Designation: E 1789 – 96

Standard Guide for Writing Ink Identification[1]

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:*

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[2]

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

E 1422 Guide for Test Methods for Forensic Writing Ink Comparison[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*—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,

[1] This guide is under the jurisdiction of ASTM Committee E-30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.02 on Questioned Documents.

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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 Standards, Office of Standard Reference Materials, R. B311, Chemistry Building, Gaithersburg, MD 20899.

comprehensive, and up-to-date as possible, although this will vary between ink libraries.

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.[6]

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.[7] 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.[8]

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.[6]

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.

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

[7] 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.

[8] 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.

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