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Standard Guide for Test Methods for Forensic Writing Ink Comparison[1]

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INTRODUCTION

This guide is intended to be a general guide for forensic ink examinations, both for the experienced document examiner **(E 444)** and for those unfamiliar with previously reported procedures. The aim is to include those techniques that will provide the most information about an ink with the least damage to the document. Therefore, this guide refers to well-reported and thoroughly tested techniques currently in use by document examiners in general practice and dedicated forensic ink comparison facilities.

By following the procedures outlined here, an examiner can accurately discriminate ink formulas and reduce the possibility of false matches of ink samples from different sources or incorrect differentiation of ink samples with a common origin.

1. Scope

1.1 This Guide is intended to assist forensic examiners comparing writing or marking inks. Included in this analysis scheme are the necessary tools and techniques available to reach conclusions as to the common or different origin of two samples of ink.

1.2 Identifying ink formulas as to their manufacturer or time of manufacture as well as performing ink dating examinations are beyond the scope of this guide.

1.3 *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 limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1535 Test Method of Specifying Color by the Munsell System[2]

E 131 Terminology Relating to Molecular Spectroscopy[3]

E 284 Terminology of Appearance[4]

E 444 Guide for Descriptions of Scopes of Work Relating to Forensic Sciences for Questioned Document Area[4]

2.2 *NIST Standards:*

NBS Standard Sample No. 2106 ISCC-*NBS Centroid Color Charts*[5]

NBS Special Pub. 440 *Color: Universal Language and Dictionary of Names*[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *chromatography*—a method of separating substances that is widely used in analytical and preparative chemistry. It involves the flow of a liquid or gas mobile phase over a solid or liquid stationary phase. As the mobile phase flows past the stationary phase, a solute will undergo repeated adsorption and desorption and move along at a rate depending, among other factors, on its ratio of distribution between two phases. If their distribution ratios are sufficiently different, components of a mixture will migrate at different rates and produce a characteristic pattern (chromatogram).

3.1.2 *fluorescence*—a process by which radiant flux of certain wavelengths is absorbed and reradiated nonthermally at other, usually longer, wavelengths. **(E 284)**

3.1.3 *infrared (IR)*—referring to radiant flux having wavelengths longer than the wavelengths of light, usually wavelengths from about 760 nm to about 3 mm. **(E 284)**

3.1.4 *light*—electromagnetic radiant energy that is visually detectable by the normal human observer, radiant energy having wavelengths from about 380 nm to about 780 nm. **(E 284)**

3.1.5 *luminescence*—the emission of radiant energy during a transition from an excited electronic state of an atom, molecule or ion to a lower electronic state. **(E 131)**

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[2] *Annual Book of ASTM Standards*, Vol 06.01.

[3] *Annual Book of ASTM Standards*, Vol 03.06.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Available from U.S. Department of Commerce, National Bureau of Standard Reference Materials, R. B311, Chemistry Building, Gaithersburg, MD 20899.

3.1.6 *metamers*—specimens differing in spectral reflectance but having colors that match in light of one spectral composition, when viewed by one observer, but may not match in light of other spectral compositions, or when viewed by another observer. **(E 284)**

3.1.7 *spectroscopy*—in the most general sense spectroscopy is the study of the absorption or emission of electromagnetic energy by a chemical species as a function of the energy incident upon that species.

3.1.8 *source*—an object that produces light or other radiant flux. **(E 284)**

3.1.9 *ultraviolet (UV)*—referring to radiant flux having wavelengths shorter than the wavelengths of light, usually wavelengths from about 10 nm to 380 nm.

3.1.9.1 *Discussion—Long-wave UV* usually refers to the spectral range of UV-A, with wavelengths from about 315 nm to 380 nm. *Short wave UV* usually refers to the spectral range of UV-C, with wavelengths from about 100 nm to 280 nm.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *ballpoint pen ink*—writing or marking media intended for use in a ball point pen. Typically, a thick, high viscosity ink with an oil, glycol or rubber base.

3.2.2 *dichroic filter*—a filter with two transmission bands. These bands are usually widely separated, and can be of significantly different size.

3.2.3 *gel pen ink*—writing or marking media intended for use in a "gel-type" roller pen. Gel pen inks constitute a unique class of non-ballpoint pen inks. Typically, an aqueous ink of high viscosity, capable of maintaining a stable dispersed or dissolved state of the coloring material even after a prolonged period and exhibiting high fluidity under a shearing force. The ink contains a coloring material (pigment or dyes), acid-modified heteropolysaccharide and aqueous medium (water and water-soluble organic solvent), in which water constitutes at least 50 % by weight. Due to the incorporation of pigments into these formulations, the procedures outlined in this guide for TLC evaluations will be of limited value.

3.2.4 *infrared luminescence (IRL)*—the emission of radiant energy during a transition from an excited electronic state of an atom, molecule or ion to a lower electronic state (fluorescence or phosphorescence, or both), where the spectrum of the excitation source is in the ultraviolet (UV) or visible region of the electromagnetic spectrum, or both, and the spectrum of the emitted energy is in the far red or infrared (IR) region of the electromagnetic spectrum.

3.2.5 *ink formula*—a precise recipe or set of ingredients and their quantities that the manufacturer specifies for the final ink product. These ingredients are colorants (dyes and pigments) and vehicle components (volatile solvents, resins, etc.).

3.2.6 *match between ink samples*—the inability to distinguish between ink samples at a given level of analysis.

3.2.7 *non-ballpoint pen ink*—writing or marking media intended for use in a writing or marking instrument other than a ballpoint pen, including a dip or fountain pen, porous point pen, roller pen, marking instrument, etc. Typically, a thin, low viscosity ink with a water or solvent base.

4. Significance and Use

4.1 Ink comparisons are usually performed to answer four basic categories of question: (*1*) whether an ink is the same (in formula) as that on other parts of the same document or on other documents; (*2*) whether two writings with similar ink have a common origin, that is, the same writing instrument or ink well; (*3*) whether the ink of entries dated over a period of time is consistent with that dating or indicates preparation at one time; (*4*) whether ink is as old as it purports to be **(1)**.[6]

4.2 The procedures set forth in this guide are directly applicable to giving a full answer to only the first of these four questions.

4.3 With regard to the second question, differentiation of formula (question one) would indicate a negative answer to this question, as would differentiation with any of the additional methods listed in Section 3. When dealing with contemporary inks, however, a match of ink samples involving agreement in all observable aspects of all the techniques considered in this guide, while consistent with common origin, would not be sufficient to support a definite opinion of common origin **(2)**. Contemporary ink rarely has sufficient individuality to support a determination of common origin at less than the manufacturing batch level.

NOTE 1—Contemporary mass-produced inks are usually distributed as a component in a complete writing instrument or in a cartridge. With such packaging the ink is not subject to the mixing of inks and exposure to environmental contamination that could individualize ink from a given ink well at a specific point in time **(1, 3)**. This sort of analysis, potentially useful in the examination of older documents or those prepared under certain circumstances, is beyond the scope of this guide, as is examination of the ink line to individualize the writing instrument that produced it based on its performance characteristics.

4.4 As to the third and fourth questions involving the age of ink, dating techniques for determining either the relative age of ink samples (from the same or different documents) or the absolute amount of time since the writing of an ink line are also beyond the scope of this guide.

4.5 However, regarding question three, it may be of great importance in a forensic situation involving writing dated over a period of time to determine that one or more than one ink formula is present, that the use of various ink formulas fits a pattern, that a particular ink formula matches samples of a known date, etc.

4.6 As to the last question, a limit as to the possible age of an ink entry can be inferred by establishing the date of first production of the ink formula. Although beyond the scope of this guide, identifying ink formulas as to their manufacturer or time of manufacture utilizes many of the analytical procedures described here. Specialized knowledge and experience on the part of the examiner, as well as access to a collection or *library* of ink reference samples is also required.

4.6.1 Such an ink library consists of samples of ink formulas from known sources, usually manufacturers of ink, or writing or marking instruments, or a combination thereof. The ink reference samples are usually cataloged, analyzed, and stored according to the methods described in Refs **(2, 4, 5** and **6)**. Even with access to a comprehensive collection, association of an unknown ink sample with a single known formula is not

[6] The boldface numbers in parenthesis refer to the list of references at the end of this guide.

always possible. This is because some ink formulas are not distinguishable, however, in most cases the analytical procedures outlined here are sufficiently discriminating that formulas are distinguishable.

4.7 Comparison of ink samples by analysts without an ink library can still provide valuable information. However, added significance can be given to the meaning of a match if the relative rarity or commonness of the ink formula is known. Familiarity with or access to a comprehensive reference collection of inks is useful for this purpose.

4.8 In expressing conclusions it should be remembered that a match indicates that the ink samples are of the same formula or of two similar formulas with the same nonvolatile components. The possibility that other analytical techniques might be able to differentiate them should always be considered **(2)**.

4.8.1 Therefore, conclusions in this situation should never indicate that two ink samples are "identical" or "the same ink," but must be limited to statements indicating "inability to distinguish the ink samples at this level of analysis" or "exhaustive chemical and physical testing failed to detect any differences between the ink samples" **(2)**.

5. Interferences

5.1 Most interferences with ink examinations come from variables that interact with the ink. These can be part of the writing process, such as blotting wet ink **(1, 2)**, or variations in the paper **(7)**, or various forms of contamination on the document **(7, 8)**, or a combination thereof. Simple precautions can usually avoid problems.

5.2 Note and record any differences in the substrate, such as the use of different paper for different documents or pages of a multipage document. Also note and record variations in the document, such as a signature written over a photograph on an identity document, multicolored paper with different dyes or colors of underprinting, intersections with printed or typed material, etc. **(7, 8)**.

5.3 The results of prior handling or testing should also be noted and recorded. These effects can include discoloration or fading from ageing, exposure to light or heat, as well as stains from food or drink, dirt or grease, perspiration or finger smudges, water, or chemicals, including ninhydrin or other reagents for visualizing latent friction ridge impressions, etc. **(7, 8, 9)**.

5.4 In optical examinations care should be taken to consider the potential effects of these variables **(7, 8)**. In chemical analyses paper blanks should be run as controls for these variables **(4, 5)**.

6. Reagents and Equipment

NOTE 2—It is important that all reagents are uncontaminated.

6.1 *Purity of Reagents*—Reagent Grade.

6.2 *Purity of Water*— Distilled or equivalent.

6.3 *Reagents for Spot Testing, Solubility Testing, and TLC Extraction Solvents*:

6.3.1 Pyridine.

6.3.2 Ethanol.

6.3.3 Water.

6.3.4 Other reagents as required by Refs (**1, 3,** and**23**).

6.4 *Reagents for Thin Layer Chromatography (TLC) Developing Solvents*:

6.4.1 *Solvent System I*— Ethyl acetate, ethanol, water (70 + 35 + 30).

6.4.2 *Solvent System II*—N-butanol, ethanol, water (50 + 10 + 15).

6.5 Other ink extracting solvents and developing solvents in accordance with Refs **(5, 6,** and **10)**.

6.6 *Equipment for Optical Examinations*:

6.6.1 *Stereomicroscope*:

NOTE 3—Five to one hundred power total magnification is a range that has been found useful.

6.6.2 *UV Lamps or View Box*, with both long-wave UV and short-wave UV lamps.

6.6.3 *Colored Filters*, (gelatin, colored glass, interference filters) as needed for visual and photographic differentiation of inks.

6.6.4 *Dichroic Filters*, See Ref **(11)**.

6.6.5 Photographic equipment with appropriate film, lighting, and filters for differentiation of ink samples.

6.6.6 Photographic equipment with appropriate film, lighting, and filters for recording reflected infrared (RIR) and infrared luminescence (IRL).

6.6.7 IR image conversion device or system with appropriate light sources and filters for use in RIR and IRL modes as well as appropriate photographic equipment, computer hardware and software for image acquisition or processing, or both.

6.6.8 *Barrier Filters for RIR and IRL*—Long pass filters, preferably sharp cut, that block visible flux. Suitable gelatin, colored glass, and interference filters are commercially available **(12, 13, 14)**.

NOTE 4—Since ink reactions can vary, it is advisable to use a series of filters with cut on wavelengths from the red through the IR range of the film or detector.

6.6.9 *Excitation Source for IRL*—Sources include: a continuous spectrum lamp with a filter to eliminate flux in the IR and far red region of the spectrum, for example, a 10 % to 15 % solution of copper sulfate in a cell with a 1 cm to 3 cm light path, or appropriate colored glass or interference filters; or lasers or other monochromatic sources.

NOTE 5—A variety of sources with different spectral distributions or a variety of filters on a continuous spectrum source may be helpful in discriminating ink samples.

When using a filtered source it is advisable to use a heat absorbing filter between the source and the filter. This both protects the filter **(15)** and eliminates a significant portion of the undesirable IR flux.

6.6.10 Photographic or other equipment for recording observations as required.

6.7 *Equipment for Spot Testing, Solubility Testing, and TLC*—It is important that all equipment is uncontaminated.

6.7.1 *Stereomicroscope* (See Note 2).

6.7.2 *Hypodermic Needle*, with an approximately 20 gage hollow boring point or blunted point, scalpel or similar sampling device.

6.7.3 *Disposable Vial or Transparent Sample Container*—1 dram or smaller suggested.

6.7.4 *Disposable Micropipettes*—10 µL or smaller suggested.

6.7.5 *Precoated Plastic or Glass Sheets/Plates of Silica Gel,* without fluorescent indicator (60 Å pore size[7]).

NOTE 6—It is recommended that the TLC sheets/plates be kept in a desiccator.

6.7.6 *Glass Developing Tank with Air Tight Cover*—This tank should be the appropriate size for the sheet/plate being developed.

6.7.7 *UV Lamps or View Box,* with both long-wave UV and short-wave UV lamps.

6.8 Appropriate equipment for the optional techniques listed in Section 3.

7. Procedure

7.1 *Light Examination*:

7.1.1 *Determine the Class of Ink*—Under ambient lighting conditions (natural or artificial), with or without the aid of magnification as required, determine whether the class of the ink is ballpoint pen or non-ballpoint pen **(6)**. Observe the overall appearance of the writing. Note and record anything that might provide information about the kind of writing or marking instrument used. For example, if there is an indentation down a central *track,* then the writing instrument may be a ballpoint pen or rolling ball marker. Double indentations may indicate a bifurcated nib dip pen or fountain pen. This step may be performed with the use of reference standards prepared with various classes of writing instruments on different substrata.

7.1.2 *Determine the Condition of the Ink and the Overall Appearance of the Writing*—Note and record the presence of anything that might have induced a change in the ink as described in Section 2; for example, stains, burns, aging, blotting, fading, attempts at mechanical erasure or chemical eradication, discolorations, etc.

7.1.3 *Determine the Color of the Ink*—Inks that are metamers can sometimes be differentiated by the use of illuminants with varying color temperatures or spectral characteristics, as well as by narrow band or laser illumination. Various filters can also be used for direct viewing, photography, or electronic viewing, including wide and narrow band, short and long pass, and dichroic filters **(1, 6, 11, 16)** .

NOTE 7—The use of standard color notation may be helpful in recording these observations. (NBS Standard Sample No. 2106, NBS Special Pub. 440)

7.1.4 Microspectrophotometry **(17)** can be useful in differentiating inks by measuring their wavelengths of maximum transmission or reflectance spectra, or both.

7.2 *Ultraviolet (UV) Examination*:

7.2.1 Observe the ink sample under both long-wave UV and short-wave UV sources. Note and record the fluorescence characteristics of the ink as well as the emission of any fluorescence **(18)**. (See Note 7.)

NOTE 8—Except for some red formulas, few inks fluoresce in their dried state on paper. A fluorescent halo is occasionally observed around an ink line; capillary migration of a vehicle component into the substrate is a known cause.

7.2.2 Note and record any effect of the substrate. Strong fluorescence of the paper may affect the observer's perception of the ink.

7.2.3 UV examination may reveal indications that the document has been stained by chemicals or other material that may effect the ink comparison as discussed in Section 2 **(7, 8, 9)**. These can include the detection of the use of chemical ink eradicators, and liquid or dry opaquing material, that may have significance beyond the ink comparison. These should be noted and recorded.

7.3 *Infrared (IR) Examination*:

7.3.1 Determine the Reflected Infrared (RIR) and Infrared Luminescence (IRL) characteristics of the ink: As these effects are beyond the range of human vision, some technological extension of the eye is required.

7.3.1.1 These characteristics may be photographed with IR sensitive film or observed directly with an IR image conversion device **(7, 8, 11, 15, 16, 19, 20, 21)**. With either system, a suitable barrier filter is required in front of the lens to block visible flux (see 6.6.8 and Note 4). For IRL a suitable excitation source will also be required (see 6.6.9 and Note 5).

NOTE 9—Both photographic and electronic systems work well; each has its advantages and drawbacks.

Photography provides a permanent, high resolution record of results and long exposures can capture faint luminescence. However, exposures can be long (up to 20 min. for faint luminescence), and considerable experience is required before dispensing with time consuming bracketing in a series of exposures using different filters **(19, 20)**. The amount of time required for processing and printing may also be a problem.

Electronic systems, including units with image conversion tubes and closed circuit television systems, have the advantage of real time results, facilitating optimization of filter combinations, focus, exposure, etc. **(21)**. These systems are well suited to screening batches of documents (such as passports) for alterations. However, resolution is limited, some faint luminescence may not be easy to detect, and separate photographic or electronic imaging equipment is required to record results. Modern integrating infrared video cameras are able to detect faint IR information that cannot be seen otherwise.

7.3.2 *Reflected Infrared (RIR)*:

7.3.2.1 Record the characteristics as opaque or transparent, indicating the degree of opacity. The more opaque the ink (the more it absorbs), the darker it will appear; the less opaque, the lighter it will appear, until it seems to be transparent or to drop out. An arbitrary four point scale of −3 to 0 (opaque to transparent) may assist in recording these observations.

7.3.3 *Infrared Luminescence (IRL)*:

7.3.3.1 Record the IRL characteristics of the ink relative to the substrate as darker, similar, or lighter, indicating degree as appropriate. Ink that luminesces more brightly than the substrate will appear lighter than the substrate; strongly luminescent ink may appear to glow brightly. If ink does not luminesce or does not luminesce as brightly as the substrate, the ink will appear darker than the substrate (this is sometimes referred to as *black luminescence* or *negative luminescence*). Ink that luminesces at an intensity similar to that of the substrate appears invisible, and is said to *drop out.* An arbitrary seven point scale of −3 to 0 to +3 (black to indistinguishable to very bright) may assist in recording these observations.

NOTE 10—Depending on the characteristics of the substrate and the combination of source or filters, or both, the appearance of ink samples

[7] Merck Silica Gel, Whatman PE SIL G, and Merck HPTLC Silica Gel 60 have been found satisfactory.