientation of the sample area of interest perpendicular to the electron beam is critical for accurate and reproducible EDS results. As such, extreme care should be taken when embedding a multilayered sample or when placing an unembedded chip or manual cross section on an SEM mount.
- c. When analyzing a cross-section of a thin layer, such as a factory-applied automotive basecoat (~10 µm), care must be taken to ensure that the excitation volume does not extend into adjacent layers.
- d. With a “stair step” preparation, the excitation volume can penetrate through a thin layer to an adjacent underlying layer.
- d. If spectral differences are detected, it is likely that the materials that produced them are not similar in composition; however, several alternative explanations are possible. These include dissimilar sample geometry, heterogeneity of the sample, and X-ray contribution from extraneous material.

## 12 Safety

Use standard precautions for the handling of potentially biohazardous materials, chemicals, or sharps. Refer to the *FBI Laboratory Safety Manual* and appropriate Safety Data Sheet(s) for further details. Personal radiation monitors (dosimeters) are administered by the Health and Safety group to monitor exposure to ionizing radiation. Operators should familiarize themselves with the specific User's Guide safety section of the instrument prior to use.

## 13 References

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ASTM E2809 Standard Guide for Using Scanning Electron Microscopy/ X-Ray Spectrometry in Forensic Paint Examinations, ASTM International, West Conshohocken, PA

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Rev. #	Issue Date	History
1	03/14/12	Updated Section 2 for consistency with Section 1 as to types of samples analyzed. Also, updated what will affect the examination in Section 2. Updated instruments in Section 4. Removed “embedded and polished” in Section 5.2 when referring to manganese. Added statement to Section 7 to refer reader to other PPSU SOPs regarding sample selection. Update Section 8 to include changing relevant information from General Chemistry’s SOP for General SEM Methods and omitted all references to it, elaborated on sample preparation methods, omitted aluminum coating of tape samples to enhance fabrication markings. Updated section 9 to include adding specific number for beam current, providing range for live counting time, and adding amp time. Omitted references to pulse processor time constant, when to increase/decrease beam current, and dead time in Section 9 and relettered. Clarified when spectra are considered different and consistent in section 10. Change “of” to “on” when referring to sample in section 13. Added precautionary statements for various sample preparation methods in Section 14. Updated references in section 16.
2	02/03/14	Made minor grammatical changes and updated references.
3	09/18/18	Deleted Introduction and Principle sections and renumbered. Revised Scope to describe who document applies to. Updated Equipment Section, deleted Calibration Section and renumbered, Updated remaining sections in the document for clarity including expanding decision criteria. Updated references.

### **Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 09/17/2018

Chemistry Unit Chief:

Date: 09/17/2018

### **QA Approval**

Quality Manager:

Date: 09/17/2018

## X-Ray Diffraction Analysis of Tapes

### 1 Scope

This procedure applies to Chemistry Unit caseworking personnel who analyze paints and polymers via X-ray diffractometry (XRD). This document describes the sample preparation and suggested instrumental parameters for the XRD analysis of pressure sensitive adhesive tapes.

### 2 Equipment/Materials/Reagents

- a. X-ray Diffractometer system (e.g., PANalytical X'Pert PRO MPD, Rigaku MiniFlex, or equivalent) and accompanying search software
- b. Sample holder equipped with low background substrate
- c. Stereo microscope (~6X to ~50X) with appropriate lighting
- d. Scalpel handle with blades
- e. Glass microscope slides
- f. Tweezers
- g. Disposable wipes
- h. Cotton-tipped applicators
- i. Acetone (Reagent grade)
- j. Chloroform (Reagent grade)
- k. Hexane (Reagent grade)
- l. Methanol (Reagent grade)

### 3 Standards and Controls

#### 3.1 Standards

Manufacturer-supplied and commercially available tapes, polymers, and adhesives are maintained in reference collections within the FBI Laboratory. These materials are used in casework in accordance with the Chemistry Unit's *Procedures for the Use of Reference Materials and Known Materials*.

### 3.2 Performance Checks

Performance checks for the Trace Evidence Unit (TEU) XRD instrument are conducted by personnel in the Mineralogy Subgroup. Refer to both the instrument logbook as well as the *X-ray Powder Diffractometry Using X'Pert MPD SOP* (TEU XRD SOP) to determine if this check is current.

The performance check for the Rigaku Miniflex XRD instruments is conducted on the day of analysis according to the guidance set forth in the *Performance Monitoring Protocol (QA/QC) for the Rigaku MiniFlex X-Ray Diffractometer (XRD) SOP* (CU XRD SOP).

## 4 Sample Selection

Refer to the current version of the Paints and Polymers Standard Operating Procedure (P&P SOP) (i.e., PPSU 100, PPSU 101, PPSU 102) *General Approach for Tape Casework* for guidance on sample(s) selection. Record the samples selected for analysis in the case notes.

## 5 Procedure

1. Sample preparation will depend on sample type, size, and condition. If the sample is contaminated or too limited in size, record as such in the case notes. All tapes are generally analyzed intact. Additionally, the film backing can be analyzed separately.
  - a. Prior to analysis, clean the tape backings using a cotton swab or disposable wipe and as necessary, an appropriate solvent (e.g., methanol, acetone).
  - b. To analyze an intact specimen, obtain a sample area large enough to fill the sample holder. Using the sample holder as a template, cut a sample of the specimen with a scalpel blade. If the specimen is a partial roll of tape, the roll should be unwound approximately three to four inches before sampling in order to eliminate stretching or distorting the film.
  - c. The same sample preparation should be used for the analysis of the tape backing. The adhesive and scrim fabric, if present, is removed using a suitable solvent (e.g., hexane or chloroform for rubber-based adhesives, acetone for acrylic-based adhesives).
  - d. Place the specimens on the sample holder in a manner that results in a flat, uniform surface for X-ray beam interaction. For example, specimens containing adhesive can be affixed directly onto the XRD sample holder. When only the film backing is analyzed, a small amount of adhesive can be applied to affix the sample to the sample holder outside the sampling area where the X-ray beam impinges.
2. Ensure that the quality assurance procedures to include performance checks have been conducted. Refer to the instrument logbook for this information.

3. Analyze the sample(s) by XRD. Note: depending upon the instrument used, manually adjust operating conditions if they are not automatically set when selecting an analysis method. Further, as applicable, ensure that the appropriate slits and beam mask are inserted prior to beginning an analysis. For specific operating conditions, refer to the instrument operator's manual associated with the instrument being used.
4. If the compositions of two specimens are to be compared, analyze them both under the same instrumental conditions.
5. Ensure the instrument identification and the operating parameters are recorded on the printed spectra or elsewhere in the case notes.
6. If phase identification in the resulting diffraction pattern is necessary, use the Powder Diffraction File, or analyze an appropriate standard under the same operating conditions as the unknown sample.
7. Upon completion of the analysis, remove all samples from the sample chamber and, if necessary, return the instrument to standby conditions.

## 6 Instrumental Conditions

**6.1** Suggested operating conditions for the PANalytical X'Pert PRO MPD XRD are listed in the TEU XRD SOP. The following instrumental conditions serve as a guide for analysis of tape and adhesive standards and samples described in this SOP:

### Generator Settings:

Standby: 40 kV 10 mA  
Analysis: 45 kV 40 mA  
Anode material: Copper (Cu)  
Filter type: Nickel (Ni)

### Incident Beam Path:

Soller slits: 0.04 rad.  
Fixed divergence slit:  $\frac{1}{4}^{\circ}$   
Fixed anti-scatter slit:  $\frac{1}{2}^{\circ}$   
Fixed beam mask: 10 mm

Scan range:  $8 - 80^{\circ} 2\theta$   
Step size:  $0.0170^{\circ} 2\theta$  (~8 mins/sample)  
Scan type: Continuous

**6.2** Suggested operating conditions for the Rigaku MiniFlex XRD are listed in the CU XRD SOP. The following instrumental conditions serve as a guide for analysis of tape and adhesive standards and samples described in this SOP:

Anode material: Copper (Cu)  
Scan range:  $5 - 75^{\circ} 2\theta$   
Scan width:  $0.0200^{\circ} 2\theta$   
Scan speed: 2.000 degrees/min (slow scan, ~ 15mins/sample)  
Scan type: Continuous

## 7 Decision Criteria

- a. If differences are observed between the diffraction patterns of two (or more) samples being compared, then it is concluded that the specimens differ from one another.
- b. If no differences are observed between the diffraction patterns of two (or more) samples being compared, then it is concluded that they are indistinguishable from one another.
- c. If XRD is being used to characterize a material, the diffraction pattern should compare favorably to a corresponding reference powder diffraction file or known material analyzed in-house.
- d. Proper sample preparation is critical for XRD analysis. The following are some common sample preparation problems encountered in XRD analysis:
  - i. The displacement of the surface of the specimen away from the diffraction axis is the primary source of error in the measurement of diffraction peak positions. The sample must be spread flat on the specimen holder without extending above or below the instrument's diffraction axis.
  - ii. Any sample preparation technique should minimize introducing stress and orientation effects ("preferred" orientation) while maximizing a random distribution of crystallites with a uniform particle size (1-50  $\mu\text{m}$ ).

## 8 Calculations

Not applicable.

## 9 Measurement Uncertainty

Not applicable.

## 10 Limitations

- a. On average, the lower limit of detection of a component in a mixture is approximately 1%. This limit will vary depending on composition, degree of crystallinity, and crystallite size.
- b. Limited sample size can preclude analysis by this technique.
- c. Contamination of the adhesive is not always readily visible under macroscopical or microscopical examination, yet can affect a comparative analysis with a pristine sample.

- d. Accurate or reproducible results can be affected when one or more of the following circumstances occur:
  - i. The specimen is not homogeneous.
  - ii. Crystallites are not randomly oriented; “preferred” orientation or stress exists within the specimen.
  - iii. The specimen is amorphous.
  - iv. The specimen contains variable or mixed, hydrated forms where each form yields a distinct diffraction pattern.

## 11 Precautionary Statements

- a. As with any procedure involving trace evidence, ensure actions minimize the potential for loss or contamination of the sample.
- b. If two (or more) components in a sample yield a similar diffraction pattern, the peaks can overlap one another; therefore, comparing the composite pattern with known powder diffraction patterns of individual compounds can be difficult.
- c. The elasticity inherent in some polymers can cause the flattened backing sample to curl up if insufficient mounting media is used to hold the sample flat and stationary.

## 12 Safety

Take standard precautions for the handling of potentially biohazardous materials, chemicals, or sharps. Refer to the *FBI Laboratory Safety Manual* and appropriate Safety Data Sheet(s) for further details. Personal radiation monitors (dosimeters) are administered by the Health and Safety group to monitor exposure to ionizing radiation. Operators should familiarize themselves with the specific User’s Guide safety section of the instrument prior to use.

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Rev. #	Issue Date	History
0	05/05/06	New document that replaces previous document also titled <i>XRD Analysis of Tapes</i> .
1	09/30/09	Updated sampling section and references.
2	03/14/12	Corrected chemical name of corundum in section 4d. Changed “sampling” plan to “sample selection” plan in section 7. Removed reference to contacting TEU’s Mineralogy Subgroup in section 8, part 3. Updated decision criteria in sections 10a and 10b. Changed macroscopic/microscopic to macroscopical/microscopical as well as clarified sample condition in section 13c. Updated references in section 16.
3	02/11/13	Updated section 3. Added more detail to section 8, step 4 to clarify that operating conditions are not automatically set with the analytical method selected.
4	02/03/14	Made minor changes to equipment list to make item descriptions less specific; minor change to specify that performance check monitoring is evaluated within QA review prior to analysis, and updated references.
5	09/18/18	Modified scope to align with LOM revisions; deleted equipment or performance monitoring already contained in instrumental QA/QC SOPs; added flexibility to accommodate broader instrument use; minor grammatical changes throughout; aligned safety section with other P&P SOPs.

Redacted - Signatures on File

### **Approval**

Paints and Polymers  
Technical Leader:

Date: 09/17/2018

Chemistry Unit Chief:

Date: 09/17/2018

### **QA Approval**

Quality Manager:

Date: 09/17/2018



## **Raman Analysis of Paints and Polymers**

### **1 Scope**

This document applies to Chemistry Unit caseworking personnel who analyze paints and polymers via Raman spectroscopy. This document describes the sample preparation and suggested instrumental parameters for the Raman analysis of paints and other polymeric materials.

### **2 Equipment/Materials/Reagents**

- a. Raman spectrometer with microscope attachment (Horiba or other manufacturer)
- b. Lasers installed in spectrophotometer: 785 nm and 532 nm; others if applicable
- c. Objectives for spectrophotometer microscope attachment: 10x, 100x; others as applicable
- d. Spectral search library (e.g., Know-It-All by Bio-Rad or equivalent)
- e. Stereomicroscope (~6X to ~100X) with appropriate lighting (e.g., annular ring light, fiber optic light)
- f. Polystyrene film
- g. Tweezers
- h. Scalpel handle with blades
- i. Microscope slide(s) (e.g., aluminum, gold, quartz)
- j. Disposable paper wipes

### **3 Standards and Controls**

#### **3.1 Standards**

Manufacturer-supplied and commercially available paints, pigments, polymers, or tapes are maintained in reference collections within the FBI Laboratory. These materials are used in casework in accordance with the *Procedures for the Use of Reference Materials and Known Materials*.

### 3.2 Performance Checks

Refer to the *Performance Monitoring Protocol (QA/QC) for the Raman Spectrometers* for details on the performance checks and necessary supplies to conduct these checks and operate the instrument.

Additionally, polystyrene is analyzed prior to sample analysis on a given day using the following parameters: the 532 nm laser with the 1800 lines/mm grating, 100X objective, the number of acquisitions as 10, and a collection time of 10 seconds. The Decision Criteria for acceptable peak positions for polystyrene are also listed in the *Performance Monitoring Protocol (QA/QC) for the Raman Spectrometers* SOP. This spectrum should be printed and placed in the instrument logbook with the option to also print it for the casefile as applicable.

## 4 Sampling

Refer to the current version of the relevant material's *General Approach* Paints and Polymers Standard Operating Procedures (P&P SOP) (e.g., PPSU 100, PPSU 101, PPSU 102) for guidance on sampling. Record the samples selected for analysis in the case notes.

## 5 Procedure

Cease comparison examinations whenever a test reveals an exclusionary difference between two (or more) samples being compared. Record any factors limiting the analysis (e.g., sample size, condition) in the case notes.

1. Turn on the laser and allow to warm up for approximately 20 minutes prior to use.
2. Perform the daily performance monitoring procedure. See the Instrument's *Performance Monitoring (QA/QC) Protocol for the Raman Spectrometers* for specific details.
3. Sample preparation will depend on sample type, size, and condition. Where possible take all samples from a clean, core area (e.g., cut into the sample and discard the top portion, or clean the surface). If appropriate, sample dried material (e.g., cured spray paint on the nozzle) from the container of an uncured specimen. Alternatively, mix an uncured sample (glues, two-part adhesive systems, liquid paint), apply an aliquot to a clean microscope slide or other suitable substrate, and permit it to dry/harden according to the manufacturer's recommendations.

Analyze samples directly or use a preparation method to obtain a sample suitable for analysis.

- a. **Paint:**  
Most samples are analyzed as is, but multi-layered specimens can also be prepared as thin peels of individual layers. Thin peels can be achieved by cutting through individual layers with an angled scalpel blade or similar tool. Create cross-sections manually or with a microtome.
4. Place an aluminum microscope slide or other appropriate sample holder containing the sample onto the microscope stage. Adjust the compensator to focus the sample. View the sample of interest through the microscope oculars. When in focus, the sample easily fills the field of view (~10 microns). Using the video option, focus the laser beam onto the sample. Collect a spectrum using the instrumental conditions listed in section 6. Repeat the measurement using a different area of the sample, or after changing instrumental conditions (e.g., laser type, laser power) as needed to achieve the best, reproducible signal.
5. Save the spectrum.
6. Use spectral search software (e.g., KnowItAll) to compare the paint, polymer, or tape against the reference samples maintained within the databases.
7. Record use in the instrument-specific binder. After last use on a given day, shut off the laser and close out of the software.

## 6 Instrumental Conditions

The following instrumental conditions are a guide for all standards and samples described in this SOP, and as such, are set within the instrument method and can be adjusted as necessary:

Parameter	785nm laser	532nm laser
Detector:	CCD: Silicon	CCD: Silicon
Laser power:	10%	10%
Grating (g/m):	1800 max	2400 max
Spectral range (cm <sup>-1</sup> ):	3200 – 200	3200 – 250
Number of acquisitions:	5	5
Collection time:	10 sec	10 sec

Generally, changes in the suggested instrumental conditions, listed above, are required under the following circumstances:

- a. Bleaching time can be increased to mitigate any observed fluorescence.
- b. The number of acquisitions or collection time can be increased to reduce noise.

- c. The laser power can be increased if intensity is weak (e.g., below 800 counts/s).
- d. If the intensity is too high (e.g., sample burning, increased fluorescence), the laser power can be reduced, the laser can be changed (e.g., green to red), or the slit or hole can be decreased.

Conditions for processing of spectra prior to use in the KnowItAll software:

Parameter	Baseline Correction	Smoothing and Filtering
Type:	Line	deNoise
Degree:	20	33
Max Points:	256	
Noise Points:	64	
Size:		8

## 7 Decision Criteria

In general, characterization should be based on a comparison of the spectral data of the resulting peaks with data from a contemporaneously analyzed reference material. In situations where a reference material is not available for comparison, major components are still able to be determined.

- a. If exclusionary differences are observed between the spectra of two (or more) samples being compared (e.g., the presence or absence of observable components), then the specimens are considered different.
- b. If no exclusionary differences are observed between the spectra of two (or more) samples being compared (e.g. agreement in the presence and relative intensity of observable components), then it is concluded that they are indistinguishable.
- c. If Raman is being used to characterize a material type, the spectrum should compare favorably to a corresponding reference or library spectrum (e.g., in-house library, reference, published in peer-reviewed format).

## 8 Calculations

Not applicable.

## 9 Measurement Uncertainty

Not applicable.

## 10 Limitations

- a. Fluorescence effects cannot always be controlled to the extent necessary for adequate spectral interpretation and comparison.
- b. The spectrum of a mixture can be difficult to interpret due to spectral overlap.
- c. The sub-generic class of polymers cannot always be determined by Raman analysis.
- d. Proper sample area selection is critical for adequate sample characterization. The following are some common sample selection problems encountered in analysis.
  - i. Binder determination can be hindered by focusing on a large pigment particle. Multiple areas of the sample should be analyzed to ensure major components are adequately recorded.
  - ii. Minor pigment constituents or smaller-sized pigments can be masked by larger particle-sized filler pigments. Re-analyze the sample using an area relatively free of larger pigment particles.
  - iii. Fluorescence is often observed in the spectrum of a sample collected using a particular laser or laser power. To reduce this effect, re-analyze the sample at a lower laser power or increase the bleaching time. Alternatively, repeat the analysis using a lower intensity laser.

## 11 Precautionary Statements

- a. As with any procedure involving trace evidence, ensure actions minimize the potential for loss or contamination of the sample.
- b. The presence of some large particle pigments (e.g., calcium carbonate) within a mixture can cause difficulty with interpretation of the resulting spectrum.

## 12 Safety

Use standard precautions for the handling of potentially biohazardous materials, chemicals, or sharps. Refer to the *FBI Laboratory Safety Manual* and appropriate Safety Data Sheet(s) for further details. Operators should familiarize themselves with the specific User's Guide safety section of the instrument prior to use.

## 13 References

*FBI Laboratory Safety Manual*

*Performance Monitoring Protocol (QA/QC) for the Raman Spectrometers*, FBI Laboratory, Chemistry Unit - Instrument Operation and Systems Support SOP

*Chemistry Unit Procedures for the Use of Reference Materials and Known Materials*, FBI Laboratory, Chemistry Unit Quality Assurance Manual

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Rev. #	Issue Date	History
0	01/10/19	Original Issue
1	02/03/20	Removed reference to Instrument manual and made changes to conform to LOM revisions; minor edits throughout.

**Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 01/31/2020

Chemistry Unit Chief:

Date: 01/31/2020



## **Visual, Microscopical, and Microchemical Examination of Paint and Coating Evidence**

### **1 Scope**

This procedure applies to Chemistry Unit caseworking personnel who perform visual, microscopical, and solubility/microchemical examinations that are used to characterize and compare a variety of paint and coating specimens.

### **2 Equipment/Materials/Reagents**

- a. Stereo microscope (~6X to ~100X) with two lighting conditions (e.g., ring light oriented ~180° from sample, fiber optic light oriented ~45° from sample)
- b. Compound microscope with bright field and polarizing light sources
- c. Glass microscope slides
- d. Scalpel handle with blades
- e. Tweezers
- f. Probes (e.g., steel, tungsten, wood, Teflon<sup>®</sup>)
- g. Disposable wipes
- h. Well slides
- i. Pillboxes
- j. Compressed gas duster
- k. Large, flat-blade spatula
- l. Large sheets of untreated kraft paper (or equivalent)
- m. Spot plates
- n. Acetone (Reagent grade)
- o. Xylene (Reagent grade)

- p. Chloroform (Reagent grade)
- q. Eyedropper bottles and/or disposable pipettes
- r. Diphenylamine (DPA) (Reagent grade)
- s. Sulfuric acid (Reagent grade)
- t. Acetic acid (Reagent grade)
- u. DPA solution  
Dissolve 0.3 g of DPA in 20 mL concentrated sulfuric acid and then slowly add 10 mL glacial acetic acid. Store the solution at room temperature in a labeled brown glass bottle. The solution will be stable for at least 1 year and can be tested on a positive control sample to determine effectiveness beyond the expiration date.
- v. Analytical balance
- w. Munsell Book of Color, Matte and Glossy Collections available from the Macbeth Division of Kollmorgen Instruments Corporation, New Windsor, NY
- x. Munsell Neutral Value Scale, Matte Edition available from the Macbeth Division of Kollmorgen Instruments Corporation, New Windsor, NY
- y. Minolta CHROMA METER CR-241 Colorimeter (or equivalent)
- z. PPG RapidMatch® Color Match spectrometer tool with Paint Manager® library (or equivalent)
- aa. Digital camera
- bb. Digital microscope

### 3 Standards and Controls

These materials are stable in a laboratory setting and do not expire.

- a. Positive Enamel:  
Enamel paint standard, such as NAPF PID 6320.
- b. Positive Dispersion Lacquer:  
Dispersion lacquer paint standard, such as NAPF PID 1910.

- c. **Positive Acrylic Solution Lacquer:**  
Acrylic solution lacquer paint standard, such NAPF PID 415.
- d. **Positive Nitrocellulose Lacquer:**  
A commercially available colored nail polish with nitrocellulose listed as a main ingredient can be used. A sample should be spread out in a thin layer on a clean glass microscope slide using the product's applicator. Allow the film to dry prior to use as a standard. Store the bottle according to manufacturer's recommendations.
- e. **Negative Control:**  
As it does not react with any of the solvents or chemicals used in the scheme, the positive enamel paint standard can be used as a negative control for the other solvents/microchemical tests.

## 4 Sampling

Refer to the Paints and Polymers Standard Operating Procedure (P&P SOP) *General Approach for Paint and Coating Casework* for guidance for sample(s) selection. Record the samples selected for analysis in the case notes.

## 5 Procedure

### 5.1 Visual and Microscopical Examination

Use written descriptions, sketches, photography, or other imaging methods to capture both visual and microscopical characteristics and observations. If the items are suitable for further examination, record a detailed description of each item to include comparative features or any unusual conditions (e.g., commingled material).

- 1. Process each item separately to prevent cross-contamination
- 2. Transfer the item from its original container to a suitable substrate (e.g., paper, glass microscope slide, pillbox) to examine both visually and microscopically. Some specimens require processing or preparation prior to examination as described below.
  - a. **Clothing:** Examine each article of clothing visually and microscopically for evidence of a contact paint transfer.
    - i. If a potential paint transfer is embedded or abraded onto the fabric, take a cutting which includes a representative portion of the transferred substance and preserve it for future examination. See 2.b. for further instructions regarding smears.

- ii. Process each article of clothing as it was received (i.e., individually or collectively packaged) and isolate the debris in the same manner (i.e., one pillbox per package).
  - iii. Suspend the item from a rack over a large sheet of paper and carefully scrape all surfaces in a downward motion with the edge of a large flat-bladed spatula or similar tool to dislodge any remaining paint evidence.
  - iv. Collect the deposited debris and transfer it to a pillbox or other container for microscopical examination. Label the top and bottom of the container with the laboratory number, item number, and initial. See 2.c. for further instructions regarding debris.
- b. Smears: The considerable force required to cause a paint transfer often results in the paint being abraded and damaged; the layers of a multiple-layer paint system can be mixed together or smeared across a surface.
- i. If fused or embedded onto a surface, remove particles and fragments using a scalpel blade, probe, tweezers, or similar tool while observing under a microscope. If the item will be subsequently examined for toolmark comparisons, relatively soft, pliable materials such as wood or Teflon<sup>®</sup> should be used to dislodge paint from the surface. Metal blades should not be used as they can alter the surface and thereby affect a toolmark examination.
  - ii. The fabric weave of an article of clothing can be stretched in order to facilitate removal/dislodging of particles of paint.
  - iii. Transfer isolated particles/fragments to a well slide or pillbox for future examination. Label the slide or pillbox with the laboratory number, item number, and initial.
  - iv. Smeared paint can be contaminated with material from the surface upon which it is impacted (e.g., fibers, painted substrate, wood) thereby affecting the chemistry and/or color of the sample. If appropriate, take a control sample of the substrate close to but not within the area containing the smear.
- c. Debris: Paint evidence can be observed as a mixture with other materials that are not probative for examination by P&P personnel (e.g., fibers, soil, glass).

- i. Examine the contents of the debris microscopically, manipulating it with the appropriate tools (e.g., tweezers, scalpel) and isolate any paint-like materials.
    - ii. Transfer these materials to a well slide or pillbox for future examination. Label the slide or pillbox with the laboratory number, item number, and initial.
    - iii. To decrease the likelihood that paint evidence has been overlooked, a second P&P examiner can examine the debris. Alternatively, the primary examiner should re-examine the debris on a different day. Results of these subsequent analyses are recorded in the case notes.
  - d. Liquid paint samples: If appropriate, sample dried material (e.g., cured spray paint on the nozzle) from the container of an uncured specimen. Alternatively, mix an uncured sample (e.g., liquid paint), apply it to a clean glass microscope slide or other suitable substrate as a thin film, and allow it to dry/cure according to the manufacturer's recommendation.
3. Once isolated, observe the surface of the paint and record color, presence of effect pigment(s), morphology, degree of gloss, texture, presence of surface striae, defects, weathering, or any other characteristics that aid in the description of the item.
4. If conducting a comparative examination, observe paint chips for possible physical matches such as a fracture match from broken-edge characteristics and/or surface anomalies (e.g., striae).
  - a. Fracture matches are the most conclusive type of examination. Record observed fracture matches with descriptive notes and imaging techniques.
  - b. Include a measuring scale, when practicable. If not, annotate the photograph with the magnification used to capture the image.
  - c. A second P&P examiner must confirm and record the suspected fracture matches between known and question specimens.
5. Observe the layer structure of any paint specimen(s) by viewing it at ~6X to ~100X magnification.
  - a. Obvious layers can be exposed/observed by a number of techniques which include, but are not limited to, viewing the sample on edge, cross-sectioning by hand, cross-sectioning by encapsulation and microtomy or polishing, making an oblique (bias) cut through the sample, or taking a series of thin peels through each layer.

- b. A combination of techniques can be used to fully characterize the layer structure. The extent of sample manipulation and preparation will depend on the amount of sample available, its complexity, and its characteristics.
6. Record the number and sequence of layers and their relative thicknesses.
7. For each layer, record the description of its color, texture (i.e., primer layer versus color coat, presence of inclusions), presence or absence of effect pigment (e.g., metallic flake, pearlescent, flat), and any other observations (e.g., homogeneity, uniformity of layer thickness across sample, body-filler material evident, tinted clear coat, color-coordinated primer, metal pre-treatment).
8. Microscopical examination of a thin cross-section of a multiple layer paint sample in transmitted light can also be conducted. Higher magnifications using a compound microscope can allow for better detail regarding the number of layers present and the presence/dispersion of effect and/or other pigments. In addition, the thinness of the cross-section also permits better color discrimination. The use of a polarized light source can also aid in the examination of layer structure and characterization of pigments.
9. If appropriate, conduct color measurements on comparative items.
- 9.1 The tristimulus value of an exposed surface of a paint sample can be measured using the Minolta Colorimeter. Refer to the *Performance Monitoring Protocol (QA/QC) for the Minolta CR-241 Chroma Meter* for more detail.
  - a. The surface of the sample must be at least 0.3 mm in diameter.
  - b. Samples should lay flat and possess little or no surface damage or contamination.
  - c. Measurements obtained on samples being compared need to be collected using the same measurement area (0.3 mm or 1.8 mm diameter). Record which measurement area was used. When sample size permits, three readings are recommended for the 1.8 mm diameter measurement area. A single measurement is recommended for the 0.3 mm diameter measurement area.
- 9.2 Alternatively, evaluate color designations using the Munsell Book of Color or Neutral Value Scale.
- 9.3 For automotive paint, the PPG RapidMatch® color matching spectrometer with accompanying Paint Manager® library can be used to determine automotive paint color codes if the sample is at least 3 cm x 2 cm in size.
  - a. Calibrate the RapidMatch® spectrometry tool prior to each day's use by aligning it flat onto the white surface of the calibration plate, and pressing the

measurement button on the top of the tool. Once the reading is returned on the display window, align the orange-brown metallic surface (i.e., simulated automotive paint) with the spectrometer window, and again press the measurement button. The spectrometer will take a set of three measurements in rapid succession before indicating the tool is ready for use. Record use of the tool in its QA/QC logbook according to the procedures described in section 8.4 of the *General Instrument Maintenance Protocol*.

- b. For best results, ensure that the samples lay flat and possess little or no surface damage or contamination.
  - c. Move the tool on the surface of the paint chip to collect five measurements. Once the measurements are recorded by the spectrometer, interface it to the Paint Manager<sup>®</sup> library in order to generate a hit list of manufacturer color codes that best align with the color measurements recorded by the spectrometer. Record the results of the library search.
10. In cases involving automotive paint sourcing, determine whether a paint sample is a factory-applied, original equipment manufacturer (OEM) finish or an aftermarket repaint based on the layer structure (e.g., number, sequence, order, relative thickness).
- a. If the sample is clearly an aftermarket repaint with no original layers, report the top coat color and characterization of the evidentiary paint as a non-OEM. Refer to the *General Approach to Report Writing* P&P SOP for guidance in report language.
  - b. If the sample appears to be OEM:
    - i. Refer to Step 9.1 above for guidance in recording the tristimulus value of the various layers (e.g., basecoat, ecoat).
    - ii. Alternatively, refer to Step 9.2 above to evaluate color designations using the Munsell Book of Color or Neutral Value Scale.
    - iii. For comparisons of an item's topcoat color to available reference samples, refer to Step 9.3 above to determine manufacturer and color code information or the P&P SOPs *Conducting Color Comparisons Using Automotive Refinishers Color Chips* and *Conducting Motor Vehicle Make-Model-Year Searches Using the National Automotive Paint File (NAPF) Database* for make-model-year determination of OEM automotive paint for further details.

## 5.2 Solubility and Microchemical Tests

Depending on the type of binder, these tests can be destructive or provide no additional information. Therefore, before conducting these tests, consider if the limited, general information

obtained would be probative. This evaluation should be done after binder characterization of the paint layer(s). Refer to Figure 1 in the P&P SOP *General Approach for Paint and Coating Casework* for guidance on appropriate techniques.

1. A flow chart/decision tree for the basic solubility and microchemical tests is shown in Figure 1. These tests are intended for evaluation of individual layers of an automotive finish but can be applied to industrial, architectural, and non-automotive vehicular paints if appropriate.
  - a. If the specimen is an enamel (e.g., melamine in Fourier transform infrared spectroscopy (FTIR) spectrum), solubility and microchemical tests are unnecessary.
  - b. If the specimen is a lacquer or the binder characterization is not obvious by IR, provided there is adequate sample, proceed with the solubility and microchemical tests.
  - c. When conducting solubility and microchemical reactivity tests, analyze negative and positive controls concurrently with the sample(s).
2. Separate a thin peel of each layer from adjacent layers and place the sample on a glass slide or in the well of a spot plate. Subject the layer to one or two drops of acetone and observe the reaction under low power magnification. Record the results.
  - a. If the layer dissolves, proceed to step 3.
  - b. If the layer does not dissolve in acetone, record it as an enamel. No additional solubility testing is necessary for this layer. However, in the case of a highly filled layer, it may not be readily apparent that a portion of the sample is soluble. See step 5 for further detail.
3. Subject a different thin peel of the same layer to one or two drops of xylene. Observe the reaction under low power magnification. Record the results.
  - a. If the layer dissolves, record it as a dispersion lacquer. No additional solubility testing is necessary for this layer.
  - b. If the layer does not dissolve, proceed to step 4.
4. Subject a different thin peel of the same layer to one drop of DPA solution. Observe the reaction under low power magnification. The reaction may not occur immediately, so it should be observed periodically for a period of 5 to 10 minutes before a negative conclusion is reached. Record the results.

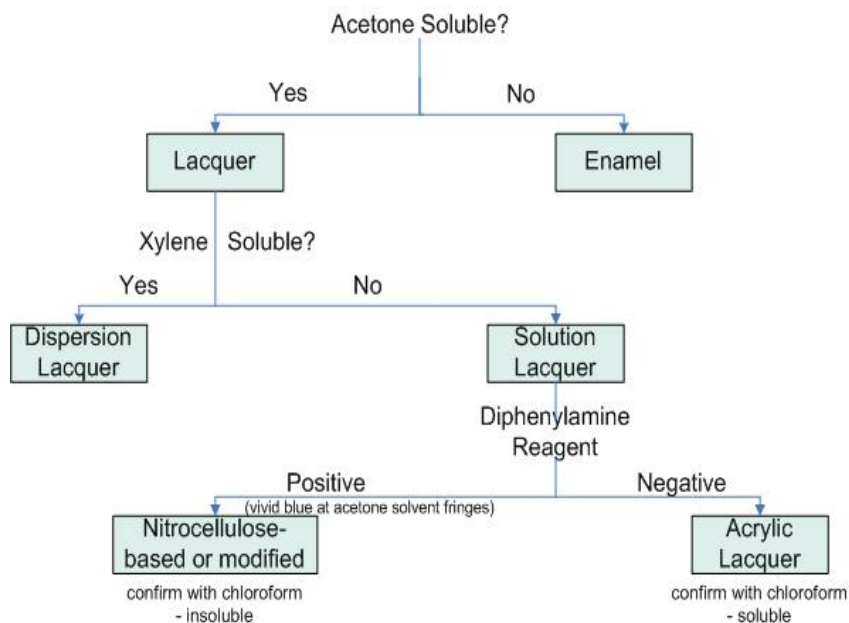


- a. A positive reaction is a vivid blue color. This is indicative of a nitrocellulose lacquer. Confirm this result by subjecting a different thin peel of the same layer to one or two drops of chloroform. A nitrocellulose lacquer will not dissolve in chloroform.

Note: Certain dyes and inorganic fillers will react with the concentrated acids in the DPA solution. Record observations such as color changes or effervescence as a point of comparison between two samples; however, these results are not a positive reaction for nitrocellulose lacquer which can be confirmed with observation of the chloroform response.

- b. If a vivid blue color is not observed, the reaction is negative. Record the layer as an acrylic lacquer. Confirm this result by subjecting a different thin peel of the same layer to one or two drops of chloroform. An acrylic lacquer will dissolve in chloroform.
5. If the layer being tested is highly filled, it may not be readily apparent that a portion of it is soluble in acetone and therefore it could be misclassified as an enamel. To ensure that a misclassification has not occurred, add one drop of DPA reagent to the thin peel that had been subjected to acetone. If it is a nitrocellulose lacquer, a vivid blue color will appear where the acetone has evaporated around the thin peel. This is indicated in Figure 1 as the acetone solvent fringes.

**Figure 1. Basic Microchemical Scheme<sup>1</sup>**



## 6 Instrumental Conditions

Refer to the *Performance Monitoring Protocol (QA/QC) for the Minolta CR-241 Chroma Meter* for instrumental conditions.

## 7 Decision Criteria

- For initial characterization, assess physical characteristics known to be exhibited by paint such as color, texture, and layer structure.
- If physical characteristics of two (or more) specimens being compared differ, cease examinations cease and report the specimens as different.
- Binder classification by the solubility and microchemical tests utilized in this procedure are described in section 5.2.

<sup>1</sup> Ryland, S.G. Infrared microspectroscopy of forensic paint evidence. Chapter 6 in *Practical Guide to Infrared Microspectroscopy*. (ed. H.J. Humecki) NY: Marcel Dekker, Inc., 1995.

## **8 Calculations**

Not applicable.

## **9 Measurement Uncertainty**

Not applicable.

## **10 Limitations**

- a. Sample size and condition can preclude conducting certain examinations, including color assessment(s).
- b. If the sample is a smear, layers can blend and contaminate one another. Microscopical examinations of layer structure, texture, number of layers, color, etc. can be affected.
- c. Paint layers less than 15 microns thick can be difficult to distinguish using standard stereo microscopical examinations.
- d. Adjacent layers similar in color and texture can be difficult to resolve using standard stereo microscopical examinations.
- e. A factory-applied, OEM automotive finish is required for a possible motor vehicle make-model-year determination.
- f. The RapidMatch<sup>®</sup> tool is intended to provide color code information for OEM automotive coating colors and may not be suitable for automotive parts that are repainted or repaired with non-OEM colors. Further, the search may not always yield results.

## **11 Precautionary Statements**

- a. As with any procedure involving trace evidence, ensure actions minimize the potential for loss or contamination.
- b. Solubility and microchemical tests can be destructive. Consider this factor when evaluating the probative value of such tests.

## 12 Safety

Use standard precautions for the handling of biohazardous materials, chemicals or sharps. Refer to the *FBI Laboratory Safety Manual* for details.

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Rev. #	Issue Date	History
0	06/21/06	New document that replaces previous document also titled <i>Macroscopic, Microscopic, and Microchemical Examination of Paint and Coating Evidence</i> .
1	09/30/09	Provide reference for sampling plan guidelines and updated references.
2	03/14/12	Updated microscopic/macroscopic to microscopical/macrosopical where appropriate throughout document. Changed “confirm” to “can aid in” in Section 3, 2 <sup>nd</sup> paragraph. Added digital microscope to section 5. Changed “sampling” plan to “sample selection” plan in section 8. Clarified title of the CU colorimeter performance monitoring SOP throughout the document. Updated references in section 17.
3	02/03/14	Changed title, changed “macroscopical” to “visual” throughout to simplify terminology, edited equipment list to be less restrictive regarding material specifications, changed examples in Section 9.1, 2c, edited Section 9.1, 2d to describe sampling of liquid paint when it’s cured on container, edited Section 9.1, 4b to expand options for displaying a scale in images, clarified criteria for measurement area in Section 9.1, subsections 9c and 10bic, edited Section 9.1, subsection 10biii to refer to appropriate SOPs not actual procedural steps in those SOPs, removed redundant first sentence in Section 9.2, and updated references.
4	09/18/18	Deleted Introduction, Principle and Specimen sections and renumbered. Modified Scope to describe who document applies to. Removed equipment already described in referenced IOSS SOP and deleted ‘Calibration’ section and renumbered. Updated remaining sections in the document for clarity. Updated references.
5	01/15/20	Added PPG RapidMatch <sup>®</sup> Color Match spectrometer tool with Paint Manager <sup>®</sup> library to equipment list; added section 9.3 to describe use of this tool and section 10f to describe some limitations of the tool. Deleted QA Manager approval per latest LOM revision. Minor edits throughout. Added a reference.

### **Approval**

Paints and Polymers  
Technical Leader:

Redacted - Signatures on File

Date: 01/14/2020

Chemistry Unit Chief

Date: 01/14/2020

## **Conducting Color Comparisons Using Automotive Refinisher Color Chips**

### **1 Introduction**

The FBI Laboratory maintains a collection of automotive paint refinisher color chips. These color standards are provided by automotive paint manufacturers and represent the original factory color available on most imported and domestic vehicles marketed in North America. Although this collection does not contain actual paint samples, it can be utilized to determine potential manufacturer(s), model(s), and year(s) information about automotive paint evidence based on its top coat color and appearance. These refinisher chips can be used in conjunction with other resources such as the National Automotive Paint File (NAPF) and Paint Data Query (PDQ).

### **2 Scope**

This procedure applies to Chemistry Unit caseworking personnel who use automotive paint refinisher color chips for motor vehicle make-model-year searches.

### **3 Equipment/Materials/Reagents**

- a. Automotive paint manufacturer refinish color standards, available as plastic chips or paper swatches (e.g., BASF, Axalta, PPG)
- b. Stereo microscope (~6X to ~100X) with two different lighting conditions (ring light oriented at ~180° and fiber optic light oriented ~45° from sample surface)

### **4 Standards and Controls**

Not applicable.

### **5 Sampling or Sample Selection**

Not applicable.



## 6 Procedure

1. Utilizing Paints and Polymers Standard Operating Procedure(s) (P&P SOP) *Visual, Microscopical, and Microchemical Examinations of Paint and Coating Evidence* and Fourier transform infrared spectroscopy (*FTIR*) *Analysis of Paints, Tapes, and Polymers*, determine if a paint specimen is a factory-applied, original equipment manufacturer's (OEM) automotive finish.
2. Using the data obtained from Paint Data Query (PDQ) searches and/or National Automotive Paint File (NAPF) searches (following P&P SOPs for *Conducting Motor Vehicle Make-Model-Year Searches Using the Paint Data Query (PDQ) Databases* and *Conducting Motor Vehicle Make-Model-Year Searches Using the National Automotive Paint File (NAPF) Database*), record any trends in automotive manufacturer, assembly plant, and/or year(s) of production.
  - a. If trends such as a particular manufacturer and/or production year exist, refer to the corresponding repaint pages for direct color comparisons.
  - b. If no trends exist, search for color chips on repaint pages for model years that coincide with the incident date(s), as well as relevant previous model years.
3. Record potential color matches from the color standards in the repaint pages and compare the color and appearance of observed candidates to the sample using a stereo microscope. For details, refer to the P&P SOP *Visual, Microscopical, and Microchemical Examination of Paint and Coating Evidence*.
  - a. Conduct color comparisons at low power magnification (~6X to ~100X) using two different lighting conditions.
  - b. Record the results of the comparisons.
4. A second qualified P&P examiner evaluates and records their results for any possible candidates that compare favorably in color and appearance.
5. If a particular color is considered a candidate, contact the manufacturer to obtain or to verify model and year information printed on the refinisher pages for a given color.

## 7 Decision Criteria

- a. If the paint refinisher standard compares favorably in color and appearance to the sample, record it as a candidate.
- b. Determine if a particular manufacturer's color is predominant.

## 8 Calculations

Not applicable.

## 9 Measurement Uncertainty

Not applicable.

## 10 Limitations

- a. A factory-applied, OEM automotive finish is required for a possible motor vehicle make-model-year determination.
- b. Color assessment and comparison can be affected by sample size and/or condition.

## 11 Precautionary Statements

- a. Paint color standards on paper or plastic substrates can differ slightly from the appearance of a specimen from an automobile.
- b. Some data entry errors may exist in the refinisher pages. Verify search results using orthogonal resources when practicable.
- c. Automotive manufacturer color names can change for a given color between model years. Refer to the paint manufacturer codes to determine if colors issued in different model years are intended to be the same color.

## 12 Safety

Not applicable.

## 13 References

BASF Customer Assistance Call Center: 800-825-3000

*Conducting Motor Vehicle Make-Model-Year Searches Using the Paint Data Query (PDQ) Databases*, FBI Laboratory, Chemistry Unit - Paints and Polymers SOP

*Conducting Motor Vehicle Make-Model-Year Searches Using the National Automotive Paint*

*File (NAPF) Database, FBI Laboratory, Chemistry Unit - Paints and Polymers SOP*

Axalta (DuPont) Refinish Group: 800-338-7668

*FTIR Analysis of Paints, Tapes, and Polymers, FBI Laboratory, Chemistry Unit - Paints and Polymers SOP*

*Visual, Microscopical, and Microchemical Examinations of Paint and Coating Evidence, FBI Laboratory, Chemistry Unit - Paints and Polymers SOP*

PPG: 800-848-2683 or, Color Library/Code/Formula: 440-572-6100

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Rev. #	Issue Date	History
0	06/21/06	New document that replaces previous document also titled <i>Conducting Color Comparisons Using Automotive Refinishers Color Chips</i> .
1	09/30/09	Clarified introduction.
2	03/14/12	Changed macroscopic and microscopic to macroscopical and microscopical as appropriate throughout document. Updated references in section 15.
3	02/03/14	Changed “macroscopical” to “visual” throughout to simplify terminology, made minor editorial changes, and added a reference
4	09/18/18	Modified scope and deleted sections to comply with LOM changes, minor grammatical changes (document to record); minor grammatical edits throughout.

### **Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 09/17/2018

Chemistry Unit Chief:

Date: 09/17/2018

### **QA Approval**

Quality Manager:

Date: 09/17/2018

## **Conducting Motor Vehicle Make-Model-Year Searches Using the Paint Data Query (PDQ) Databases**

### **1 Introduction**

Paint Data Query (PDQ) is an automotive paint database that contains information about the factory-applied topcoat and undercoat (primer) paints applied to most imported and domestic vehicles marketed in North America. It is reserved exclusively for law enforcement agencies involved in forensic investigations. As a sourcing tool, PDQ can be used in conjunction with other automotive paint collections such as refinisher color chips and the FBI Laboratory's National Automotive Paint File (NAPF).

### **2 Scope**

This general procedure applies to Chemistry Unit caseworking personnel who use the PDQ databases for motor vehicle make-model-year searches. Users receive hands-on training to learn how the database is designed, how to characterize and code various paint systems, and to gain the basic interpretive skills necessary to effectively evaluate the search results.

### **3 Equipment/Materials/Reagents**

- a. (Automotive) Paint Database Query (PDQi) program, current update (Royal Canadian Mounted Police, PDQ Maintenance team, Edmonton, Alberta, Canada)
- b. PDQi User's Manual, current version will be contained within PDQi program
- c. PDQi Code Book, current revision (Royal Canadian Mounted Police, PDQ Maintenance team, Edmonton, Alberta, Canada)
- d. PDQi Contents Manual, most current revision (Royal Canadian Mounted Police, PDQ Maintenance team, Edmonton, Alberta, Canada)
- e. PDQi Spectral Libraries, current revision (Royal Canadian Mounted Police, PDQ Maintenance team, Edmonton, Alberta, Canada)
- f. PDQi Spectral Information Manual, current revision (Royal Canadian Mounted Police, PDQ Maintenance team, Edmonton, Alberta, Canada)

- g. Bio-Rad Know-It-All spectral search software, current revision
- h. PC with operating system and specifications as recommended by the PDQ Maintenance Team

#### 4 Standards and Controls

Validation of all data is conducted by the PDQ Maintenance Team prior to release of each revision. To ensure the database is installed and functioning properly, a “QA/QC test” query is supplied to each user. This query is to be returned to the RCMP prior to expiration of the previous version of the database.

#### 5 Sampling or Sample Selection

Not applicable.

#### 6 Procedure

1. Utilizing Paints and Polymers Standard Operating Procedures (P&P SOPs), determine if a paint sample is a factory-applied, original equipment manufacturer’s (OEM) automotive finish.
2. If the sample contains an OEM automotive finish, conduct Fourier transform infrared spectroscopy (FTIR) analysis of the relevant layers utilizing P&P SOP *FTIR Analysis of Paints, Tapes, and Polymers*.
3. Using the IR peak assignment charts in Appendix A as a guide, code the FTIR spectrum of each layer.
4. If sample size permits, elemental analysis can also be conducted to further characterize the paint layers.
5. Code only inorganic information for the primer (undercoat) layer(s).
6. (Optional): Assign Munsell color designations to the primer layer(s). For details, refer to the P&P SOP *Visual, Microscopical, and Microchemical Examination of Paint and Coating Evidence*.

7. Conduct layer system queries through the PDQ database and/or search collected FTIR spectra using the PDQ spectral libraries.
8. Evaluate potential manufacturer candidates using the P&P SOPs *Conducting Color Comparisons Using Automotive Refinishers Color Chips* and/or *Conducting Motor Vehicle Make-Model-Year Searches Using the National Automotive Paint File (NAPF) Database*.
9. Analyze any available archived sample(s) or potential candidates using the appropriate P&P SOP(s) and directly compare the results to the evidentiary paint sample.

## 7 Decision Criteria

- a. For a layer system query search:
  1. Evaluate the candidates acquired. Search criteria can be adjusted to broaden or narrow the search results. FTIR spectra of possible hits can be compared to further evaluate a candidate.
  2. Once the search criteria are established, determine if a particular manufacturer and/or assembly plant is predominant.
- b. For a spectral library search:
  1. Compare each spectral candidate to the spectrum in question. If the spectra and color assignment compare favorably, that layer is considered a candidate.
  2. Alternatively, conduct simultaneous multilayer spectral searches in order to target a particular plant more efficiently.

## 8 Calculations

Not applicable.

## 9 Measurement Uncertainty

Not applicable.

## 10 Limitations

- a. A factory-applied, OEM automotive finish is required for a possible motor vehicle make-model-year determination.
- b. Not all makes and/or years of vehicles produced by each manufacturer are present in the PDQ database.
- c. Sample size and condition can preclude conducting certain examinations, including color assessment and layer structure.

## 11 Precautionary Statement

Some data entry errors have been noted in the PDQ database. Verify search results using orthogonal resources when practicable.

## 12 Safety

Not applicable.

## 13 References

ASTM E1610, Standard Guide for Forensic Paint Analysis and Comparison. ASTM International, West Conshohocken, PA

ASTM E2937, Standard Guide for Using Infrared Spectroscopy in Forensic Paint Examinations. ASTM International, West Conshohocken, PA

ASTM E2809 Standard Guide for Using Scanning Electron Microscopy/ X-Ray Spectrometry in Forensic Paint Examinations, ASTM International, West Conshohocken, PA

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Buckle, J.L., MacDougall, D.A., and Grant, R.R. PDQ- Paint Data Queries: The history and technology behind the development of the Royal Canadian Mounted Police forensic laboratory



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*FTIR Analysis of Paints, Tapes, and Polymers*, FBI Laboratory, Chemistry Unit - Paints and Polymers SOP

*Visual, Microscopical, and Microchemical Examinations of Paint and Coating Evidence*, FBI Laboratory, Chemistry Unit - Paints and Polymers SOP

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*SEM Analysis of Paints, Tapes, and Polymers*, FBI Laboratory, Chemistry Unit - Paints and Polymers SOP

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Wright, D.M. A Make-Model-Year Case Involving Unusual Primer Chemistry and Good Resources, *J Amer. Soc. Trace Evid. Examiners*, 2010, 2(2): 137-148.

Wright, D.M. Sourcing Paint Smears: A Hate Crime Highlights the Utility of the Paint Data Query (PDQ) Database. *Can. Soc. Forensic Sci. J.*, 2012, 45(2): 79-88.

Rev. #	Issue Date	History
0	06/21/06	New document that replaces previous document also titled <i>Conducting Motor Vehicle Make-Model-Year Searches Using the Paint Data Query (PDQ) Databases</i> .
1	09/30/09	Updated spectral search software options, references, and Appendix.
2	03/14/12	Updated numbers for PDQ in Section 3. Changed macroscopic and microscopic to macroscopical and microscopical throughout document as appropriate. Updated references in section 15.
3	02/03/14	Changes made throughout document to reflect increased capabilities of PDQ software and functionality, new software and hardware requirements, edited Scope to state users should receive hands on training to effectively utilize PDQ, changed “macroscopical” to “visual” throughout, made minor editorial changes, and added reference.
4	09/18/18	Modified scope, deleted “calibration” section, changed section titles as needed to reflect LOM or practice changes, corrections and grammar edits, deleted prescribed SOPs for some analyses to allow for flexibility and updated references.

### **Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 09/17/2018

Chemistry Unit Chief:

Date: 09/17/2018

### **QA Approval**

Quality Manager:

Date: 09/17/2018

## Appendix A <sup>1</sup>:

**Table 10.2** Diagnostic peaks of common binders/resins used in automotive paints

Binder/Resin	Coding	Key peaks (cm <sup>-1</sup> )
Acrylic	ACR	1450 1380 1260 1170 1150
<i>Ortho</i> -phthalic alkyd	ALK OPH	1450 1380 1270* 1130* 1070* 740 700
Isophthalic alkyd	ALK IPH	1475 1373 1305 1237* 1135 1074 730*
Terephthalic alkyd	ALK TER	1270 1250 1120 1105 1020 730
Benzoguanamine	BZG	1590 1540 825 789 710
Cyano	CYA	
Acrylonitrile N≡C	CYA NIT	2238
Isocyanate residue N=C=O	CYA ICN	2272
cf. ferrocyanide, Fe(CN) <sub>6</sub>		(2092)
Epoxy	EPY	1610 1510* 1240 1180 830*
Melamine	MEL	1550 815
Nitrocellulose	NCL	1650 280 840
Polybutadiene	PBD	970 915
Polyurethane	PUR	1690 1530 1470 1250 1070
single peak		1690
modified epoxy		1730 1510 (non-asymmetric broadening)
water based		1690 770
Styrene	STY	1490 1450 760 700
Urea	REA	1655

<sup>1</sup> Reproduction of Table 10.2 in Beveridge, A, et al. Use of Infrared Spectroscopy for the Characterization of Paint Fragments. Chapter 10 in *Forensic Examination of Glass and Paint* (ed. B. Caddy) Taylor and Francis: NY, 2001.

\* indicates a major peak

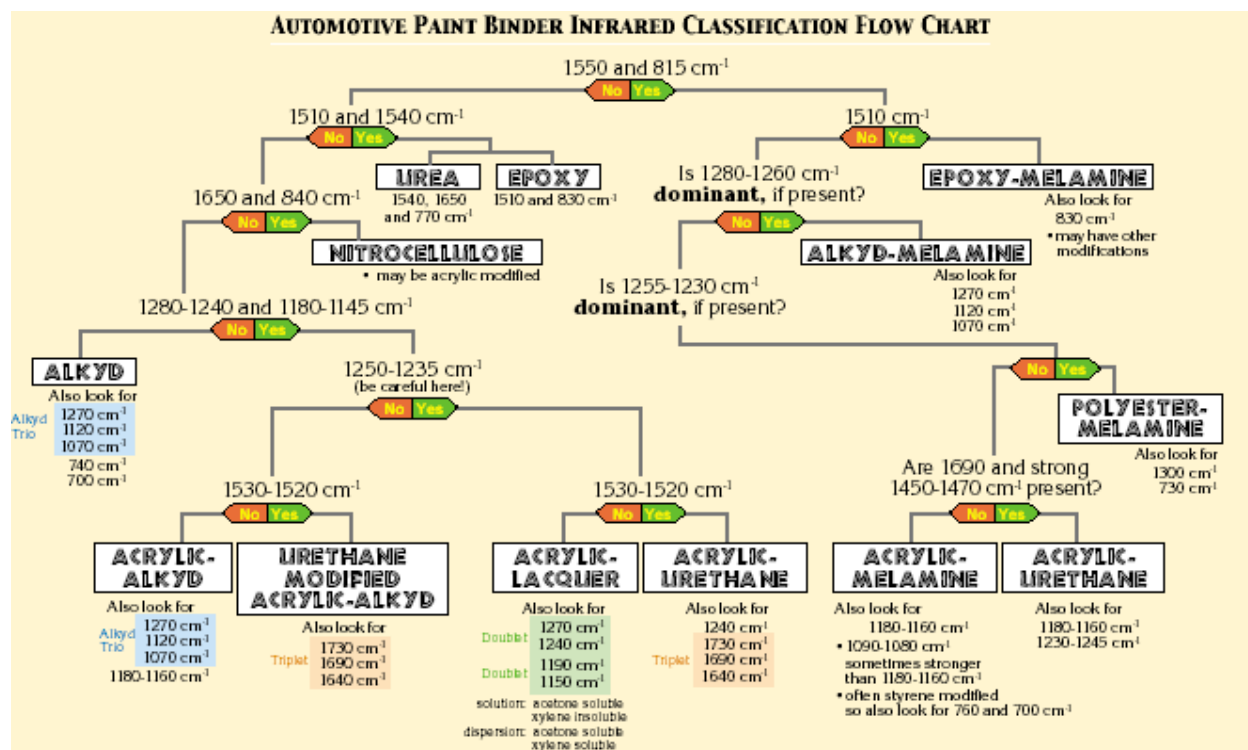
## Appendix A (continued)<sup>2</sup>:

**Table 10.3** Diagnostic peaks of common pigments and extenders used in automotive paints

Pigment and extender	Coding	Key peaks (cm <sup>-1</sup> )
Calcium carbonate	CAR CAC	
Aragonite	CAR CAC ARA	1445 870 857 712 317
Calcite	CAR CAC CAL	1445 870 712 317
Chromate	CHR	
Barium chromate	CHR BCH	935 896 860
Potassium zinc chromate	CHR KZC	950 880 805
Strontium chromate	CHR SCH	920 885 860 840
Oxide	OXI	
Iron oxide	OXI FEO RED	560–530 480–440 350–310
Iron oxide	OXI FEO YEL	899 797 606 405 278
Lead oxide	OXI PBO	530 450
Zinc oxide	OXI ZNO	420–500
Silicon dioxide	OXI SIO	
Cristobalite	OXI SIO CRI	1090 795 621 485 387 300
Opal, diatomaceous silica	OXI SIO OPA	1099 795 475
Quartz	OXI SIO QUA	1081 798 779 512 460 397 373
Titanium dioxide	OXI TIO	
Rutile	OXI TIO RUT	600 (broad suppression) 410 340
Anatase	OXI TIO ANA	600 (broad suppression) 340
Zinc phosphate	PHO ZNP	1120 1080 1020 950 630
Silicate	SIL	
Magnesium (talc)	SIL MGS TAL	1015 670 465 450 420 390 345
Aluminium (kaolinite)	SIL ALS KAO	1035 1005 940 910 540 470 430 350 280
Barium sulphate	SUL BAS	980 630 610

<sup>2</sup> Reproduction of Table 10.3 in Beveridge, A., et al. Use of Infrared Spectroscopy for the Characterization of Paint Fragments. Chapter 10 in *Forensic Examination of Glass and Paint* (ed. B. Caddy) Taylor and Francis: NY, 2001.

### Appendix A (continued)<sup>3</sup>:



<sup>3</sup> Courtesy of Scott Ryland (retired), Florida Department of Law Enforcement, from Course entitled "Paint Examination and Comparison"

## **Conducting Motor Vehicle Make-Model-Year Searches Using the National Automotive Paint File (NAPF) Database**

### **1 Introduction**

The FBI Laboratory's National Automotive Paint File (NAPF) is a collection of automotive paint color standards provided by automobile manufacturers and represent the original factory color available on most imported and domestic vehicles marketed in North America. This physical collection can be utilized in hit-and-run investigations to possibly identify manufacturer(s), model(s), and model year(s) information about automotive paint evidence. NAPF can also be used in conjunction with other automotive paint collections such as refinisher color chips and the PDQ database.

### **2 Scope**

This general procedure applies to Chemistry Unit caseworking personnel who use the NAPF reference panels and database for motor vehicle make-model-year searches.

### **3 Equipment/Materials/Reagents**

- a. FBI Laboratory's National Automotive Paint File (NAPF)
- b. NAPF database software (Microsoft Access format)
- c. Munsell Book of Color, Matte and Glossy Collections available from the Macbeth Division of Kollmorgen Instruments Corporation, New Windsor, NY
- d. Munsell Neutral Value Scale, Matte Edition available from the Macbeth Division of Kollmorgen Instruments Corporation, New Windsor, NY
- e. Minolta CHROMA METER CR-241 Colorimeter, or equivalent, with standard calibration plate
- f. PC computer able to run Microsoft Access 2000 (or better)
- g. Stereo microscope (~6X to ~100X) with two different lighting conditions (ring light oriented at ~180° and fiber optic light oriented at ~45° from sample surface).

#### 4 Standards and Controls

Not applicable.

#### 5 Sampling or Sample Selection

Not applicable.

#### 6 Procedure

1. Utilizing Paints and Polymers Standard Operating Procedure(s) determine if a paint specimen is a factory-applied, original equipment manufacturer's (OEM) automotive finish. Refer to *General Approach for Paint and Coatings Casework* for guidance.
2. Assign a Munsell color designation to the color coat layer. For details, refer to the P&P SOP *Visual, Microscopical, and Microchemical Examination of Paint and Coating Evidence*.
3. Conduct a search through the NAPF database using a range for the Munsell color designation.
  - a. The range for the Munsell hue, value, and chroma can be adjusted to narrow or broaden the search results.
  - b. Additional search criteria can be selected (e.g., metallic/non-metallic, visual color, manufacturer) to further narrow the search results.
4. Compare the color and appearance of the sample to the selected paint standards. For details, refer to the P&P SOP *Visual, Microscopical, and Microchemical Examination of Paint and Coating Evidence*.
  - a. Conduct color comparisons at low power magnification (~6X to ~100X) using two different lighting conditions.
  - b. Record the results of the comparisons.
5. A second P&P examiner evaluates and records their results for any potential candidates.
6. If a paint standard is considered a candidate and contains a OEM factory-applied coating, conduct an Fourier transform infrared spectroscopy (FTIR) examination and comparison of the all applicable layers according to the P&P SOP *FTIR Analysis of Paints, Tapes*,

*and Polymers.*

7. If a particular color is a candidate, contact the manufacturer to obtain information regarding model and year information for the color.

## **7 Decision Criteria**

- a. If the paint reference standard compares favorably in color and appearance to the sample, record it as a candidate.
- b. Determine if a particular manufacturer's color is predominant.

## **8 Calculations**

Not applicable.

## **9 Measurement Uncertainty**

Not applicable.

## **10 Limitations**

- a. Not every color of vehicles produced by each manufacturer is represented in NAPF.
- b. A factory-applied, OEM automotive finish is required for a possible motor vehicle make-model-year determination.
- c. Sample size and condition can preclude conducting certain examinations including color assessment and layer structure.

## **11 Precautionary Statements**

- a. The paint formulation of the NAPF panel may not be representative of all chemistries used for a particular color. Chemistries may differ between manufacturing plants and for paints being applied to different substrates.
- b. Some data entry errors may exist in the database. Verify search results using orthogonal resources when practicable.



- c. The primer layer on a paint standard may not be indicative of the primer system used at a particular manufacturing plant.

## 12 Safety

Not applicable.

## 13 References

Cartwright L.J., et al. The Classification of Automotive Paint Primers Using the Munsell Color Coordinate System – A Collaborative Study. *Canadian Society of Forensic Science Journal*, 1984, 17(1): 14-18.

*Conducting Color Comparisons Using Automotive Refinishers Color Chips*, FBI Laboratory, Chemistry Unit - Paints and Polymers SOP

*Conducting Motor Vehicle Make-Model-Year Searches Using the Paint Data Query (PDQ) Databases*, FBI Laboratory, Chemistry Unit - Paints and Polymers SOP

*General Approach to Paint and Coatings Casework*, FBI Laboratory, Chemistry Unit - Paints and Polymers SOP

*Performance Monitoring Protocol (QA/QC) for the for the CHROMA METER CR-241 Colorimeter*, FBI Laboratory, Chemistry Unit - Instrument Operation and Systems Support SOP

Rev. #	Issue Date	History
0	06/21/06	New document that replaces previous document also titled <i>Conducting Motor Vehicle Make-Model-Year Searches Using the National Automotive Paint File (NAPF) Database</i> .
1	09/30/09	Minor equipment changes and added EUCAP as source of some panels.
2	03/14/12	Added “standard calibration plate” to section 4e. Updated microscopic/macrosopic to microscopical/macrosopical where appropriate throughout document. Clarified criteria for procedure usage in section 8, step 6. Updated references in section 15.
3	02/03/14	Changed “macroscopical” to “visual” throughout document to simplify terminology, expanded introductory description of NAPF panel sources, edited Scope to state users should receive hands on training to effectively utilize NAPF, and made minor grammatical edits.
4	09/18/18	Modified scope, deleted “calibration” section, changed section titles as needed to reflect LOM or practice changes; minor changes to grammar throughout.

### **Approval**

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 09/17/2018

Chemistry Unit Chief:

Date: 09/17/2018

### **QA Approval**

Quality Manager:

Date: 09/17/2018

## **Visual and Microscopical Examination of Polymeric Material Evidence**

### **1 Scope**

This procedure applies to Chemistry Unit caseworking personnel who perform visual and microscopical examinations to characterize and compare a variety of polymeric samples.

### **2 Equipment/Materials/Reagents**

- a. Stereo microscope (~6X to ~100X) with appropriate lighting (such as an annular ring light or fiber optic lights)
- b. Glass microscope slides
- c. Scalpel handle and blades
- d. Tweezers
- e. Probes (e.g., steel, tungsten, wood, or Teflon™)
- f. Disposable wipes
- g. Well slides
- h. Pillboxes
- i. Compressed-gas duster
- j. Large sheets of untreated kraft paper (or equivalent)
- k. Large, flat-bladed spatula
- l. Micrometer, 0-1" range, accurate to 0.0001", or equivalent
- m. Ruler with a minimum of 1/16" gradations
- n. Digital camera

### 3 Standards and Controls

Not applicable.

### 4 Sample Selection

Refer to the Paints and Polymers Standard Operating Procedure (P&P SOP) *General Approach for Polymeric Casework* for guidance on sample(s) selection. Record the samples selected for analysis in the case notes.

### 5 Procedure

#### 5.1 Visual and Microscopical Examination

Use written descriptions, sketches, photography, or other imaging methods to capture both visual and microscopical characteristics and observations. If the items are suitable for further examination, record a detailed description of each item to include comparative features or any unusual conditions (e.g., commingled material).

1. Process each item separately to prevent cross-contamination.
2. Transfer the item from its original container to a suitable substrate (e.g., paper, glass microscope slide, pillbox) to examine both visually and microscopically. Evidence that is too large or bulky to fit under a conventional stereomicroscope for examination can be handled as follows.
  - a. Use a modified base for the stereomicroscope.
  - b. Alternatively, section and examine an area of interest from the bulk material.
3. Some specimens require processing or preparation prior to examination.
  - a. Clothing: Examine each article of clothing visually and microscopically for evidence of a polymeric material transfer.
    - i. If a potential polymeric material transfer is embedded or abraded onto the fabric, take a cutting which includes a sample of the transferred substance and preserve it for future examination. See 3.b. for further instructions regarding smears.

- ii. Process each article of clothing as it was received (i.e., individually or collectively packaged) and isolate the debris in the same manner (i.e., one pillbox per package).
  - iii. Suspend the item from a rack over a large sheet of paper and carefully scrape all surfaces in a downward motion with the edge of a large flat-bladed spatula or similar tool to dislodge any remaining polymeric evidence.
  - iv. Collect the deposited debris and transfer it to a pillbox or other container for microscopical examination. Label the top and bottom of the container with the laboratory number, item number, and initial. See 3.c. for further instructions regarding debris.
- b. Smears: Oftentimes, the amount of energy imparted in a transfer of polymeric material will cause it to melt or soften and resolidify, fusing the polymer to the substrate.
- i. If fused or embedded onto a surface, remove particles and fragments using a scalpel blade, probe, tweezers, or similar tool while observing under a microscope. If the item will be subsequently examined for toolmark comparisons, relatively soft, pliable materials such as wood or Teflon<sup>TM</sup> should be used to dislodge the suspected polymeric material from the surface. Metal blades should not be used as they can alter the surface and thereby affect a toolmark examination.
  - ii. The fabric weave of an article of clothing can be stretched in order to facilitate removal/dislodging polymeric particles.
  - iii. Transfer isolated particles/fragments to a well slide or pillbox for future examination. Label the slide or pillbox with the laboratory number, item number, and initial.
  - iv. Smeared samples can be contaminated with material from the surface upon which it is impacted (e.g., soil, fibers, paint, wood) thereby affecting the chemistry and/or color of the sample. If appropriate, take a control sample of the substrate close to but not within the area containing the smear.
- c. Debris: Polymeric evidence can be mixed in with other materials that are not probative for examination by P&P personnel (e.g., fibers, glass, soil).

- i. Examine the contents of the debris microscopically, manipulating it with the appropriate tools (e.g., tweezers, scalpel, probe) and isolate any plastic-like materials.
  - ii. Transfer these materials to a well slide or pillbox for future examination. Label the slide or pillbox with the laboratory number, item number, and initial.
  - iii. To decrease the likelihood that polymeric evidence has been overlooked, a second P&P examiner can examine the debris. Alternatively, the primary examiner should re-examine the debris on a different day. Record the results of these subsequent analyses in the case notes.
4. Once isolated, observe the surface of the specimen(s) and record color, morphology, degree of gloss, texture, the presence of manufacturer markings, the presence of surface striae, defects, or any other characteristics that help to describe the item.
5. Record the overall shape and nominal dimensions of the item such as length, width, and thickness. Nominal measurements can be taken with a ruler. If appropriate, obtain thickness measurements for comparison between specimens.
  - a. Using a micrometer, obtain thickness measurements from at least three different areas on the sample. Record the instrument manufacturer, ID number, date of next calibration, and any individual readings taken (to the nearest 0.00005"). Some polymers will permanently deform when stretched or stressed; therefore, only conduct thickness measurements on items that do not appear to have been severely distorted or degraded.
6. View the specimen(s) at ~6X to ~100X magnification and determine if it is multi-layered. Record all observations in the case notes.
  - a. Obvious layers can be exposed/observed by a number of techniques which include, but are not limited to viewing the sample on edge, cross-sectioning by hand, cross-sectioning by encapsulation and microtomy or polishing, making an oblique (bias) cut through the sample, or taking a series of thin peels through each layer.
  - b. A combination of techniques can be used to fully characterize the layer structure. The extent of sample manipulation and preparation will depend on the amount of sample available, its complexity, and its characteristics.

## **5.2 Sourcing Examination**

1. Record any observed manufacturer markings found on a sample with descriptive notes to include any letters, numbers, or symbols observed on the item as well as the relative location of the marking. If imaging techniques such as photography are used, include a scale or notation of the magnification in the image.
2. Employ resources within and outside the FBI Laboratory as applicable to develop additional information about the potential source(s) of the item.

## **5.3 Physical Reconstruction Examination**

1. Observe the specimens for possible fracture (physical) matches. A fracture match can be recognized by the alignment of broken edges, manufacturer markings, and/or surface anomalies (e.g., striae, texture).
  - a. Fracture matches are the most conclusive type of examination and must be recorded with descriptive notes and imaging techniques
  - b. Include a measuring scale in any collected images when practicable. If not practicable, annotate the image with the magnification used.
2. A second P&P examiner must confirm and record suspected fracture matches between known and question specimens.

## **6 Decision Criteria**

- a. If physical characteristics of two (or more) specimens being compared differ, cease examinations and report that the specimens differ.
- b. Decision criteria for a fracture match are described in section 5.3.

## **7 Calculations**

Not applicable.

## **8 Measurement Uncertainty**

Not applicable.

## 9 Limitations

- a. Sample size and condition can preclude conducting certain examinations, including color assessment.
- b. Sourcing capabilities of common synthetic polymeric materials is limited. This is directly related to the abundance of such materials in the marketplace and the number of end uses for a particular polymeric material.
- c. Reporting a potential source for automotive parts (i.e., vehicle make/model/year) is limited to the manufacturer's part numbers. For automotive parts, the SAE (Society of Automotive Engineers) numbers can only provide information as to the function of the part on the automobile.

## 10 Precautionary Statement

As with any procedure involving trace evidence, ensure actions minimize the potential for loss or contamination.

## 11 Safety

Use standard precautions for the handling of potentially biohazardous materials, chemicals or sharps. Refer to the *FBI Laboratory Safety Manual* for guidance.

## 12 References

Alger, M.S.M. *Polymer Science Dictionary*. NY: Elsevier Science, 1989

*FBI Laboratory Safety Manual*

*General Approach for Polymeric Casework*, FBI Laboratory, Chemistry Unit – Paints and Polymers SOP

Parsons, N.S., and Mountain, C.A. Investigating Polyurethane Foam as a Form of Trace Evidence. *Science and Justice* 2007; 47:24-33.

Seymour, R.B., Carraher, Jr., C.E. *Polymer Chemistry: An Introduction*, 2d ed. NY: Marcel Dekker, 1988.



Rev. #	Issue Date	History
0	06/21/06	New document that replaces previous document also titled <i>Macroscopic and Microscopic Examination of Polymeric Material Evidence</i> .
1	09/30/09	Changed the sampling plan guidelines and updated references.
2	03/14/12	Updated microscopic and macroscopic to microscopical and macroscopical where appropriate throughout document. Changed “sampling” plan to “sample selection” plan in section 8. Addressed decision criteria for comparison of thickness measurements in section 10.
3	02/03/14	Changed “macroscopical” to “visual” throughout to simplify terminology, edited equipment list to be less specific, removed discussions related to thickness comparisons in Section 9.1, 5a, and made minor grammatical editing. Photography documentation requirements edited in Section 9.3, 1b. Deleted calculations in Section 11.
4	09/18/18	Modified scope, deleted sections that do not describe procedural content, changed section titles as needed to reflect LOM or practice changes, added procedural details as warranted for clarification, minor grammatical edits throughout.

### Approval

Redacted - Signatures on File

Paints and Polymers  
Technical Leader:

Date: 09/17/2018

Chemistry Unit Chief:

Date: 09/17/2018

### QA Approval

Quality Manager:

Date: 09/17/2018

## **Physical Examinations of Tape Evidence**

### **1 Scope**

This procedure applies to Chemistry Unit caseworking personnel who conduct physical examinations that are used to characterize and compare a variety of tape specimens.

### **2 Equipment/Materials/Reagents**

- a. Stereo microscope (~6X to ~50X magnification) with appropriate light source (e.g., annular ring light, fiber optic light)
- b. Compound microscope (~35X to ~400X magnification) with appropriate light source
- c. Ultraviolet light with long wavelength (~365nm) source
- d. Cold source (e.g., liquid nitrogen, freezer)
- e. Heat source (e.g., air dryer, heat lamp)
- f. Scalpel handle with blades
- g. Single edge razor blades
- h. Probes (e.g., steel, tungsten, wood, Teflon™)
- i. Digital micrometer, 0-1" range
- j. Ruler with metric (1 mm) and/or English (1/64") graduations
- k. Tweezers
- l. Glass microscope slides
- m. Disposable wipes
- n. Cotton swabs
- o. Thin-layer chromatography glass chamber, or equivalent, with lid
- p. Acetone (Reagent grade)

- q. Hexane (Reagent grade)
- r. Methanol (Reagent grade)
- s. Chloroform (Reagent grade)
- t. Eyedropper bottles and/or disposable pipettes
- u. Heavy-gauge transparency film or KAPAK<sup>®</sup> tubular rollstock
- v. Munsell Neutral Value Scale and Soil Color Charts available from the Macbeth Division of Kollmorgen Instruments Corporation, New Windsor, NY
- w. Polarizing light microscope (PLM) with a quarter wave or full wave plate
- x. Digital camera
- y. Digital microscope

### 3 Standards and Controls

Not applicable.

### 4 Sample Selection

Refer to the Paints and Polymers Standard Operating Procedure (P&P SOP) *General Approach for Tape Casework* for guidance on sample(s) selection. Record the samples selected for analysis in the case notes.

### 5 Procedure

If the tape evidence is received as a tangled mass or a series of overlapping strips, separate, flatten, and arrange each piece on heavy-gauge transparency film (or equivalent clear, colorless plastic sheets) for comparison of physical properties. Vinyl plastic document protectors are not suitable due to the presence of volatile plasticizers (e.g., phthalates) within their matrix, which can migrate into the adhesive layer of the tape; however, clear and colorless plastic evidence storage bags (e.g., KAPAK<sup>®</sup>) are acceptable.

Tape specimens that are wadded together or overlapped require special attention in order to free the adhesive layers. Suggested methods to aid in the separation are described below and should

be ordered to minimize alteration of the items received (e.g., adhesive removal, fabric or backing distortion, damage to the free ends).

- A. Gently heat the tape with an air dryer while applying tension on opposite sides of the wadded area in order to gradually reduce the tack of the adhesive without tearing or distortion of the tape during manipulation.
- B. Fill a porcelain dish or crucible with liquid nitrogen and immerse the tape for approximately one minute. If freezing does not enable quick and easy unpeeling, attempt this process a second time before trying another separation technique. Alternatively, the tape can be placed in an evidence freezer overnight (under proper seal and appropriately labeled).
- C. If the tape is so entwined as to risk damage or tearing with the methods mentioned above, suspend the mass in a thin layer chromatography tank along with approximately 100 mL of chloroform (in two 50 mL beakers placed at opposite ends of the tank with the wadded tape in between). Cover the appropriately-labeled tank, and place it under proper seal in a fume hood for several hours or overnight. The solvent-saturated atmosphere should reduce the tack of the adhesive to facilitate separation and flattening of the pieces.
- D. Under a vented fume hood, apply an appropriate solvent drop wise to the edges of a wadded area or adhesive/substrate interface and gently pry the edges apart. Initially employ a mild solvent (e.g., hexane for rubber-based adhesives or acetone for acrylic-based adhesives). If a more aggressive solvent is required, chloroform can be used. Moderate heating can be helpful in conjunction with the solvent application.

Use written descriptions, sketches, photography, or other imaging methods to capture both macroscopical and microscopical characteristics and observations. A flow chart of the physical examinations used to conduct a tape comparison is provided in Figure 1. The discrimination value of the physical characteristics depends on the type and condition of the tape. Therefore, the order of the examinations depicted in Figure 1 is left to the discretion of the examiner.

- 1) Observe the separate components of the tape specimen using both the unaided eye and a stereo microscope with ~6X to ~50X magnification.
  - a) Adhesive Examinations.
    - i) Record observations regarding the adhesive color. Provided color attributes have not been obscured by environmental effects or previous forensic examinations (e.g., weathering, latent fingerprint processing), differences will quickly disassociate items of evidence. Important distinctions include a clear, colorless adhesive versus a black adhesive on vinyl electrical tape, or a tan adhesive versus a clear, colorless adhesive

on brown packaging tape. Duct tape adhesive color comparisons can be facilitated using the matte version of the Munsell Neutral Value Scale or Soil Color Charts.

b) Backing Examinations.

- i) Record observations describing the tape backings such as color, degree of gloss, texture, or fabrication markings. If necessary, clean the tape backing with a mild solvent (e.g., methanol, water) and a cotton swab. Evaluate the layer structure of a duct tape backing. This can be accomplished by freezing the backing to ensure rigidity, taking a cross-section with a single-edged razor blade, and viewing the cross-section in transmitted light with a compound or digital microscope.
- ii) Tape width is another possible point of comparison between full width specimens. Measure and record the observed widths to the nearest  $\frac{1}{64}$ " or 0.5 mm using a ruler. A simple observation is often all that is needed when widths are different (e.g., 2" versus 3" wide duct tape). Additionally, some tapes will permanently deform when stretched, torn, or stressed. Therefore, only conduct width measurements on items that do not appear to have been severely distorted or degraded.
- iii) Overall tape and backing thicknesses are other possible points of comparison between specimens. Observe differences between thicknesses using transmitted light microscopy of tape cross-sections with a common scale/magnification. Alternatively, measure thicknesses using a micrometer. If measurements are performed, take a minimum of three measurements along a representative sample, and record these values to the nearest 0.00005" along with the manufacturer and ID number of the micrometer. For backing thicknesses, remove adhesives prior to measurement. Only conduct thickness measurements on items that do not appear to have been severely distorted, degraded, or contaminated.
- iv) If the backing is a clear, colorless polymeric film, affix a sample to a glass microscope slide oriented with the machine direction parallel to the edge of the slide. Observe the tape under a PLM using crossed polars and compare the extinction angle and birefringence colors.

c) Fabric Examinations.

- i) Tape samples containing fiber or fabric reinforcement can be differentiated based on several factors: yarn count, yarn composition and construction, and fabric weave. To expose the fabric or fiber reinforcement, remove rubber-based adhesives with hexane or chloroform; remove acrylic-based adhesives with acetone.
  - (1) Determine the weave pattern and a general description of the warp and fill yarns. Plain weave and weft-insertion are common examples of fabrics encountered in

duct tape samples. Record the weave and yarns in the case notes (e.g., description, sketch, photocopy, photograph).

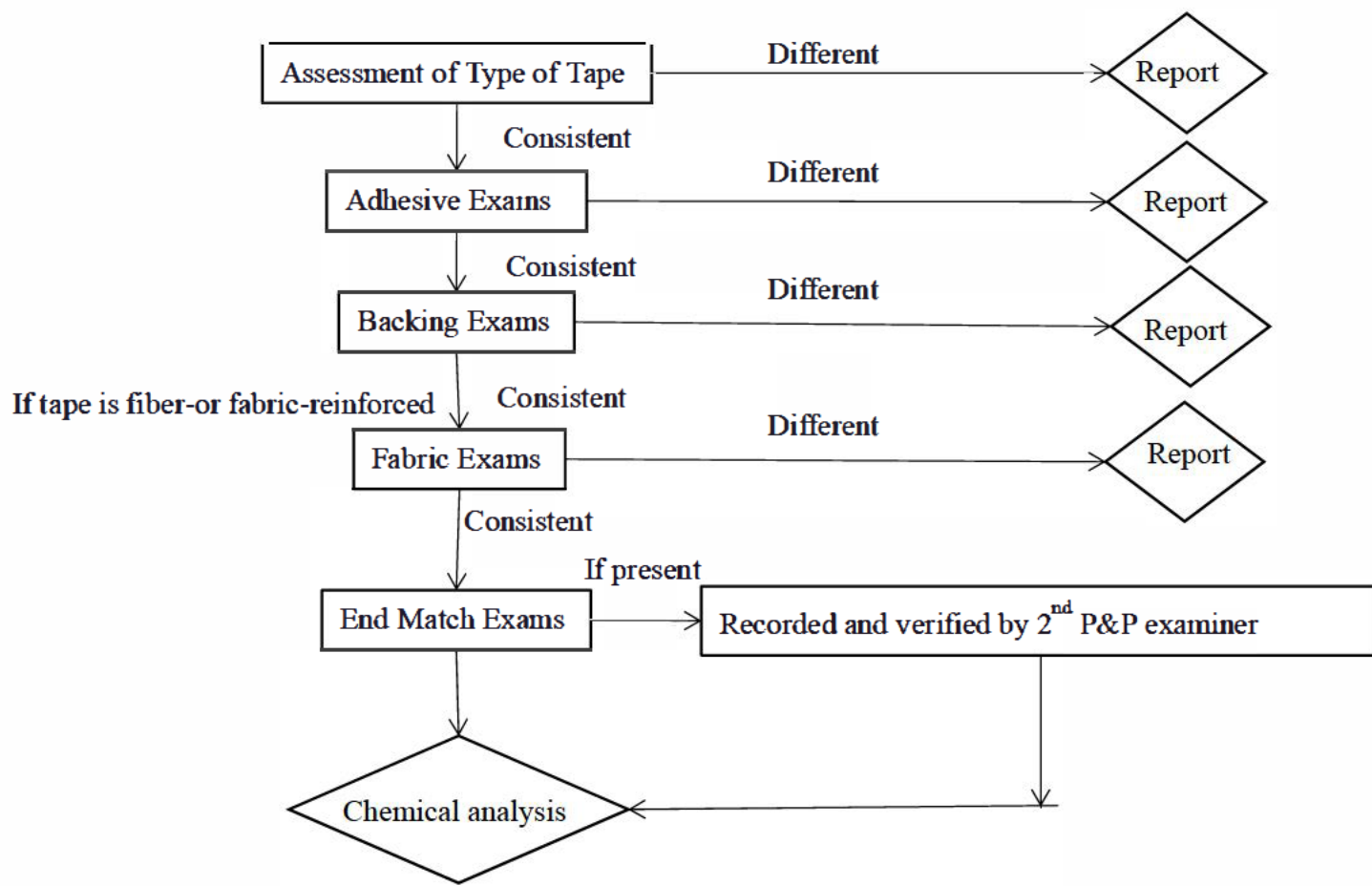
- (2) Determine the number of yarns in a one square inch section of the tape in both the machine direction (m.d., warp) and cross direction (c.d., fill). If the sample is large enough, consider taking repeat measurements. If the sample is limited in size, calculate the yarn count in the machine and/or cross direction on an area less than one inch. Record the area measured in the case notes.
- (3) Determine and record whether yarns in the machine and/or cross directions fluoresce under UV exposure at ~365 nm.
- (4) If two or more tape samples have not been discriminated at the conclusion of all examinations, forward samples containing fabric or glass filament reinforcement to the appropriate personnel for additional testing. Those results will be provided in a separate Laboratory Report by the assigned examiner, but P&P personnel should disclose the additional (pending) tape exams.

2) Examine tape evidence for possible end matches between specimens.

- a) If a possible end match is observed, examine the tear pattern from the backing and adhesive sides of both specimens with the aid of a stereomicroscope to determine whether an end match remains plausible.
- b) If the backing is distorted or folded over and adhered to the adhesive layer, carefully straighten it out to restore the edge. This can be facilitated with the use of tweezers, heat, or mild solvent.
- c) Determine if there are individualizing characteristics such as a defect in the backing or in the fabric reinforcement that extends across the fracture. The beginning and end point of the defect must be apparent on both specimens to ensure that it is not continuous along the entire roll of tape.
- d) If the tape has fiber or fabric reinforcement, use an appropriate solvent to remove enough of the adhesive layer to expose the reinforcing yarns. Ensure that the yarns in the machine (and if present, fill) direction line up along the fracture.
- e) Depending on the type of tape, fabrication marks such as striations from extruders or embossed marks from calendar rolls can align across fractured edges providing additional features to corroborate an end match.
- f) Record any end matches with descriptive notes.

- g) A second P&P examiner must verify and record suspected fracture matches between known and question specimens. This confirmation can occur before or after the end matches are imaged with a measuring scale. In the case of fabric-reinforced tapes, include a diagram or map of the severed yarns to illustrate each complementary pair of warp yarns rejoining along a common margin at the fracture.
- h) Unless deemed necessary based on case details, a second P&P examiner does not need to confirm end matches that are observed within an item or between items from the same location. This step is therefore disregarded in Figure 1. Furthermore, one piece of the reconstructed strip can be used for the remaining examinations.
- 3) If all physical characteristics are consistent between specimens being compared, proceed with instrumental examinations according to the applicable P&P SOPs.

**Figure 1. Basic Approach to Physical Examinations of Tape Specimens**



## **6 Decision Criteria**

- a. If physical characteristics of two specimens being compared differ, cease examinations and report that the specimens are different.
- b. Since width and thickness are known to vary along the length of a roll of tape, exercise caution when minor differences are observed between samples (e.g.,  $\pm 1.0$  mm or  $\pm 10\%$ , respectively).
- c. According to duct tape industry contacts, a yarn count variation of  $\pm 1$  yarn in either or both directions is acceptable within-roll variation.
- d. Decision criteria for a tape end match are described in section 5, step 2.

## **7 Calculations**

Not applicable.

## **8 Measurement Uncertainty**

Not applicable.

## **9 Limitations**

- a. Sample size and condition can preclude conducting certain examinations.
- b. Color, width, thickness, and/or scrim count assessments can be affected by sample condition.
- c. In the absence of an end/fracture match, a tape specimen cannot be definitively associated to a particular roll of tape.

## **10 Precautionary Statement**

As with any procedure involving trace evidence, ensure actions minimize the potential damage to the sample, particularly with respect to tearing or distorting of the tape ends.



## 11 Safety

Use standard precautions for the handling of potentially biohazardous materials, chemicals (including liquid nitrogen), and sharps. Refer to the *FBI Laboratory Safety Manual* for guidance.

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Rev. #	Issue Date	History
0	06/21/06	New document that replaces previous document also titled <i>Macroscopic and Microscopic Examination of Tape Evidence</i> .
1	09/30/09	Changed the sampling plan guidelines and updated references.
2	02/27/12	Updated microscopic and macroscopic to microscopical and macroscopical where appropriate throughout document. Added digital microscope to section 5. Changed "sampling" plan to "sample selection" plan in Section 8. Clarified end match procedure in Section 9, step 5f. Changed thickness measurement decision criteria in Section 10. Updated references.
3	12/23/13	Changed title. Section 9 has been reordered and Figure 1 revised. Procedures for taking width and thickness measurements have been changed in Section 9 and statistical evaluations of same have been removed from Sections 10 and 11. Section 10 has also been edited to include width and thickness variations along the length of a roll of tape. References updated. Other minor editorial changes made throughout.
4	09/18/18	Deleted Introduction, Principle and Specimens sections as they did not describe procedural content and renumbered. Modified scope, updated section titles as needed to reflect LOM or practice changes, minor grammatical edits for clarification throughout (document changed to record), and updated references.
5	07/23/19	Removed requirement to record date of calibration of micrometer; minor grammatical corrections to comply with other quality system documents (e.g., confirm to verify, documented to recorded).

### **Approval**

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Date: 07/19/2019

Chemistry Unit Chief:

Date: 07/19/2019

### **QA Approval**

Quality Manager:

Date: 07/19/2019

## **Conducting Duct Tape Sourcing Searches in the National Forensic Tape File (NFTF) Using Spectral Library Identification and Classification Explorer (SLICE)**

### **1 Introduction**

The National Forensic Tape File (NFTF) is a collection of various tapes collected by FBI Laboratory personnel from tape manufacturers or retail outlets. Acquisition information, physical characteristics, and analytical data about each tape sample are compiled in a searchable database known as Spectral Library Identification and Classification Explorer (SLICE). This collection can be utilized in duct tape sourcing investigations to develop manufacturer and product information about duct tape evidence. The NFTF can also be used in conjunction with information available from industry contacts.

### **2 Scope**

This general procedure applies to Chemistry Unit caseworking personnel who utilize the NFTF reference samples and SLICE database for duct tape sourcing.

### **3 Equipment/Materials/Reagents**

- a. NFTF
- b. PC capable of running the SLICE software program
- c. SLICE software (xk, Inc., EDAX)

### **4 Standards and Controls**

Not applicable.

### **5 Sampling or Sample Selection**

Not applicable.

### **6 Procedure**

1. Utilizing guidance provided in *General Approach for Tape Casework*, evaluate the physical characteristics and chemical composition of a tape specimen. Not every technique need be conducted before searches commence.

2. Conduct a search(es) through SLICE using the physical characteristics and/or chemical composition of the tape specimen. It is recommended that the search criteria be limited to duct tapes.
  - a. A best fit search can be conducted using the spectrum of the tapes' adhesive or backing. The candidate list will be provided in order of best to least fit.
  - b. Drop down menus for certain physical characteristics (e.g., weave, backing color) can be selected and searched. If a best fit search has not been indicated, any reference tape that meets the criteria selected will be included in the candidate list. If a best fit search has been selected, the candidate list will be in order of best to least fit and will only include references tapes that meet all selected criteria.
  - c. A range for the scrim count can be searched. Results will be displayed as in 2.b.
  - d. A text based search (e.g., organic composition) can be selected to further narrow the search results. Results will be displayed as in 2b.
3. One possible search procedure is as follows:
  - a. Perform a best fit search of the tape's adhesive. Compare the spectrum of each successive candidate until the spectra no longer compare favorably with the questioned sample spectrum. If the list of candidates is still too numerous to manage, repeat the search with additional physical characteristics included. Determine if any possible candidates can be eliminated by comparing the remaining information available for the samples in the candidate list.
  - b. Perform a best fit search of the tape's backing. Compare the spectrum of each successive candidate until the spectra no longer compare favorably with the questioned sample spectrum. If the list of candidates is still too numerous to manage, repeat the search with additional physical characteristics included. Determine if any possible candidates can be eliminated by comparing the remaining information available for the samples in the candidate list.
  - c. Cross-reference the candidates remaining from the search described in 3a to the candidates remaining from the search described in 3b. Any candidates found in common can be directly compared to the questioned tape specimen.
4. Compare the color, appearance, thickness, and the acquired Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction data of the evidentiary tape specimen to the selected tape standards.
5. If a particular manufacturer or product cannot be eliminated as a candidate, contact the manufacturer to see if the information can be corroborated.

## **7 Decision Criteria**

If a duct tape reference standard compares is consistent favorably in physical characteristics and chemical composition to the evidentiary duct tape specimen, record as a candidate.

## **8 Calculations**

Not applicable.

## **9 Measurement Uncertainty**

Not applicable.

## **10 Limitations**

- a. Not every duct tape product is represented in NFTF.
- b. Sample condition can preclude conducting certain examinations, such as color assessment and overall thickness.

## **11 Precautionary Statements**

- a. Some data entry errors may exist in the database. Verify search results using orthogonal resources when practicable.
- b. Adhesive and backing color differences can occur between questioned and reference samples, which may not eliminate the reference tape as a potential candidate.
- c. Since width and thickness are known to vary along the length of a roll of tape, and between different rolls of the same product, observed differences in these parameters between questioned and reference tapes may not be indicative of different products or manufacturers.

## **12 Safety**

Not applicable.

### 13 References

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Rev. #	Issue Date	History
0	12/18/09	New document.
1	12/23/13	Minor editorial changes in sections 3 and 12. In section 4a, provided description for NFTF. References to <i>Macroscopic and Microscopic Examinations of Tape Evidence</i> changed to reflect current title of document as <i>Physical Examinations of Tape Evidence</i> . Section 13 expanded. Updated references to include article by Wright and Mehlretter.
2	09/18/18	Modified scope, streamlined procedure for clarity, minor grammatical edits throughout, and updated references.

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