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Standard Guide for Writing Ink Identification[1]

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INTRODUCTION

This guide is intended as a general outline for use in forensic ink examinations, where the intention is to identify an ink formula or type. It is designed both for the experienced document examiner (see Guide E 444) and for those unfamiliar with previously reported procedures. The aim is to describe those techniques that will provide the most information about an ink with the least damage to the document. This guide refers to well-reported and thoroughly tested techniques currently in use by forensic document examiners, chemists, and other scientists.

Following the procedures as outlined, an examiner can accurately discriminate between ink formulas; as well as significantly reducing the possibility of reporting false matches of ink samples from different sources or incorrectly differentiating ink samples from a common source.

Identifications of ink formulas may be accomplished through the use of an adequate collection of standards. The necessary completeness of a comparison collection and limitations of conclusions will be addressed in the guide.

1. Scope

1.1 This guide covers assisting forensic examiners in identifying writing inks. Included in this analysis scheme are the necessary tools and techniques which have been successfully utilized to reach conclusions as to the common or different origin of two samples of ink.

1.2 *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:* [2]

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

E 1422 Guide for Test Methods for Forensic Writing Ink Comparison

2.2 *NIST Standards:*

NBS Standard Sample No. 2106 ISCC-NBS Centroid Color Charts[3]

NBS Special Pub. 440 Color: Universal Language and Dictionary of Names[3]

3. Terminology

3.1 *Definitions*—Terminology has been defined in Guide E 1422, with the following addition:

3.1.1 *ink library*—an organized collection of reference samples of inks and related materials.

3.1.1.1 *Discussion*—For maximum effectiveness in identification of questioned ink, an ink library should at minimum include the following elements: reference samples of ink in unused form, either in bulk samples from the manufacturer or in distribution form such as bottles, pens, or cartridges; dried ink specimens of each reference sample of ink placed on paper (scribble sheets); analysis results of each reference sample of ink, for example, TLC sheets/plates; and an ink information file for each reference sample of ink containing available relevant data. All elements of the collection should be as complete, comprehensive, and up-to-date as possible, although this will vary between ink libraries.

[1] This guide is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.02 on Questioned Documents.

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

4. Significance and Use

4.1 The reasons for identifying writing inks are to obtain information about: the origin; relative availability; distribution; and first and last (if applicable) production dates. It is this valuable information available from the manufacturer and through the use of a collection of standards that differentiates this guide from Guide E 1422.

4.1.1 The procedure set forth in this guide are applicable in determining the significance of a match obtained by performing the examinations set out in Guide E 1422 (by showing how rare or common an ink formula may be), or in determining the source of an ink. The identification of a specific ink formula can facilitate the determination of the first date of production and the discontinuance date of that ink.[4]

4.1.2 In addition to proficiency in the use of the necessary analytical procedures, specialized knowledge and experience on the part of the examiner are required.[5] Also required is a comprehensive collection of reference samples of ink and related materials (ink library). The ink reference standards are cataloged, analyzed, and stored according to the procedures described in Section 7.

4.2 Even with access to a comprehensive ink library, it is not always possible to positively identify a questioned ink sample. This is because some ink formulations are very similar; usually only non-volatile ingredients such as dyes and pigments are compared; and no matter how comprehensive the ink library is, the collection will never be complete.[6]

4.2.1 Some ink formulas are not distinguishable; they behave in the same manner under various examinations because they have similar formulas with the same nonvolatile components. Thus, it is not always possible to find a single reference ink sample in the ink library that matches a questioned ink. Even if one is found, it may not provide an identification unless the ink formula is shown to be unique because it contains a specific component. For these reasons, it will not be possible to identify every questioned ink. There is not always a forensic answer to a question at hand.

4.2.2 It must also be understood that it is not possible to create an all inclusive ink library, just as it would not be possible to obtain every fingerprint, or every paint, soil, or glass sample. Conclusions as to the identity of an ink are dependent on the completeness of the ink library used. Thus, it is possible that there are one or more inks not in the ink library that would be indistinguishable from the questioned ink.

4.3 In spite of these limitations, questioned inks can be associated with reference ink samples with a high degree of confidence using the systematic approach in this guide. The analytical procedures given here, such as TLC and TLC Densitometry, are sufficient to distinguish most inks, and therefore to match most questioned ink samples to a reference sample of ink or a relatively limited group of reference samples in an ink library.

4.3.1 Just as with other forensic tools, for example, FTIR, GC, HPLC, etc., pattern profile matching with reference samples is often sufficient to yield an identification. Individual component identification through an internal standard approach may be used, but is not usually necessary.[4]

5. Interferences

5.1 Most interferences with ink examinations and subsequent identifications are a result of variables interacting with the ink. These variables can usually be attributed to the writing process or storage conditions, or a combination thereof, and are discussed in Guide E 1422. Evaluation of these variables can avoid problems examinations.

5.2 Other interferences can be caused by changes to the TLC diffusion of fluorescent components, differences in the paper controls, differences in color due to fading either of the inks or of the components on the TLC sheet/plate, solvent depletion, or a combination of these and other factors. Evaluation of these variables, use of paper blanks, and proper storage and maintenance of the reference samples and related material in the ink library can avoid problems in examinations.

5.3 Large batch-to-batch variations in the manufacturing process can also lead to problems in evaluating a match.

6. Reagents and Equipment

6.1 Appropriate reagents and equipment for the required techniques have been listed in Guide E 1422, with the following additions:

6.1.1 *Low Resolution Precoated Plastic or Glass Sheets/Plates of Silica Gel*, without fluorescent indicator (60 angstrom pore size).

NOTE 1—Low resolution sheets/plates are generally not as sensitive to external effects, for example, temperature, humidity, and development conditions. They have the quality of exhibiting excellent reproducibility and as such are an appropriate choice for storage media of the ink library TLC plates.

6.1.2 *High Resolution Precoated Plastic or Glass Sheets/Plates of Silica Gel*, without fluorescent indicator (60 angstrom pore size).

NOTE 2—It is recommended that the TLC sheets/plates be kept in a desiccator.

7. Procedure

7.1 *Collection, Preparation, and Analysis of Reference Materials for the Ink Library*:

7.1.1 *Reference Samples of Ink*:

7.1.1.1 The core of the ink library consists of reference samples of ink formulas, usually obtained from ink manufacturers. Additionally, ink and pens should be purchased at retailers on a regular basis (at least once a year), because it is not always possible to obtain samples directly from all manufacturers of ink. Because of international trade and travel patterns, reference samples of ink should be obtained on a world-wide basis.

7.1.1.2 Accession information for each reference sample of ink should be recorded, such as date of acquisition, source, etc.

[4] Brunelle, R. L. and Pro, M. J., "A Systematic Approach to Ink Identification," *Journal of Official Analytical Chemistry*, Vol 55, 1972, pp. 823–826.

[5] Brunelle, R. L. and Cantu, A. A., "Training Requirements and Ethical Responsibilities of Forensic Scientists Performing Ink Dating Examinations," Letter to the Editor, *Journal of Forensic Sciences*, November, 1987.

[6] Crown, D. A., Brunelle, R. L., and Cantu, A. A., "Parameters of Ballpoint Ink Examination," *Journal of Forensic Sciences*, Vol 21, 1976, pp. 917–922.

For an assembly of reference samples of ink to be considered a collection rather than an accumulation, it must be organized and cataloged. If a computerized database is used, searching can be on any criteria; if not, the features noted in a light examination performed in accordance with Guide E 1422 can be used to organize the collection.

7.1.1.3 Reference samples of ink should be stored under optimal laboratory conditions (sealed containers, darkness, temperature and humidity controlled) to retard drying, oxidation, and other changes related to aging.

7.1.2 *Dried Ink Specimens*:

7.1.2.1 Prepare a specimen by making lines or marks on a sheet of paper (scribble sheet). Record the date of preparation. Allow the ink to dry for up to 1 h under ambient conditions before storing.

NOTE 3—Dried ink specimens can be effectively stored on filter type paper that does not contain optical brightener additives. A sample of any paper being considered for a library storage media should be analyzed following the laboratory procedures as indicated in this standard. This will determine if the paper will interfere with the examination procedure.

7.1.2.2 Dried ink specimens should be stored under optimal laboratory conditions (darkness, temperature and humidity controlled) to retard fading and other changes.

7.1.3 *Results of Analysis of Reference Samples*—Because questioned ink samples will be analyzed in accordance with Guide E 1422 for comparison with the ink library (see 7.2), the reference samples in the library should undergo the same analyses with results preserved for future searching.

7.1.3.1 Perform the light, ultraviolet (UV), and infrared (IR) examinations in accordance with Guide E 1422.

7.1.3.2 Perform the spot testing and solubility testing in accordance with Guide E 1422.

7.1.3.3 Perform the thin layer chromatography TLC examination in accordance with Guide E 1422.

7.1.3.3.1 Note and record the extraction solvent used. Where appropriate, prepare duplicate extractions using all the different solvents likely to be employed in extraction from various substrata. Prepare a TLC of each extract, recording the solvent used. Appropriate TLC sheets/plates will then be available for comparison with questioned samples.

7.1.3.3.2 The TLC analysis should be conducted on low resolution type sheets/plates. Low resolution sheets/plates are generally not as sensitive to external effects, for example, temperature, humidity, or development conditions. They have the quality of exhibiting excellent reproducibility and as such are an appropriate choice for storage media of the ink library TLC sheets/plates.

NOTE 4—Plastic backed 60 angstrom size silica gel without fluorescent indicator sheets/plates has been found to be satisfactory.

7.1.3.3.3 Ink library TLC sheets/plates should be stored under optimal laboratory conditions (darkness, temperature and humidity controlled) to extend the useful life of the sheets/plates. TLC sheets/plates have a limited useful life: the sheets/plates themselves will degrade after 10 to 20 years, and the band colors and fluorescence characteristics may fade or undergo other changes sooner. Deteriorating TLC sheets/plates should be replaced as needed.

7.1.4 *Ink Information Files*:

7.1.4.1 All available relevant data on each reference ink sample should be collected and maintained. This can include information on the manufacturer; ink formula; manufacturer's designation(s) and marketing name(s); other user's (for example, pen manufacturers) and their designation(s) and marketing name(s); volume of ink manufactured; area(s) of distribution; first production date; date first released to the public; last production date; etc.

NOTE 5—Some information may be considered proprietary by the ink manufacturer or other source. Such information should be treated with the appropriate confidentiality.

7.1.4.2 Analytical results and other data from 7.1.3 should be maintained. Efficient organization of this information can facilitate searches of the ink library.

7.2 *Ink Identification*—Ink identification is a two step process. The first step involves comparative analysis techniques described in Guide E 1422. The second step includes comparison of any resulting TLC plate from the initial analysis to an ink library.

7.2.1 Perform the light, ultraviolet (UV), and infrared (IR) examinations and record results in accordance with Guide E 1422.

7.2.2 Perform the spot testing and solubility testing and record results in accordance with Guide E 1422.

7.2.3 Perform the thin layer chromatography TLC examination in accordance with Guide E 1422.

7.2.3.1 The comparison reference inks in the ink library must have been extracted using the same solvent. If there is no TLC plate in the ink library that meets this requirement, prepare one in accordance with Guide E 1422 using the appropriate solvent before proceeding.

7.2.4 *First TLC Interpretation*:

7.2.4.1 Samples of ink with qualitatively different colorant compositions can be easily distinguished by comparison of the characteristics described in Guide E 1422.

7.2.5 *Comparison Against a Library of Standards*:

7.2.5.1 Where comparison against a library of standards is desired, the initial TLC analysis should be conducted on low resolution type sheets/plates of the same type used to prepare the TLC sheets/plates in the ink library.

7.2.5.2 Using the results of the light, ultraviolet (UV), and infrared (IR) examinations (see 7.2.1) search the library for samples known to produce these results. Physically compare the questioned ink sample in situ with the dried ink samples from the ink library. Note and record all ink library reference samples that are consistent with the questioned ink at this stage.

7.2.5.3 Physically compare the chromatogram of the questioned ink with the chromatograms of all the reference samples in the ink library that were not eliminated in 7.2.5.2. Observe the band colors, Rf separations, and fluorescence characteristics. Note and record all ink library reference samples that are consistent with the questioned ink at this stage.

7.2.5.4 Those reference samples that match at every level of the examination are selected as possible matches in preparation for the second TLC comparative examination.

7.2.5.4.1 Reference samples from the ink library having explicable differences should also be selected as possible matches. Such over-selection of standard inks reduces the possibility that a true match is not eliminated from consideration. Explicable differences include characteristics arising from diffusion of fluorescent components, differences in the paper controls, differences in color due to fading either of the inks or of the components on the TLC sheet/plate, solvent depletion, or a combination of these and other factors.

7.2.6 *Second TLC Analysis*:

7.2.6.1 Begin a second TLC comparison between the questioned ink and the potential matches from the ink library. This examination may further reduce the number of standard library inks that could match the questioned ink.

NOTE 6—The TLC sheets/plates used at this stage should be very high resolution. TLC sheets/plates that are high resolution are generally very sensitive both to their surroundings and to development conditions. The reproducibility within a plate is extremely good; however, plates should not be inter-compared due to potential variations.

7.2.6.2 Remove a suitable amount of sample from each of the reference ink samples in the ink library whose physical and chemical TLC results are consistent with the questioned ink's. There may be many potential library matches at this stage of the examination. Every potential match should be sampled.

7.2.6.3 Perform a TLC analysis in accordance with Guide E 1422.

NOTE 7—Glass backed 60 angstrom size silica gel without fluorescent indicator plates has been found to be satisfactory. Variations within plates of the same type and manufacturer have been noted.

NOTE 8—Spot all inks and the paper control samples (blanks) on the same plate. This is necessary based on the sensitivity of the high resolution TLC plates. If more than one plate is needed (one 20 by 20 cm plate can accept approximately 18 spots 2 to 3 mm wide) respot the questioned ink(s) and paper control(s) on each additional plate.

7.2.7 *Second TLC Interpretation*:

7.2.7.1 Physically compare the chromatograms of the questioned and selected standard ink(s). Note and record the consistencies in band colors, Rf values, and any fluorescence characteristics. Also note and record any inconsistencies.

7.2.7.2 These comparative examinations between the questioned and standard inks provide the necessary information to eliminate non-matching inks and to locate one or more matching reference ink samples in the ink library (if any matches are present).

8. Additional Analyses

8.1 To date, most forensic analyses of writing inks involve thin layer chromatography. TLC provides a reproducible method that allows for storage of standards and for subsequent comparisons with unknowns. Sometimes, optical techniques along with TLC are insufficient to narrow the field of possible matches to a single reference sample in the ink library. The previously described analysis methods are not by any means the only techniques that can be used, nor are they represented to be the best of all possible methods. Each examination should be considered as an individual matter involving decisions regarding the best method(s) of analysis. The analyst must use the best analytical techniques available, be aware of advantages and shortcomings and determine as many identification criteria as necessary. If more information is needed regarding a particular ink, the additional techniques listed in Guide E 1422 can be tried.

9. Reporting Conclusions

9.1 In reporting conclusions of comparative examinations with an ink library, three necessary elements should be included: (*1*) a listing of the examinations performed; (*2*) the matches found; and (*3*) the conclusions drawn.

9.2 *Examinations Performed*—The report should include a listing of the laboratory examinations conducted. This section should discuss, but does not need to be limited to, the techniques found in Sections 7 and 8.

9.2.1 *Examples*—"Optical (physical) and chemical examinations were performed on the questioned ink from exhibit (give exhibit designation) and the results were compared with those from inks in our ink library. The examinations conducted include (list examinations performed)."

NOTE 9—If the exhibit bears several questioned inks, the report should state their location on the document and that the results of their individual examination were compared with each other. The report should identify questioned inks that are different from each other by sorting the questioned inks into distinct groups consisting of inks that match each other.

9.3 *The Matching Standard Ink(s)*—The cumulative set of comparative examinations (see Sections 7 and 8) will determine the number of reference ink samples (if any) that match a questioned ink. Depending on the level of analysis, a questioned ink can be said to match one or more reference samples in the ink library.

9.3.1 *Differentiation*:

9.3.1.1 If significant, reproducible, inexplicable differences between the questioned ink sample and a reference sample are found at any level of the physical, or chemical analyses, or both, it may be concluded that the inks do not have a common origin.

9.3.1.2 However, when inks give differing test results, the possibility of batch-to-batch variation within an ink formula must be considered; this kind of slight variation may be detectable utilizing sophisticated instrumentation, generally limited to FTIR, GC/MS, HPLC and/or XRF. The potential influences of interfering factors that can alter the composition of an ink sample must also be considered (see Section 5).

9.3.2 *Matches*—When the comparison of the questioned ink sample and a reference sample by optical and chemical analyses reveal no significant, reproducible, inexplicable differences and there is significant agreement in all observable aspects of the results, it may be concluded that the ink samples match at that level of analysis and that the results of the examination indicate that the ink samples are of the same formula or of two similar formulas with the same components.[6]The possibility that other analytical techniques might be able to differentiate the samples should be considered.

NOTE 10—Each comparative examination has its own criteria for determining if a match exists. These are determined by the examiner, based on the examiner's training and experience. Matching criteria should not include inexplicable differences that are too vague (since this may unnecessarily increase the number of matching possibilities) or too specific (since this may eliminate an actual match).

NOTE 11—When a comparative examination yields no inexplicable

differences, the items compared may be said to match or to be indistinguishable at that level of analysis. These terms are not synonymous with the term similar, a term sometimes used for near matches where the results are close but do not meet all the necessary criteria.

9.3.3 An important concern when reaching a conclusion regarding ink matches is whether the matching inks are the same to the exclusion of all other inks. The possibility that the questioned ink matches an ink formula not in the ink library must be assessed based on the experience of the examiner, who evaluates the characteristics of the questioned ink, the examinations performed, the comprehensiveness of the ink library, and information from the ink manufacturer. Based on the above cited factors, this possibility can range from highly probable to extremely unlikely.

9.4 *Single Library Match*—The questioned ink matches only one reference ink sample in ink library to the exclusion of all other reference ink samples.

9.4.1 The matching reference ink sample must be the only one in the library that matches (see 9.3.2) when compared by each examination with the questioned ink sample.

9.4.2 Furthermore, it must be possible to differentiate (see 9.3.1) the questioned ink sample from each of the other (nonmatching) reference ink samples in the library by at least one comparison, thereby eliminating all other reference samples in the ink library as a possible match for the questioned ink.

9.4.3 In the absence of a unique component in the ink formula or some other reason to discount the possibility that the questioned ink may also match one or more additional inks not in the ink library, conclusions should not be reported in absolute terms as an identification, even though based on the comprehensiveness of the standard ink library, the level of examinations performed, and the characteristics determined, this possibility can be remote.

9.4.3.1 *Examples*—"These findings suggest that the questioned ink matches only one standard reference ink from the ink library." Alternatively, "these findings suggest that the matching standard ink is the only standard ink that could not be eliminated as being, the questioned ink." An equivalent statement can be substituted.

9.4.4 If it is determined that the questioned ink sample matches a reference sample that is unique, the report of the findings and of the conclusions should reflect this.

9.4.4.1 *Examples*—"The questioned ink was found to uniquely match a reference sample ink." The conclusion should also state that "The questioned ink is (identified as) the matching standard ink."

9.4.5 Depending on the information requested by the submitter, the report may include the ink manufacturer's name; the manufacturer's designation for the formula; the first production date and last production date; the area(s) of distribution; the brand and type of pens using the formula. If a first commercial production date of the questioned ink was requested, report that the questioned ink matches a reference sample in the ink library that was first manufactured on (state first production date of the matching reference sample ink). Identification of specific dyes, components, and ratios should be avoided as this information may be considered proprietary to the manufacturer.

9.5 *Multiple Library Match*—The questioned ink matches a group of two or more reference ink samples in the ink library to the exclusion of all other reference ink samples outside the group.

9.5.1 The matching reference ink samples must be the only ones in the library that match (see 9.3.2) when compared by each examination with the questioned ink sample.

9.5.2 Furthermore, it must be possible to differentiate (see 9.3.1) the questioned ink sample from each of the other (nonmatching) reference ink samples in the library by at least one comparison, thereby eliminating all other reference samples as a possible match for the questioned ink.

9.5.3 Conclusions should be reported in a manner similar to a single library match (see 9.5.3), while reflecting the multiple matches found.

9.5.3.1 *Example*—"These findings suggest that the questioned ink is one of these matching standard inks or another ink with the same determined characteristics."

9.5.4 Reporting these findings may also include informational items regarding the inks (see 9.5.3). If a first commercial production date of the questioned ink was requested, then it is necessary to report the earliest first production date found within the group of matching reference samples. As noted above, no information should be reported that may be deemed proprietary to the manufacturer.

9.6 *No Match*—The questioned ink does not match any reference samples of ink in the ink library.

9.6.1 Inability to find a matching reference sample in the ink library could be due to one or more of several causes: The ink formula of the questioned ink sample exists outside of the library; but a reference sample of that ink formula is not in the ink library. A reference sample of the ink formula is in the ink library but does not match the questioned ink sample because of significant batch to batch variations in the manufacturing process. The questioned ink sample has changed to the point that it no longer will match a reference sample of the same ink formula in the library.

9.6.2 The report can list some of the possible reasons for these results.

9.6.2.1 *Examples*—"The questioned ink was not found to match any reference sample ink in the ink library. The questioned ink's appearance and characteristics may have changed (have been altered) due to storage conditions, contamination, etc. Another possibility is that the questioned ink may be one that is not in the ink library."

10. Keywords

10.1 forensic sciences; ink identification; questioned documents