



Standard Guide for Forensic Paint Analysis and Comparison¹

This standard is issued under the fixed designation E1610; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 Forensic paint analyses and comparisons are typically distinguished by sample size that precludes the application of many standard industrial paint analysis procedures or protocols. The forensic paint examiner must address concerns such as the issues of a case or investigation, sample size, complexity and condition, environmental effects, and collection methods. These factors require that the forensic paint examiner choose test methods, sample preparation schemes, test sequence, and degree of sample alteration and consumption that are suitable to each specific case.

1.2 This guide is intended as an introduction to standard guides for forensic examination of paints and coatings. It is intended to assist individuals who conduct forensic paint analyses in their evaluation, selection, and application of tests that may be of value to their investigations. This guide describes methods to develop discriminatory information using an efficient and reasonable order of testing. The need for validated methods and quality assurance guidelines is also addressed. This document is not intended as a detailed methods description or rigid scheme for the analysis and comparison of paints, but as a guide to the strengths and limitations of each analytical method. The goal is to provide a consistent approach to forensic paint analysis.

1.3 This guide cannot replace knowledge, skill, or ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgment.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *Some of the methods discussed in this guide involve the use of dangerous chemicals, temperatures, and radiation sources. This guide does not purport to address the possible safety hazards or precautions associated with its application. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility*

of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory requirements prior to use.

2. Referenced Documents

2.1 *ASTM Standards:*²

D16 Terminology for Paint, Related Coatings, Materials, and Applications

D1535 Practice for Specifying Color by the Munsell System

E308 Practice for Computing the Colors of Objects by Using the CIE System

E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory

E2808 Guide for Microspectrophotometry and Color Measurement in Forensic Paint Analysis

E2809 Guide for Using Scanning Electron Microscopy/X-Ray Spectrometry in Forensic Paint Examinations

E2937 Guide for Using Infrared Spectroscopy in Forensic Paint Examinations

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide other than those listed in 3.2, see Terminology **D16**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *additive (modifier)*—any substance added in a small quantity to improve properties. Additives may include substances such as driers, corrosion inhibitors, catalysts, ultraviolet absorbers, plasticizers, etc.

3.2.2 *binder*—a non-volatile portion of a paint which serves to bind or cement the pigment particles together.

3.2.3 *coating*—a generic term for paint, lacquer, enamel, or other liquid or liquifiable material which is converted to a solid, protective and/or decorative film after application.

3.2.4 *discriminate*—to distinguish between two samples based on significant differences; to differentiate.

3.2.5 *discriminating power*—the ability of an analytical procedure to distinguish between two items of different origin.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.6 *known sample*—a coating sample of established origin.

3.2.7 *paint*—commonly known as a pigmented coating (see 3.2.3).

3.2.8 *pigment*—a finely ground, inorganic or organic, insoluble, dispersed particle. Besides color, a pigment may provide many of the essential properties of paint, such as opacity, hardness, durability and corrosion resistance. The term pigment includes extenders.

3.2.9 *questioned sample*—a coating sample whose original source is unknown.

3.2.10 *significant difference*—a difference between two samples that indicates that the two samples do not have a common origin.

4. Quality Assurance Considerations

4.1 A quality assurance program must be used to ensure that analytical testing procedures and reporting of results are monitored by means of proficiency tests and technical audits. General quality assurance guidelines may be found in “Trace Evidence Quality Assurance Guidelines” (1).³

5. Summary of Practice

5.1 Paint films are characterized by a number of physical and chemical features. The physical characteristics may include color, layer sequence and thickness, surface and layer features, contaminants and weathering. Chemical components may include pigments, polymers, additives and solvents. These features can be determined and evaluated by a variety of macroscopical, microscopical, chemical, and instrumental methods. Limited sample size and sample preservation requirements mandate that these methods be selected and applied in a reasonable sequence to maximize the discriminating power of the analytical scheme.

5.2 Searching for differences between questioned and known samples is the basic thrust of forensic paint analysis and comparison. However, differences in appearance, layer sequence, size, shape, thickness, or some other physical or chemical feature can exist even in samples that are known to be from the same source. A forensic paint examiner’s goal is to assess the significance of any observed differences. The absence of significant differences at the conclusion of an analysis suggests that the paint samples could have a common origin. The strength of such an interpretation is a function of the type or number of corresponding features, or both.

5.3 An important aspect of forensic automotive paint analysis is the identification of the possible makes, models and years of manufacture of motor vehicles from paint collected at the scene of a crime or accident. The color comparison and chemical analysis of both the undercoat and top coat systems requires knowledge of paint formulations and processes, collections of paint standards, and databases of color and compositional information.

5.4 The test procedure selected in a paint analysis and comparison begins with thorough sample documentation. Some features of that documentation are described in Practice E1492. Analysis generally begins with appropriate nondestructive tests. If these initial tests are inconclusive or not exclusionary, the examination may proceed with the selection of additional tests based on their potential for use in evaluating or discriminating the samples of interest, or both.

6. Significance and Use

6.1 This guide is designed to assist the forensic paint examiner in selecting and organizing an analytical scheme for identifying and comparing paints and coatings. The size and condition of the sample(s) will influence the selected analytical scheme.

7. Collection of Suitable Samples

7.1 The potential for physical matches between known and questioned samples must be considered before selecting the method of paint sample collection. Care should be taken to preserve the potential for a physical match.

7.2 Questioned Samples:

7.2.1 Questioned samples should include all loose or transferred paint materials. Sources of questioned samples can include tools, floors, walls, glass fragments, hair, fingernails, roadways, adjacent structures, transfers or smears on vehicles, or transfers to or from individuals such as damaged fabric with paint inclusions. Whenever possible, items with paint transfers should be appropriately packaged and submitted in their entirety for examination. If sampling is necessary, the procedures listed in “Trace Evidence Recovery Guidelines” (2) may be used. When paint evidence is recognized, every effort should be made to manually remove it before using tape lifts to collect other types of evidence. If paint is collected with tape lifts, one should be aware of the possible difficulty encountered when attempting to manipulate paint samples bearing adhesive residues. In addition, components of the adhesive could contaminate the paint sample and change its apparent chemistry.

7.2.2 Smeared transfers can exhibit mingling of components from several layers or films that could preclude application of some of the analytical methods discussed in this guide. Due to the difficulties associated with collecting smeared or abraded samples, the entire object bearing the questioned paint should be submitted to the laboratory whenever possible.

7.2.3 When contact between two coated surfaces is indicated, the possibility of cross-transfers must be considered. Therefore, if available, samples from both surfaces should be collected.

7.3 Known Samples:

7.3.1 When feasible, known paint samples should be collected from areas as close as possible to, but not within, the point(s) of damage or transfer. Due to the possible presence of cross transferred materials, these damaged areas are usually not suitable sources of known samples. The collected known samples should contain all layers of the undamaged paint film. Substantial variations in thickness and layer sequences over short distances can exist across a painted surface. This is particularly true in architectural paint and for automotive films

³ The boldface numbers in parentheses refer to the list of references at the end of this standard.

where the curves, corners, and edges are often impact points and may have been subjected to previous damage, sanding or over-painting. If necessary, several known paint samples should be taken to properly represent all damaged areas because different areas of the painted surface may contain different paint systems. Known paint samples collected from different areas should be packaged separately and labeled appropriately.

7.3.2 When possible, the surface underlying the suspected transfer area should be included for analysis. Sections adjacent to a suspect transfer area can be valuable for assessing questioned and known sample differences and evaluating the possible cross transfer of trace materials. Wall and ceiling, door and window, implement handle and automobile door, fender and hood are examples of adjacent items.

7.3.3 Paint flakes can be removed from the parent surface by a number of methods. These include, but are not limited to, lifting or prying loosely attached flakes, cutting samples of the entire paint layer structure using a clean knife or blade, or dislodging by gently impacting the opposite side of the painted surface. When cutting, it is important that the blade be inserted down to the parent surface. It should be noted that no one method of sampling should be relied upon exclusively.

8. Procedure

8.1 Discussions of forensic paint analysis are provided in dated but detailed form by Crown (3), and more recently by Nielsen (4), Thornton (5), Maehly and Strömberg (6), Stoecklein (7), Caddy (8), and Ryland and Suzuki (9).

8.2 A reasonable scheme for forensic paint examinations is outlined in Figs. 1-4. Potentially useful techniques for the discrimination of paint binders, pigments, and additives are listed. The major steps in Fig. 1 are numbered to correspond to the discussions presented in this guide (for example 8.8, Solvent Tests). For any given comparison, not all the techniques listed in the same area in Fig. 1 are necessarily required. Sample size, condition and layer structure complexity should be considered when determining which techniques to use. The forensic coatings examiner should always use the more specific and least destructive tests prior to those that require more sample preparation or consumption. A review of the general technique descriptions, listed in 8.8 – 8.15, will provide guidance for the selection of appropriate methods.

8.3 Fig. 1 does not imply that other examinations should be excluded or that the order of the procedures in the chart is irrevocable. Samples that are neither constrained by amount

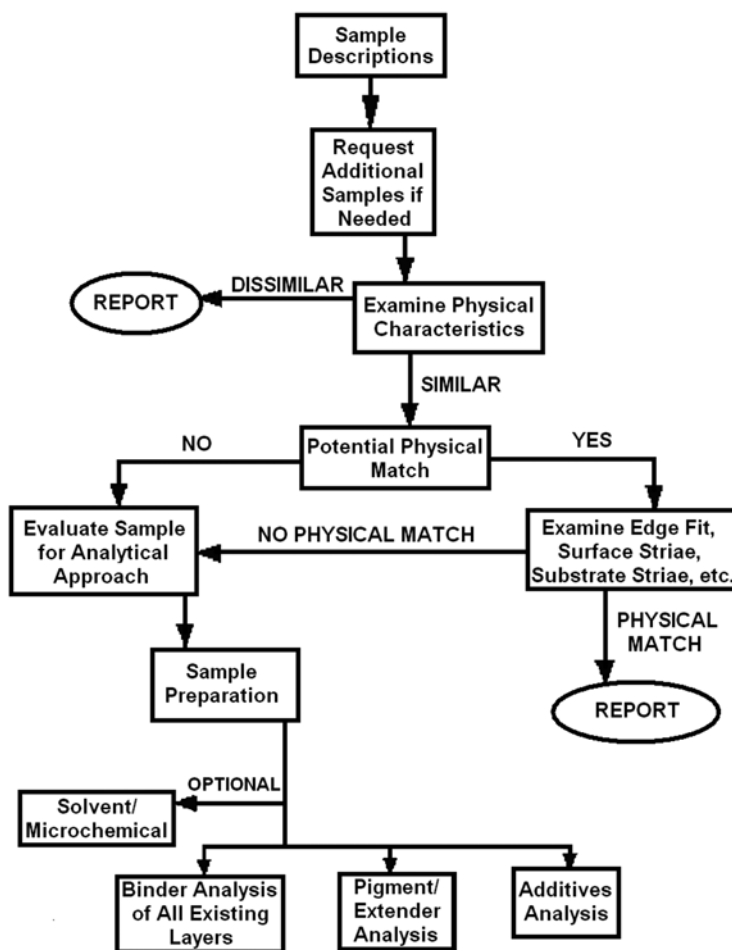


FIG. 1 Scheme for Forensic Paint Examinations

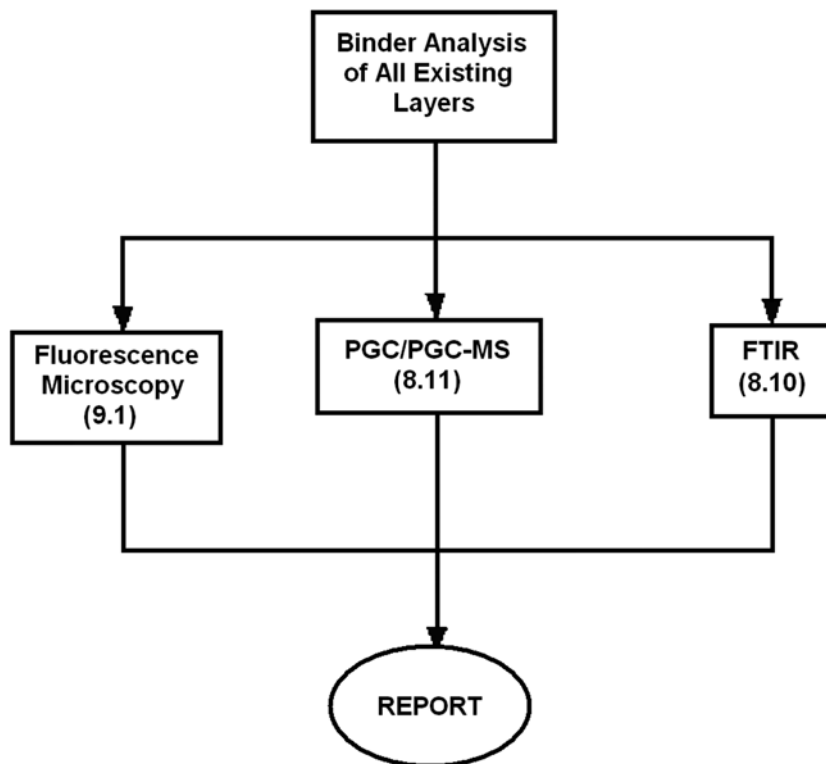


FIG. 2 Scheme for Forensic Paint Examinations

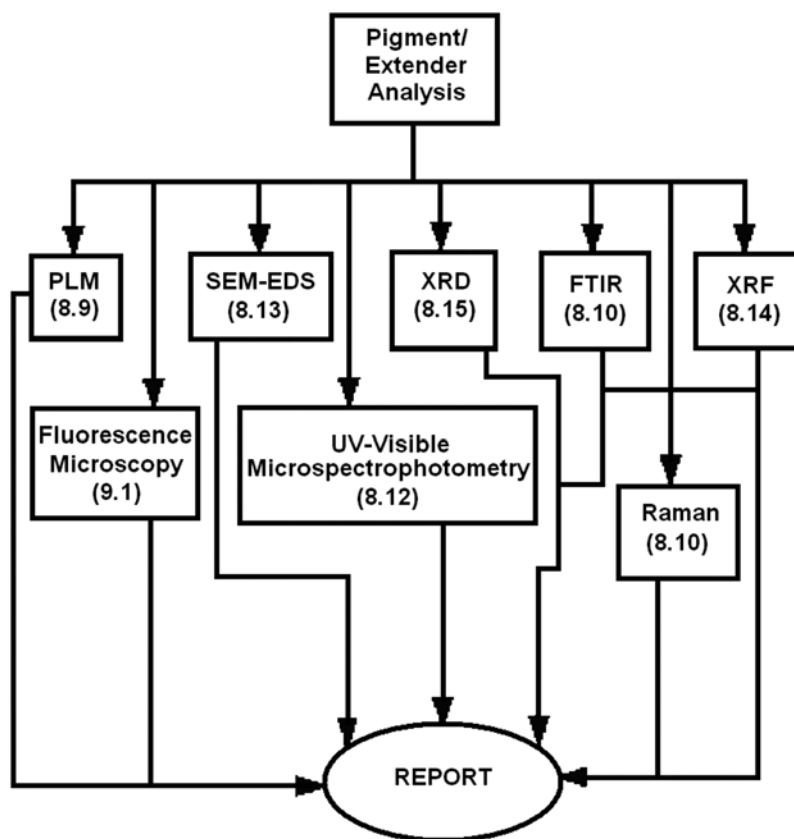


FIG. 3 Scheme for Forensic Paint Examinations

nor condition should be subjected to analyses that will determine the color and texture of the paint as well as the number,

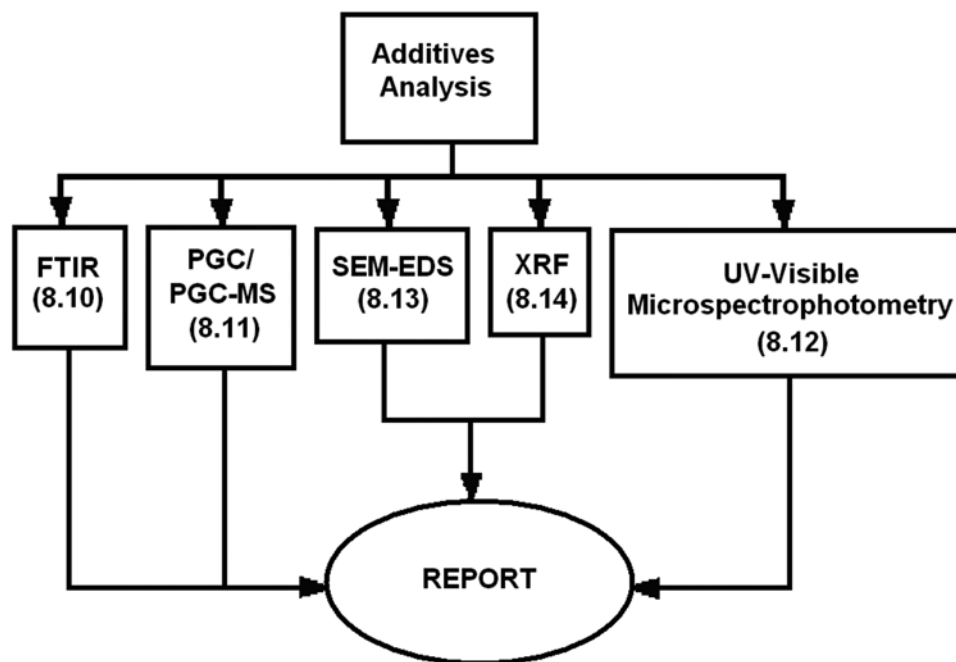


FIG. 4 Scheme for Forensic Paint Examinations

order, colors and textures of the layers in a multi-layered sample. In most cases, instrumental techniques should be employed to analyze and compare both the pigment and binder portions of the sample. A combination of techniques, which provide discrimination between as many types of paints and coatings as possible, should be used. These techniques should also be selected to provide classification and/or component identification information to be used in significance assessments. For samples that are limited in layer structure complexity, techniques for the comparison of both the binder and pigment portion of the coating must be used. The choice of techniques may change depending upon sample characteristics. For instance, pyrolysis-gas chromatography (PGC) may be utilized for identifying and comparing the binder portion of samples that exhibit a low binder concentration. Likewise, scanning electron microscopy-energy dispersive X-ray analysis (SEM-EDS), X-ray fluorescence (XRF) and X-ray diffraction (XRD) may be used for identifying and comparing the pigment portion of samples that exhibit a low pigment concentration.

8.4 The flow-chart in Fig. 5 is a guide to the determination of the possible origins of a motor vehicle paint. It is usually possible to differentiate a motor vehicle repaint from the original equipment manufacturer (OEM) paint by microscopical examination of the layer structure. If no OEM paint is present, then only the vehicle color (or partial vehicle color) can be reported. For OEM paint, both the topcoat and undercoat layers can be useful in identifying manufacturer, model and year. Both finish coat colors and primer colors can provide complementary information, since not all finish coat colors may be used for the period a particular primer system was employed. In most cases a range of possible makes/models/years will be generated by the search. Further specific information can often be developed through chemical analysis of the individual layers. Many of the techniques shown in Figs.

2-4 can be used, depending on the databases available. Reference collections and databases include books of color chips produced by automotive refinish paint manufacturers for use by body shops and automotive repair facilities, manufacturer topcoat and undercoat color and chemical standards, “street” samples collected from damaged motor vehicles, OEM information on paint formulations and collections of infrared spectra or pyrograms of known paints. Examples of these include the Royal Canadian Mounted Police (RCMP) Paint Data Query (PDQ) database and the National Automotive Paint File (NAPF) which is maintained by the Federal Bureau of Investigation (FBI).

8.5 Sample Description:

8.5.1 The initial evaluation should begin with a critical review of each samples’ chain of custody, package sealing, identification markings, and any potential cross-contamination between samples. If the items are found to be suitable for further evaluation, a detailed accounting and description of the paint sample and any co-mingled material should be documented.

8.5.2 The first step in forensic paint analysis is the visual evaluation, description, and documentation of the original condition of the sample(s). This involves describing the general condition, weathering characteristics, size, shape, exterior colors, and major layers present in each sample. This description can be accomplished by examining each item using a stereomicroscope. In some instances, this may be the final step in an analysis if exclusionary features or conditions in the sample(s) are identified.

8.5.3 Written descriptions, sketches, photography or other imaging methods must be used to document each sample’s characteristics. The goal is to produce documentation that will be meaningful to a reviewer. The resulting notes must be

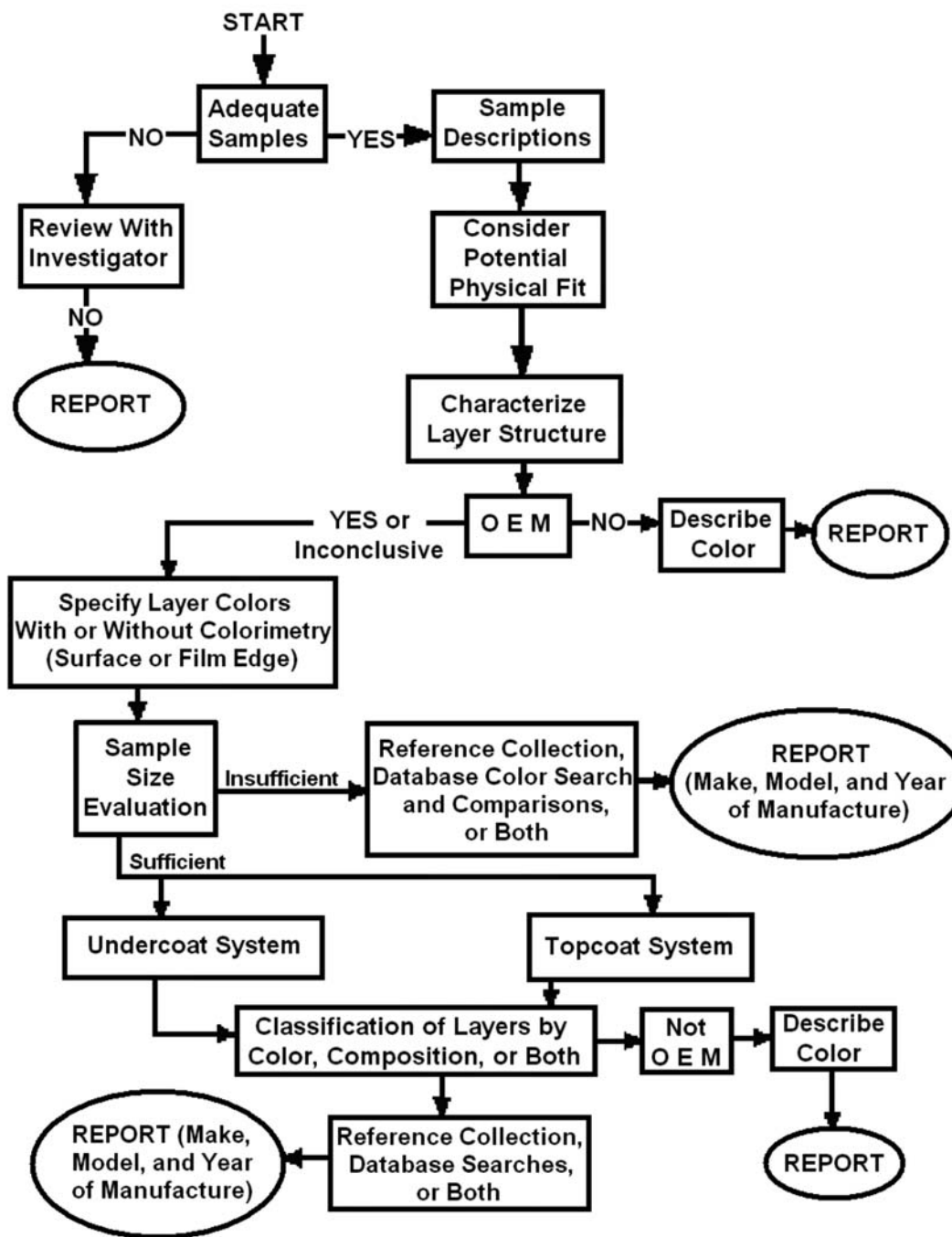


FIG. 5 Guide to the Determination of the Possible Origins of a Motor Vehicle Paint in an Investigative Case

sufficient to document the conclusions reached in the examiner's report. Although documentation is discussed at this point in this guide, it is an essential part of all steps in an analysis.

8.6 Physical Match:

8.6.1 The most conclusive type of examination that can be performed on paint samples is physical matching. This may involve the comparison of edges, surface striae, or other surface irregularities between samples or between samples and an area on a damaged object e.g. a motor vehicle. Additional comparisons can be attempted between surface features on the

underside of paint samples and a substrate. The corresponding features must possess individualizing characteristics.

8.6.2 Physical matches must be documented with descriptive notes. Photography, phototransparency overlays or other appropriate imaging techniques should be used. The resulting images should contain measuring scales and be retained as part of the documentation.

8.7 Sample Preparation and Layer Analysis:

8.7.1 The layers in a paint film are identified by viewing sample edges at magnifications ranging between 5× and 100×.

The more obvious layers are generally visible without sample preparation. Definitive paint layer system characterization usually requires sample preparation techniques such as manual or microtome sectioning and/or edge mounting and polishing. A combination of techniques may be required to fully characterize the layer structure. The extent of sample manipulation and preparation will depend on the amount of paint available and its characteristics.

8.7.2 Paint layer structure can be observed by using a scalpel or razor blade to prepare a thin section. An oblique cut through a sample may enhance layer visualization and assist in the detection of layer inhomogeneities. Additionally, the separation of paint layers can be accomplished with a scalpel blade.

8.7.3 Observations of subtle differences in color, pigment appearance, surface details, inclusions, metallic and pearlescent flake size and distribution, and layer defects, may require microscopical comparisons of the edge, oblique cut and surface views of known and questioned paint samples. These comparisons must be carried out with both samples positioned side by side and in the same field of view.

8.7.4 Cross-sections (embedded or thin-section preparations) may provide additional information as to the layer sequence, layer thickness, color, pigment distribution, pigment size, and composition of the individual layers that may not be possible to obtain with gross examination. Embedded preparations can be prepared by polishing and/or microtomy. Thin-sections can be prepared using a variety of microtomy techniques. Examination and analysis of the cross-sections can be conducted using a variety of analytical techniques that may include light microscopy, UV-visible microspectrophotometry, infrared microspectrophotometry, and electron microscopy. Laing et al. (10), Allen (11), and Stoecklein and Tuent (12) offer a concise discussion of thin-section paint analysis.

8.8 Solvent/Microchemical Tests:

8.8.1 Solvent/microchemical tests have long been used for attempting to discriminate between paints of differing pigment and binder composition that are otherwise similar in visual appearance. They are described in the general references noted in 8.1. The tests are based not only on dissolution of paint binders but also on pigment and binder color reactions with oxidizing, dehydrating, and reducing agents.

8.8.2 Solvent/microchemical tests are destructive by their nature and should be used only in situations in which adequate sample is available.

8.8.3 Solvent/microchemical examinations should be applied to both questioned and known materials concurrently. The effects of various tests are recorded immediately and then at reasonable intervals for the duration of each test. It is desirable to apply such tests not only to intact paint films, but also to peels of each individual layer to avoid interaction with neighboring layers and to observe the dissolution process more critically.

8.8.4 Reactions such as softening, swelling, curling or wrinkling, layer dissolution, pigment filler effervescence, flocculation, and color changes are some of the features that may be noted. The results of these tests are inherently difficult to quantify. Therefore, they are primarily used for preliminary classification and comparison.

8.9 Polarized Light Microscopy (PLM):

8.9.1 PLM is appropriate for the examination of layer structure as well as the comparison and/or identification of particles present in a paint film including, but not limited to, pigments, extenders, additives, and contaminants. Extenders, and other components of a paint film are generally of sufficient size to be identified by their morphology and optical properties using this technique. Although some pigment particles are too small for definitive identification by this method, exclusionary features may still be evident between samples.

8.9.2 Suitable samples for examination by PLM include, but are not limited to, thin peels, thin sections, pyrolysis and low temperature ashing residues, sublimation condensates and dispersed particles in a solvent, oil or other mounting medium.

8.9.3 The use of PLM for the identification of paint components requires advanced training and experience. Preparation and identification of paint components by PLM are discussed by McCrone (13) and Kilbourn and Marx (14).

8.10 Vibrational Spectroscopy:

8.10.1 Infrared Spectroscopy (IR) may be used to obtain information about binders, pigments and additives used in various types of coating materials (Guide E2937). Because the paint fragments to be analyzed are often quite small, a beam condensing or focusing device is normally required. Both transmittance and reflectance techniques may be used for the analysis of coatings, but in most cases, transmittance methods are preferred because all the sampling wavelengths are subjected to the same pathlengths and most of the reference data of coatings, binders, pigments and additives consist of transmittance spectra. In addition, transmittance data are not significantly affected by collection parameters such as type of refractive element used, angle of incidence chosen for analysis, or the degree to which the sample makes contact with the refractive element. These factors affect spectra obtained using internal reflectance methods.

8.10.2 If a multiple layer coating system is to be subjected to an infrared examination, optimal results can be obtained if each layer is analyzed separately. Methods that use solvents to assist in the sample preparation should be used with caution because they might alter the sample or result in the production of residual solvent spectral absorptions.

8.10.3 An infrared microscope accessory permits the analysis of a small sample or a small area of a sample. Samples of individual layers can be prepared manually using scalpels, blades, needles, forceps or other similar tools. Peels or sections can be placed on a salt plate or appropriate mount for analysis. The infrared microscope accessory may also be used to sequentially sample individual layers of a multiple-layer coating system that has been cross-sectioned. Generally, it is desirable to press such a sample after sectioning to produce a wider width for each layer and to produce a more uniform thickness. The aperture for an individual layer should be chosen so that its edges are as far from the adjacent layers as practicable. This minimizes the amount of stray light produced by diffraction that may be detected. All spectra of individual layers should be examined to determine if absorptions of adjacent layers are contributing to the spectrum.

8.10.4 Certain types of coatings, including automotive undercoats and many types of architectural coatings (especially those with low luster finishes), usually contain significant amounts of inorganic pigments. These pigments tend to have most of their significant infrared absorptions in the lower frequency spectral regions, and several have all of their absorptions in the region below 700 cm^{-1} . A FT-IR spectrometer equipped with cesium iodide (CsI) optics and a deuterated triglycine sulfate (DTGS) detector can collect spectral data to 220 cm^{-1} . The DTGS detector is less sensitive than the mercury cadmium telluride (MCT) detector used with IR microscopes, and the DTGS detector also requires a longer time to acquire each spectrum. CsI optics suffer the disadvantage of lower energy throughput compared to potassium bromide (KBr) optics. Because of these factors, a far IR instrument requires longer analysis times.

8.10.5 Transfers of coatings resulting in smears on various substrates may be sampled in situ using an appropriate attenuated total reflectance (ATR) accessory or an ATR objective on an infrared microscope. As a control, the substrate itself (assuming it is not a metal) should also be analyzed to verify that its absorptions are not contributing to the spectrum of the smear. Any contributions from the substrate should be considered. If the substrate is a metal, or highly reflecting, it may be possible to obtain a reflection-absorption spectrum of the smear using the reflectance mode of an infrared microscope accessory. This produces a double-pass transmittance spectrum of the material, and a background spectrum of the substrate itself (or uncoated mirror) should be used as a reference.

8.10.6 General information about the forensic analysis of coatings using infrared spectroscopy is discussed by Ryland and Suzuki (9), O'Neill (15), Suzuki (16), and Ryland (17). Forensic infrared microsampling of coatings using a beam condenser is described by Tweed et al. (18), Rogers et al. (19), and Schiering (20). Analyses using infrared microspectroscopy are described by Wilkinson et al. (21), Allen (22), Bartick et al. (23), and Ryland (17). The identification of specific binders, pigments and additives using infrared spectroscopy is described by Rodgers et al. (19,24,25), Norman et al. (26), Ryland (17), Suzuki et al. (27-29), and Washington State Crime Laboratory (30). Infrared spectral data for a number of binders, pigments, additives and solvents are presented in a compilation produced by the Federation of Societies for Coatings Technology (31).

8.10.7 Raman spectroscopy can also be used to obtain information about binders, pigments and additives used in coatings. Because this technique is based on light scattering rather than absorption, Raman spectra provide information that is complementary to that produced by infrared spectroscopy. Some paint components, for example, may give rise to both infrared absorption bands and Raman bands, but the relative absorption or scattering intensities of these bands will differ significantly between the two techniques. Other paint components may have vibrational modes that produce no infrared absorption bands, but may produce Raman bands. In addition, Raman spectroscopy can be useful for the analysis of inorganic

pigments and additives since, like far-infrared spectroscopy, it can provide information about low frequency vibrational transitions.

8.10.8 In most cases, Raman instrumentation using near infrared lasers will be needed to avoid strong fluorescence produced by various paint components. Because near-infrared excitation produces considerably weaker Raman scattering than visible excitation, dispersive instruments equipped with diode array detection systems or Fourier transform Raman spectrometers are recommended. Some applications of Raman Spectroscopy for the analyses of coatings are discussed by Kuptsov (32), Claybourn et al. (33), Bell et al. (34-36), Buzzini et al. (37 and 38), Suzuki (39), and Massonnet and Stoecklein (40).

8.11 *Pyrolysis Gas Chromatography (PGC):*

8.11.1 Pyrolysis gas chromatography (PGC) is a destructive technique that uses pyrolytic breakdown products to compare paints and to identify the binder type. As noted by Burke et al. (41), Fukuda (42), Ryland (17), Cassista and Sandercock (43), Plage et al. (44), and Burns and Doolan (45 and 46), this method of analysis may offer improved discrimination of chemically similar paints. Several pyrolysis systems and techniques are available to the forensic scientist and are discussed in overviews by Blackledge (47), Challinor (48), Saferstein and Manura (49), Irwin (50), and Wampler (51).

8.11.2 Pyrograms, the chromatograms of the pyrolytic products, are influenced by numerous sample characteristics and instrumental parameters. These may include sample size, shape and condition, ramping rates, final pyrolytic temperature, type of capillary column(s), gas flow rates, temperature programs and detector type(s). The resulting patterns of peaks in the known and questioned sample pyrograms are used for comparison purposes. If pyrolysis and chromatographic conditions are kept constant over time, then PGC can be used as an aid in the characterization of binder types by comparison with pyrograms of paints or resins from a reference collection.

8.11.3 The applicability of this technique depends on the paint type, layer complexity and the amount of sample consumption that can be tolerated. PGC analysis may be conducted with as little as 5 to 10 μg of sample. Run times are typically 20 to 40 min in duration. PGC is best applied to individual paint layers for purposes of both binder classification and comparison. Multi-layered paint samples, layer thickness variations, sample orientation in the pyrolysis accessory, and incomplete pyrolysis make reproducible pyrograms more difficult to obtain.

8.11.4 The user must ensure that reproducibility is maintained and that there is no sample carryover between runs. The necessity and frequency of replicate and blank runs must be established for each system and sample type.

8.11.5 The identification of pyrolysis products may be accomplished by pyrolysis gas chromatography-mass spectrometry (PGC-MS). Besides the detection of binder components, the reconstructed total ion chromatogram may contain information about additives, organic pigments and impurities. McMinn et al. (52), Challinor (53), Wright et al. (54), and Wampler et al. (55) provide discussions of mass spectrometric detection for PGC.

8.11.6 Information about the binder composition of some samples can be increased if the paints have been derivatized during pyrolysis. The use of derivatizing reagents such as tetramethyl-ammonium-hydroxide (TMAH) is discussed by Challinor (56-58).

8.12 *Microspectrophotometry:*

8.12.1 Color analysis has a long history in the pigment, paint, dyestuff, and fabric industries and has led to numerous approaches to color measurement and description. Absorption spectroscopy to discriminate the color of visually similar or metameric paint samples is discussed by Cousins (59). Colors can be described by systems such as those of Munsell and the Commission International de l'Eclairage (CIE), as described by Guide E2808 and Test Methods D1535 and E308. These systems can be used to classify colors for database systems, but usually absorption spectra of known and questioned samples are directly compared in forensic color comparisons.

8.12.2 Microspectrophotometry may be used to provide objective color data for paint comparison due to the typically small size of samples. The technique can be applied to paint films using either reflectance or transmittance measurements. Refer to Guide E2808.

8.12.3 Reflectance measurements of outer paint surfaces are affected profoundly by surface conditions such as weathering, abrasion, contamination, and texture. Careful reference sampling is essential to the success of color comparisons of such surfaces.

8.12.4 Reflectance can also be used on the edges of thin paint layers much as it is on outer paint surfaces. Before analysis, questioned and known samples can be mounted side-by-side on edge and polished to a smooth surface using a polish of 3 micron grit size or less. Microtomed samples without surface defects may be used without polishing. The requirement for consistent surface finish characteristics for all samples is more readily achieved if the known and questioned samples are mounted and prepared in a single mount.

8.12.5 When required for the discrimination of similarly colored paint layers, the surface finish of a polished sample analyzed in reflectance mode must approach the size of the smallest pigment particles present.

8.12.6 Transmission microspectrophotometry offers a more definitive form of color analysis for paint samples. Consistent sample thickness and choice of measurement size and location are essential for meaningful comparisons. Although thin cross-sections can be manually prepared, improved reproducibility can be achieved using a microtome. Even when using a microtome, the slice thickness, blade angle, cutting speed, lubrication, and mounting block stiffness or resilience must be selected and controlled carefully. A discussion of these parameters is presented by Derrick (60).

8.13 *Scanning Electron Microscopy:*

8.13.1 Scanning electron microscopy/energy dispersive X-ray analysis (SEM/EDS) can be used to characterize the morphology and elemental composition of paint samples. The SEM rasters an electron beam over a selected area of a sample, producing emission of signals including X-rays, backscattered electrons, and secondary electrons. Emitted X-rays provide information regarding the presence of specific elements, and

the electron signals permit compositional and topographical visualization of a sample.

8.13.2 X-rays are produced as a result of high energy electrons creating inner shell ionizations in sample atoms, with subsequent emission of X-rays characteristic of those atoms. Detection of elements with atomic numbers ≥ 4 is possible using a detector with a thin film window or a windowless detector. Analysis can be performed in a rastered beam mode for bulk layer analysis, or static beam (spot) mode for individual particle analysis. Goldstein et al. (61) present a general treatment of all aspects of SEM and X-ray microanalysis. Discussion specific to forensic paint examinations is found in Guide E2809.

8.13.3 Comparison of the composition of layers is generally performed by a non-quantitative method, such as direct spectral comparison or peak ratioing. Because accurate quantitative EDS requires sample homogeneity to a level of several microns, quantitative methods are not generally used for paint analysis. In order to produce a representative spectrum of a paint layer, the summing of spectra from multiple areas or the use of a beam raster area larger than 30 square microns may be necessary. The homogeneity of household paints and their examination by SEM/EDS is discussed by Gardiner (62) and Wright et al. (63).

8.13.4 The analysis of individual pigment particles in paint layers by static beam (spot) analysis can be useful.

8.13.5 The depth from which X-rays are produced (the analytical volume) is dependent upon beam energy, composition and density of the sample, and energy of the X-rays. Generally, the primary X-ray spatial resolution obtained in the analysis of paint systems is less than 10 μm . Secondary X-ray fluorescence further enlarges the analytical volume beyond the scanned image area visible in the SEM image. Care must be taken to ensure that the EDS data generated are representative only of the paint layer of interest, or that any adjacent layer contributions are reproducible.

8.13.6 Because a wavelength dispersive spectrometer (WDS) generally has better spectral resolution, lower detection limits, and superior light element detection capability than EDS, its use can supplement EDS to more completely characterize the elemental composition of paints. For example, WDS may resolve overlapping Ti K and Ba L lines, not possible by EDS. Because WDS has critical X-ray focusing requirements, the sample analyzed must generally be flat and the analysis area must generally be smaller than that allowed for EDS. Goldstein et al. (61) present a complete discussion of wavelength and energy dispersive spectrometers.

8.13.7 The elemental composition of paint smears that cannot be lifted from a substrate can often be estimated by subtraction of the substrate's X-ray spectrum from the combined smear-substrate spectrum. However, co-mingling of the smeared paint with substrate surface contaminants, the low mass of the smear, and typical inhomogeneity of paint can produce significant deviations of the smear spectrum from that of the original paint.

8.13.8 Backscatter electron images, relying on contrast due to atomic number differences, assist in the characterization and comparison of the structure of paints, including layer number,

layer thickness, distribution and size of pigment particles, and the presence of contaminants.

8.14 *X-Ray Fluorescence Spectrometry (XRF):*

8.14.1 XRF is an elemental analysis technique based upon the emission of characteristic X-rays following excitation of the sample by an X-ray source. XRF analysis is less spatially discriminating than SEM/EDS due to its larger analytical beam size and the greater penetration depth of X-rays compared to electrons. However, the limits of detection for most elements are generally better than for SEM/EDS, and the higher energy X-ray lines produced by higher energy excitation typical of XRF can be useful for qualitative analysis.

8.14.2 Because of the significant penetration depth of the primary X-rays, XRF analysis will generally yield elemental data from several, if not all, layers of a typical multilayer paint fragment simultaneously. Since variations in layer thickness may cause variations in the X-ray ratios of elements present, this technique can be used only comparatively or qualitatively. Fischer and Hellmiss (64) present a general discussion of the forensic applications of X-ray fluorescence. Howden et al. (65) discuss XRF analysis as applied to single layer household paints.

8.15 *X-Ray Diffraction (XRD):*

8.15.1 XRD is a non-destructive technique for the identification of the crystal form of pigments and extenders/fillers. This method is usually not suitable for the identification of organic pigments. X-ray diffraction techniques for the analysis of paint compounds are discussed by Snider (66).

8.15.2 XRD instruments usually employ a copper target X-ray tube to generate the X-ray beam, and a diffractometer to measure both the diffraction angles and peak intensities characteristic of the crystal structure. Beam/sample geometry is critical in producing the correct diffraction pattern.

8.15.3 Commercially available databases of diffraction patterns of crystalline materials can be used to facilitate qualitative analysis. Since the diffraction pattern of a mixture may be difficult to interpret, the identification of each component may require information provided by other analytical techniques such as elemental analysis.

8.15.4 Most paints need no sample preparation; however, the minimum sample size required is greater than for most common paint analysis techniques (for example, FTIR, SEM/EDS, XRF). Individual layer analysis is preferred over multi-layer or bulk analysis in order to associate components to their respective layers.

9. Other Techniques

9.1 *Fluorescence Microscopy:*

9.1.1 Fluorescence microscopy of thin or bulk cross sections, as an aid in differentiating samples or various layers within intact paint fragments, is discussed by Stoecklein and Tuent (12). When using an excitation wavelength of 365 nm, the technique may be sensitive to differences in organic pigments, additives, and film forming components. Allen (67) reports it to be most useful with light colored architectural coatings.

9.2 *Low Temperature Ashing:*

9.2.1 The low-temperature asher is a device in which an oxygen plasma is used to remove organic materials from a complex matrix. Materials that produce volatile oxides (principally organic components) are removed from the matrix with minimal elevation of the sample temperature, in contrast to pyrolysis systems. Ashing usually continues until all such volatile oxides are removed.

9.2.2 Inorganic pigments, extenders and some additives in the different layers of the ashed paint film will remain after the organic material is volatilized. The relative size and morphology of the remaining particles helps to identify and separate these residual components for additional analysis. A brief description of the technique is provided by McCrone and Delly (68) and Brown (69).

9.2.3 Ashing residues can be analyzed by a variety of methods, including PLM, SEM-EDS, or XRD techniques.

9.3 *Solvent Extraction:*

9.3.1 Solvent extraction can be used to separate some of the organic components from paint films, depending on the paint system(s) in question. The objective of the procedure is to recover a solute that can be examined by IR, GC, or GC-MS techniques. It is especially useful for coatings such as architectural latex paints and some marine finishes where the volume of pigments or extenders is very high. The identification of the binders by FT-IR is often only possible after separation of the pigments and extenders.

9.3.2 When using solvent extraction, separation of paint layers is very important. If this is impossible, it is important that identical conditions (for example, time and temperature) be applied to both the known and questioned samples.

10. Keywords

10.1 forensic science; instrumental analysis; paint; paint comparison

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