Procedures For Paint Analysis

The SBI Laboratory utilizes a number of methods for the analysis of dry paint films. The selection of methods appropriate to each case is based on the quantity and condition of the evidence samples available. This document describes each procedure that may be used for paint analysis.

I. Documentation of evidence

A. The outermost sealed container must be marked with the date received and the analyst's initials. The evidence is then stored in a secured area until analysis begins.

B. At the beginning of the analysis, each item of evidence is to be removed so that no cross-contamination occurs and so that the item can be clearly associated with its container.

II. Visual examinations

A. Macroscopic examination

The visual and macroscopic evaluation, description, and documentation of the sample's original condition is the first step in an analysis. It may also be the final step if exclusionary features or conditions are identified.

1. Describe as many physical features as possible. These may include color, size, layers, texture, and general condition.

B. Microscopic examination

A stereomicroscope with a magnification ability of 5 -100 power or a polarizing light microscope may be used for further evaluation of the physical characteristics of paint samples.

1. Determine the number of layers and sequence of layers in a paint sample. This may be accomplished by turning the paint chip on its edge and viewing with high magnification. If the layer characteristics cannot be thoroughly determined, the paint chip may be sliced with a razor blade or scalpel blade.

III. Chemical tests

A. Solvent tests.

Paint samples may react with solvents by dissolving, swelling, curling, softening or other physical reactions. Acrylic lacquers are soluble in both chloroform and acetone; nitrocellulose lacquers are soluble in acetone and insoluble in chloroform. Enamels are insoluble in both acetone and chloroform.

1. Place the sample to be tested in spot plate well or on a glass slide over a contrasting background. Prepare the sample so that all the layers can be observed. This may be done by slicing a thin cross-section or by individually separating the layers. Place one drop of chloroform on the paint sample and observe the reaction with a microscope. Record the results.

2. Repeat the procedure with one drop of acetone. If the sample treated with chloroform does not react in any significant way, the same sample may be used for the acetone test. If the sample does react with chloroform, a new sample must be prepared.

3. Other solvents may be used as described for chloroform and acetone.

B. Chemical reagents.

1. The sample to be tested is prepared as described for solvent tests. Diphenylamine solution is prepared by mixing one gram of diphenylamine with 40 mls water and 200 mls concentrated sulfuric acid. One drop of this solution is placed on the paint sample and it is observed through a microscope to determine if a cobalt blue color develops where the solution contacts the paint. (The diphenylamine solution must be verified by testing with a known sample of nitrocellulose paint prior to use).

2. Other reagents may be employed as needed to test for dye solubility, pigment effervescence, flocculation, and color changes.

IV. Physical match

The most definitive comparison that can be made between two otherwise visually similar paint samples is the matching of reference and questioned sample edges for a physical fit or matching the surface striae on the underside of a paint fragment to those on a parent surface. This assumes that the samples in question exhibit sufficient uniqueness for comparison.

A. The edges (or striae) of the samples are visually examined macroscopically and, if appropriate, microscopically for a physical fit.

B. Any physical fits should be documented by photography or video microscopy or other appropriate means.

V. Instrumental methods of analysis

A. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is employed whenever a comparison and/or identification of paint film binders is necessary. Also, some of the inorganic components of a paint sample may be determined by FTIR.

The SBI Laboratory has two FTIRs available in the Trace Evidence Section:

Perkin-Elmer 1725X with a SpectraTech IR-Plan microscope.
Perkin-Elmer 2000 with a PE microscope

1. Paint samples may be prepared for FTIR analysis by slicing a thin cross-section or by slicing thin peels of each layer individually. The samples are then rolled out on a glass slide using the roller end of a roller knife. The prepared samples are transferred to the surface of a KBr plate.

2. The KBr plate with the prepared paint sample is placed on the stage of the FTIR microscope and transmission IR data is collected in accordance with the instrumental procedure for the particular FTIR employed.

B. Pyrolysis/Gas Chromatography (PGC)

Pyrolysis is utilized when greater discrimination of the paint binder constituents is needed. The pyrograms may show minor components that are not visible by FTIR. However, PGC may not be appropriate for samples that are contaminated or present in insufficient quantity for analysis.

1. The quartz sample tubes are cleaned for approximately one minute over a Bunsen burner and placed in a clean beaker to cool.

2. The paint samples are cut to the appropriate size and placed in a foil. The foil is folded and placed into the quartz tube. The procedures manual for this method contains a diagram of how the sample is to be prepared. (See JHP-22 Curie-Point Pyrolyzer Instrument Manual).

3. A blank must be run prior to introducing any sample.

4. Each item should be run at least two times to ensure reproducibility. A blank must be run prior to each new item.

C. Scanning Electron Microscopy/Energy Dispersive Spectrometry (SEM/EDS).

Many paint samples contain one or more inorganic extenders and/or hiding pigments. Although most of these can be detected by FTIR, some cannot. Also, if a paint contains several inorganic components, one or more may be masked.

1. Some samples, such as a clean, single-layer paint film, may not require any preparation. However, if more than one layer is present or if the sample is not clean, it may be sliced in a cross-section or peel as appropriate.

2. The prepared sample is placed on an SEM mounting stub and submitted to one of the SEM operators for analysis.

D. Microspectrophotometry

Colorimetry is used to discriminate the color of visually similar paint samples. Microspectrophotometry is required to provide colorimetric data for most forensic paint comparisons due to the typically small size of the specimens.

1. Diffuse reflectance (DR)

    a. The surface of a paint layer is cleaned or otherwise prepared for DR. The sample is placed on a glass slide and data collected in accordance with the instrumental procedure method for the Nanometrics Nano 100 UVIR Microspot Reflectometer Microspectrophotometer.

2. Transmission microspectroscopy

    a. The paint sample is sliced in a thin section by microtome or razor blade or scalpel blade and placed on a glass slide. The data is collected in accordance with the instrumental procedure method for the Nanometrics Nano 100. If a microtome is not available, care must be taken to make the paint slices as uniform as possible.

E. X-ray Fluorescence (XRF)

This procedure is seldom used for paint analysis but may be a useful complement to SEM/EDS.

1. Samples are submitted to an analyst who is trained in XRF.

F. X-ray Diffraction (XRD)

The procedure requires a larger amount of sample than what is typically available in a paint evidence case. However, if sufficient sample exists, XRD is useful for determining precisely which inorganic compounds are present.

1. Samples are submitted to an analyst who is trained in XRD.

Supervisor Approval _____ Date _____

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