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

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1 INTRODUCTION

Due to their discriminating power, visual and microscopical examinations are the initial steps in a forensic paint and coating analysis.

2 SCOPE

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

3 EQUIPMENT

- Stereo microscope (~6X to ~100X) with two lighting conditions (e.g., ring light oriented ~180^{o} from sample, fiber optic light oriented ~45^{o} from sample)
- Compound microscope with bright field and polarizing light sources
- General Laboratory Supplies (e.g., tweezers, well slides, pillboxes, flat-blade spatula, spot plates)
- Diphenylamine (DPA) (Reagent grade)
- Sulfuric acid concentrated (Reagent grade)
- Acetic acid, glacial (Reagent grade)
- 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.

4 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 as 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 is used as a negative control for the other solvents/microchemical tests.

5 SAMPLING

Refer to PP-800 for guidance for sample(s) selection. Record the samples selected for analysis in the case notes.

6 PROCEDURE

6.1 Visual and Microscopical Examination

A. 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).

B. Process each item separately to prevent cross-contamination.

C. 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.

1. 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 below 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. This container along with any others created to house recovered debris or evidence will be recorded as secondary evidence. Refer to the LAB-100 and CHEM-100 for further guidance in accounting for secondary evidence created in the lab during the course of examination. See below for further instructions regarding debris.

2. 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® 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.

3. Debris: Paint evidence can be observed as a mixture with other materials that are not probative for examination by Paints and Polymers (PP) 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 PP analyst can examine the debris. Alternatively, the primary analyst should re-examine the debris on a different day. Results of these subsequent analyses are recorded in the case notes.

4. 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.

D. 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.

E. If conducting a comparative examination, observe paint chips for possible physical fits such as an observed reconstruction between broken-edge characteristics and/or surface anomalies (e.g., striae).

1. Physical fits determinations are the most conclusive type of examination. Record observed physical fits with descriptive notes and imaging techniques.

2. Include a measuring scale, when practicable. If not, annotate the photograph with the magnification used to capture the image.

3. A second PP analyst will confirm and record the suspected physical fit(s) between known and questioned specimens.

F. Observe the layer structure of any paint specimen(s) by viewing it at ~6X to ~100X magnification.

1. 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.

2. 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.

G. Record the number and sequence of layers and their relative thicknesses.

H. 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).

I. 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.

J. If appropriate, conduct color measurements on comparative items. See PP-301 for guidance.

6.2 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 PP-800 for guidance on appropriate techniques.

A. 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. All tests should be conducted on the negative control as well as the sample.

1. If the specimen is an enamel (e.g., melamine in Fourier transform infrared spectroscopy (FTIR) spectrum), solubility and microchemical tests are unnecessary.

2. If the specimen is a lacquer or the binder characterization is not obvious by FTIR, provided there is adequate sample, proceed with the solubility and microchemical tests.

3. When conducting solubility and microchemical reactivity tests, analyze negative and positive controls concurrently with the sample(s).

B. 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.

1. If the layer dissolves, proceed to step C.

2. 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 E for further detail.

C. 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.

1. If the layer dissolves, record it as a dispersion lacquer. No additional solubility testing is necessary for this layer.

2. If the layer does not dissolve, proceed to step D.

D. 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.

1. 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.

2. 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.

E. 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.

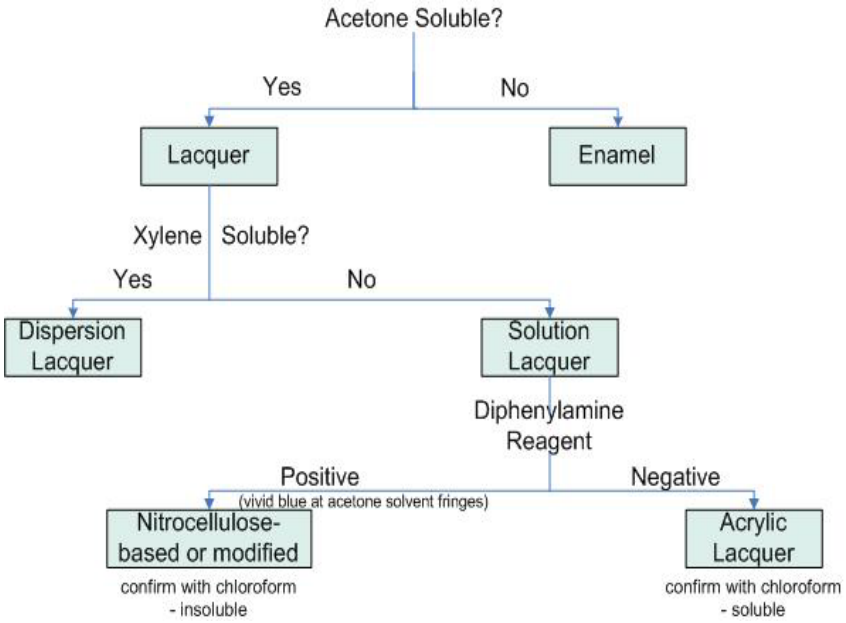

Figure 1. Basic Microchemical Scheme[1]

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, discontinue examinations and report the specimens as different.

- Binder classification by the solubility and microchemical tests utilized in this procedure are described in section 6.2.

[1] 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 LIMITATIONS

- Sample size and condition can preclude conducting certain examinations, including color assessment(s).
- 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.
- Paint layers less than 15 microns thick can be difficult to distinguish using standard stereo microscopical examinations.
- Adjacent layers similar in color and texture can be difficult to resolve using standard stereo microscopical examinations.
- A factory-applied, OEM automotive finish is required for a possible motor vehicle make-model-year determination.
- As with any procedure involving trace evidence, ensure actions minimize the potential for loss or contamination.
- Solubility and microchemical tests can be destructive. Consider this factor when evaluating the probative value of such tests.

9 REFERENCES

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

LAB-100, FBI Laboratory

CHEM-100, FBI Laboratory, Chemistry Unit

PP-800, FBI Laboratory, Chemistry Unit

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.

10 REVISION HISTORY

Revision	Issued	Changes
06	06/15/2022	Reformatted and previous sections 9 and 10 moved to PP-301.
07	01/02/2025	Minor edits to conform to latest document referencing. Changed examiner to analyst in section 6 regarding physical fit evaluations and debris exams. Added LAB-100 and CHEM-100 as references.