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## Analysis of Ballpoint Pen Inks by Diffuse Reflectance Infrared Spectrometry

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**ABSTRACT:** The primary method for the analysis of inks is thin-layer chromatography (TLC), which only permits comparison of dye components. Studies have been done on the usefulness of diffuse reflectance (DR) with Fourier transform infrared (FT-IR) spectrometry, using a direct deposit sampling technique for the analysis of ballpoint pen inks. This technique allows screening of a composite ink as a whole, including its dye components, resins, and other additives. A total of 184 ink samples were analyzed by FT-IR, and searchable spectral libraries were created of both whole and extracted inks. The results indicate that the additional information provided by the DR analysis, combined with the complementary information from the TLC analysis, provides enhanced value to the forensic examination of inks.

**KEYWORDS:** questioned documents, inks, spectroscopic analysis, ballpoint pen ink, diffuse reflectance, infrared spectrometry, dyes, resins, spectral library

Ink analysis is often requested in forensic science cases involving questioned documents. Comparison of a questioned ink from a document with a known ink obtained from a suspect could contribute valuable information to a case. In some cases, the ability to determine the manufacturer of a questioned ink could be used as an investigative lead, especially if the ink is of a less common type.

The composition of ballpoint pen ink varies among manufacturers but generally consists of a glycol-based solvent; a selection of dyes; resins, which are sometimes added to adjust the viscosity of the ink; ball lubricants; and other proprietary additives used to improve the writing quality [1]. The standard procedure for the forensic science analysis of inks consists initially of nondestructive visual examinations, such as inspection of the questioned document with visible light, using narrow-band filters and observation under ultraviolet and infrared radiation sources. Following the visual examinations, the extracted questioned and known ink samples are compared by thin-layer chromatography (TLC). The resulting TLC plate is examined using both visible and ultraviolet radiation to determine the presence of fluorescence. The  $R_f$  values are measured, the color is observed, and the relative concentrations of the components are determined [2–5]. This

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technique works well but only permits comparison of dye components. Resins cannot be detected by this procedure.

A large, comprehensive library of inks is maintained and updated in Washington, DC, by the U.S. Secret Service. Currently, a searchable text database of the inks contained in this library is being prepared based on TLC separations of the dye components. The search and identification process will involve searching a sequence of colors assigned values from 0 to 9, located within five preassigned zones, each representing 1 cm of distance traveled. Searching color sequences within zones should reduce errors, since  $R_f$  values can vary depending on plate variations and conditions at the time of separation.<sup>2</sup>

Detection of resins can play a very important role in comparison of inks, as illustrated in the case of *U.S. v. Bruno* [6] from Pennsylvania in 1971. This case, involving tax evasion and organized crime, included the identification of a Scripto ink in which identification of the resin played a leading role. An elaborate technique was used to derivatize and analyze the resin by gas chromatography (GC), but expert testimony by Richard Brunelle of the Bureau of Alcohol, Tobacco, and Firearms (ATF) "was not accepted because of the incompleteness of the ATF Ink Library at that time."<sup>3</sup>

Fourier transform infrared (FT-IR) spectrometry has been used in the past to study characteristics of inks [7–9]. In a preliminary study of FT-IR methods of ink analysis on documents, Harris determined that use of diamond cell transmission and micro-reflectance spectroscopy was unsuccessful for the analysis of ink-saturated paper fibers [10]. He concluded that diffuse reflectance (DR) FT-IR may be a viable method for nondestructive analysis of ink on paper. However, the spectra that he obtained of ink on paper do not compare with his known reference spectra of the same pure inks.

Our attempts to analyze ballpoint pen ink on paper nondestructively by both reflectance and diffuse reflectance using an FT-IR microscope were unsuccessful. Strong absorptions from the paper tend to mask any absorptions contributed by the ink when analyzed by these methods. Efforts to analyze extracted ink samples by transmission on potassium bromide (KBr) windows using an FT-IR microscope proved somewhat successful, indicating the need for further study on this technique. The process of casting a film of the extract resulted in the formation of a ring of dye components. Spectra were obtained by focusing the IR beam on the colored dye ring. In our studies, it was noted that dye components in some ink samples tend to separate on the window. On a Papermate ink, where separation occurred on the KBr window, the outer portion of the ring was sampled. The resulting spectrum was different from a reference spectrum of the pure ink, indicating that analysis of a small segment of the dye ring may not always be representative of the ink sample as a whole.

Fuller and Griffiths discussed chromatographic separation of dye by TLC followed by analysis using DR with FT-IR [11]. Further work was done by Suzuki and Gresham using DR in the development of a microsampling method for the analysis of solids in solution. They found that good spectra could be obtained from samples prepared by direct deposit of a solution onto KBr, prepacked into a microsample cup, followed by evaporation of the solution [12].<sup>4</sup>

Our studies suggest that DR, utilizing the direct deposit method, could be a useful sampling method for analysis of ballpoint pen inks. Analysis by DR allows screening of a composite ink as a whole, including dye components, resins, and other additives. Preliminary examinations indicate that sufficient differences exist between ink formulations to identify the manufacturer based on spectral characteristics. Studies were done on both whole liquid ink removed from ballpoint pen cartridges (whole ink) and on

<sup>2</sup>Stewart, L. F., Department of the Treasury, U.S. Secret Service, personal communication, 1990.

<sup>3</sup>Brunelle, R. L., Brunelle Forensic Laboratories, personal communication, 1990.

<sup>4</sup>This reference provides an excellent overview of diffuse reflectance and discusses a variety of forensic science applications for the technique.

extracted ballpoint pen ink from writing samples (extracted ink). Current FT-IR software provides the ability to create customized computer searchable spectral libraries easily. Such libraries have been prepared of both whole and extracted ballpoint pen inks analyzed by DR. Use of the complementary information provided by both TLC and FT-IR could provide more complete information for the forensic science examination of an unknown ink.

## Materials and Methods

### Equipment

All spectra were obtained with a Nicolet 20SXC spectrometer (Madison, Wisconsin) equipped with a medium-band mercury cadmium telluride (MCT) detector operating at  $4\text{ cm}^{-1}$  resolution and utilizing a Spectra-Tech Collector™ (Stamford, Connecticut) DR accessory. Samples were prepared in microsample cups 3 mm in diameter and 2 mm deep. All samples were analyzed by collecting 256 scans with a full aperture from 4000 to  $650\text{ cm}^{-1}$ . Residual carbon dioxide ( $\text{CO}_2$ ) and water ( $\text{H}_2\text{O}$ ) were removed from the spectra by subtraction when necessary, and baselines were normalized using the Nicolet correction routine.

A micro-boring tool, illustrated in Fig. 1, was made by removing the sharp tip from a 20-gage hypodermic needle and sharpening the end with a jeweler's file. A plunger removed from a 10- $\mu\text{L}$  Hamilton syringe was added and positioned with a spring. This boring tool, which is the approximate width of a written line, is ideal for removing ink samples from a document. Sampling by this technique is considered nondestructive to document examinations since the writing remains legible.

### Extraction Technique

Ink extracts were prepared by removing 20 paper punches containing the ink in question from the document using the micro-boring tool. Pyridine was chosen as the extracting solvent since it is one of the more efficient solvents for extracting ballpoint pen ink [13,14]. The punches were extracted for 30 min using gentle, periodic agitation with 10  $\mu\text{L}$  of pyridine in a limited-volume insert held inside a glass vial. A 10- $\mu\text{L}$  Hamilton

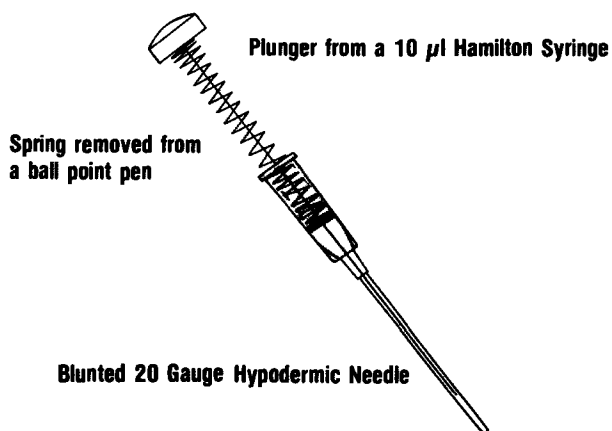


FIG. 1—Illustration of the micro-boring tool used to remove plugs of paper for sampling written inks and paper blanks.

syringe was then used to drop 3  $\mu\text{L}$  of ink extract onto a KBr-filled DR microsample cup. The pyridine was allowed to air-evaporate prior to analysis. A paper blank was obtained by removing 20 punches of blank paper near the ink sampling location. The blank was extracted and analyzed in the same manner as the ink sample. The resulting spectrum was then used to subtract spectral interference contributed by the paper from the ink spectrum.

### Results and Discussion

Analysis of replicate ink extracts demonstrates spectral reproducibility with only minor variations. Figure 2 shows spectra obtained from the analysis of six replicate extractions of the same Bic black ink. Minor differences among these spectra near  $1700\text{ cm}^{-1}$  are contributed by the paper and may be removed by careful paper blank subtraction. Spectra of paper blanks differ dramatically from page to page and, to a limited extent, even differ within the same page. Figure 3 shows spectra of three different paper blanks. These spectra have the same absorption bands but differ in peak height ratios. The relative contribution of the paper blank in a spectrum of extracted ink may be as high as 30% in some instances.

### Sampling

When sampling an ink from a document, special care is necessary to avoid ink from the back of the page and also to avoid ruled lines on the paper since these other inks could be extracted during the procedure. Vortexing should be avoided during extraction since vigorous agitation may cause the paper to break down, promoting additional interference from cellulose. A paper blank should be taken very near the ink sampling site because of the variability within the paper. If this is not possible, then the blank should

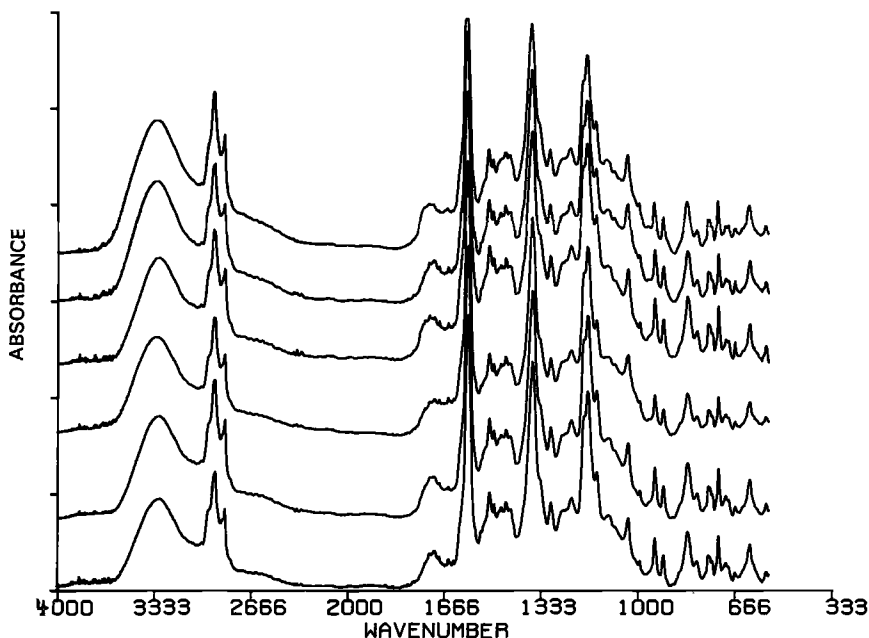


FIG. 2—Infrared spectra from six replicate pyridine extractions of the same Bic black ink.

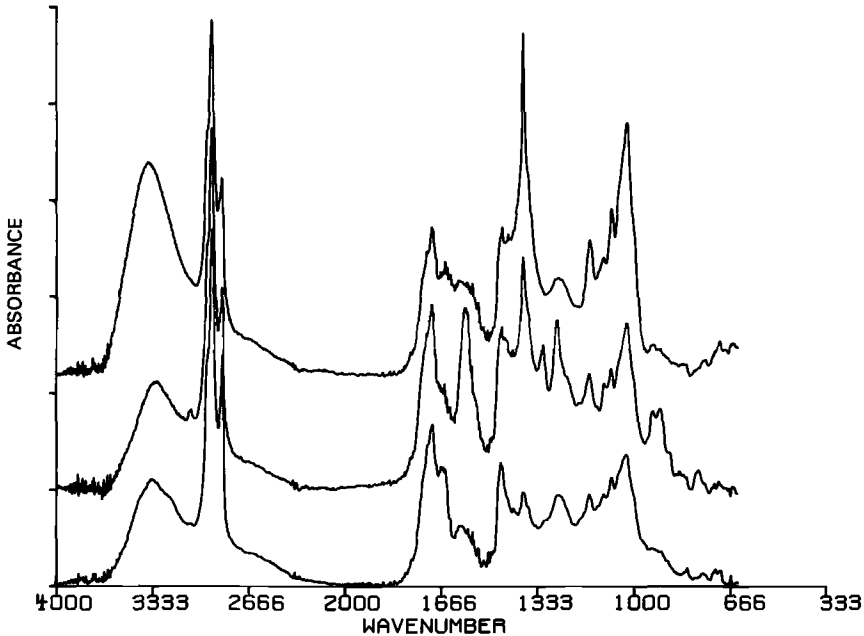


FIG. 3—Infrared spectra of three different paper blanks extracted with pyridine.

at least be taken from the same page as the ink sample and should be extracted in the same manner as the ink sample.

#### *Libraries*

A collection of 184 ballpoint pens was obtained. Whole ink was removed from each of the pens by inserting a glass capillary into the cartridge and then rinsing the capillary with pyridine. Enough pyridine was added to dilute the ink sample to approximately 1.5 mL. Samples were prepared by dropping 3  $\mu$ L of each diluted ink sample onto a KBr-filled DR microsample cup. These samples were then analyzed by DR using a full aperture and 256 scans. Inks analyzed by this technique show considerable differences among manufacturers, as illustrated in Fig. 4, which shows four spectra of inks from higher-quality pens, including Waterman, Parker, Mont Blanc, and Cross.

Reproducibility between different production runs of the same ink formulation is very good. Figure 5 shows four spectra of Bic black ink, all of the same formulation but removed from four different pens. Reproducibility can again be seen in Fig. 6, which shows four spectra of the same formulation of Papermate black ink removed from four different pens.

The 184 spectra of whole ink represent 28 manufacturers. Many of these samples were taken from promotional-type pens used for advertising purposes. Of the 184 samples analyzed, 100 were black ink, 81 were blue ink, one was green ink, and two were ink resins. A spectral library was created using entries consisting of only blue and black inks and limited to one of each of the identified ink formulations. Inks from promotional pens were not included. This library, consisting of 89 samples from the same 28 manufacturers, includes 49 black inks and 40 blue inks. It will be referred to as the Whole Ink Library. The use of whole inks for library preparation avoids added interference from paper blanks and negates the error introduced in blank subtraction.

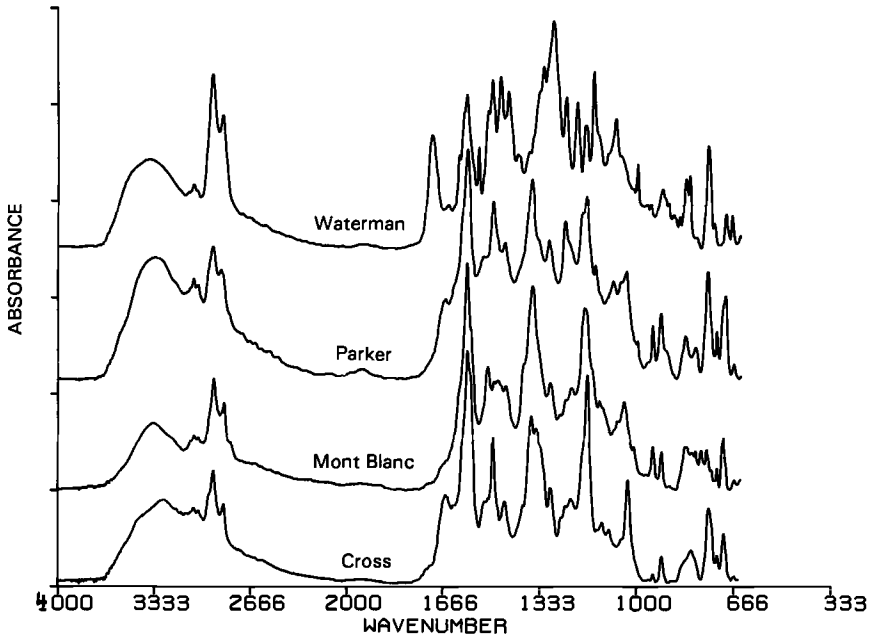


FIG. 4—Infrared spectra of four different black inks showing variations among manufacturers.

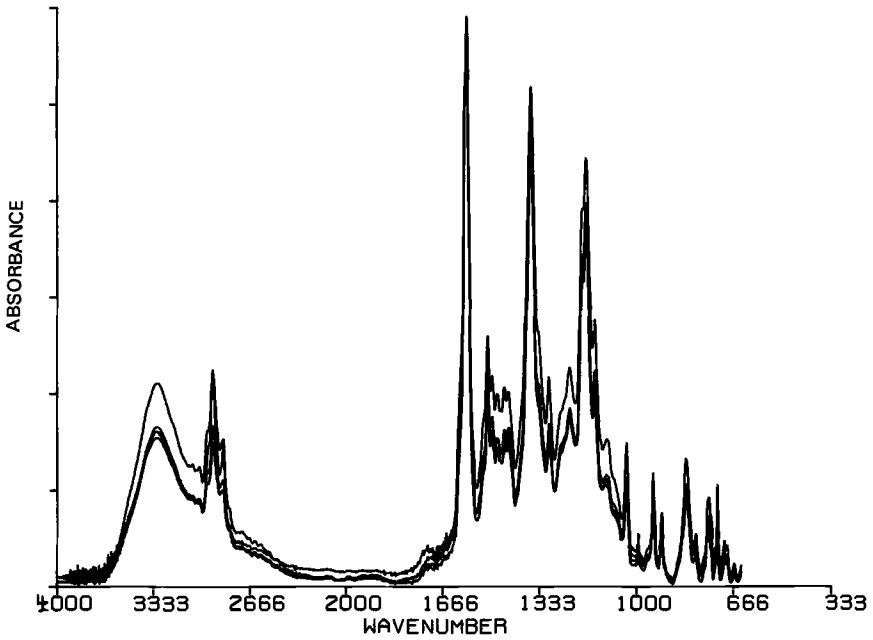


FIG. 5—Infrared spectra of Bic black ink removed from four different pens showing reproducibility between different production runs.

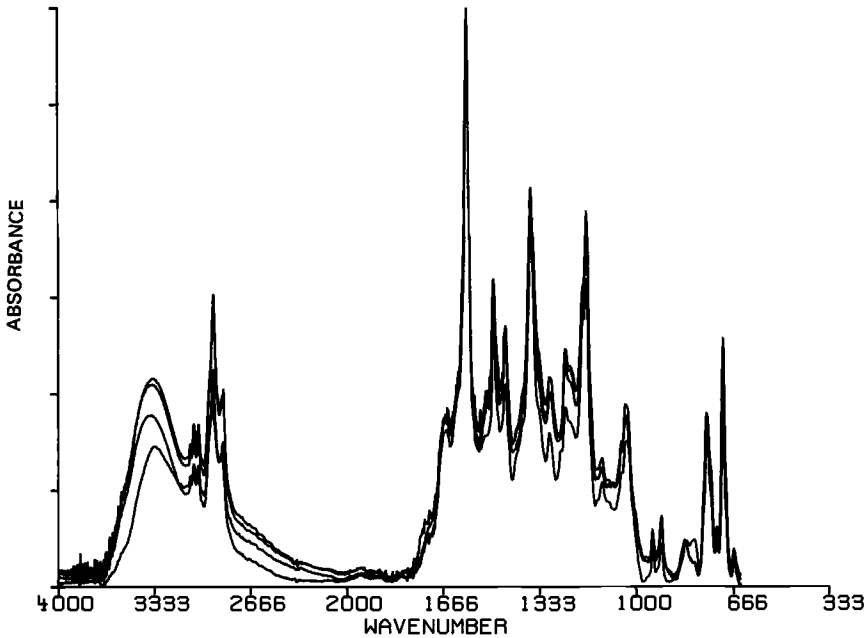


FIG. 6—Infrared spectra of Papermate black ink removed from four different pens showing reproducibility between different production runs.

Table 1 shows the reduction of entries between the original 184 ink samples and the creation of the Whole Ink Spectral Library to include only one of each formulation. It is apparent that continued sampling will be necessary to include additional ink formulations not yet analyzed. Of the 18 black Bic inks that were analyzed, three formulations were identified and all are clearly different spectrally, as is shown in Fig. 7. The top spectrum (A) is Bic's European formulation, and the next two spectra (B and C) are from formulations made in the United States.

One major advantage of DR is its ability to detect resins not visible by TLC. We currently have spectra of two resins commonly used in inks. Hi-Luk 222, a ketone resin from Japan, is sometimes seen in Japanese-made inks.<sup>5</sup> The strong carbonyl absorption at  $1705\text{ cm}^{-1}$ , indicative of a ketone, is very prominent in the black ink from an Itoya pen made in Japan, thus indicating the presence of the Hi-Luk 222 resin (see Fig. 8).

A common resin used in the United States is RJ-101, a styrene-allyl alcohol copolymer,<sup>5</sup> shown in Fig. 9. The indicative absorptions in this resin include the aromatic C-H stretching region between  $3090$  and  $3025\text{ cm}^{-1}$ , the aromatic overtones at  $1944$ ,  $1874$ ,  $1805$ , and  $1722\text{ cm}^{-1}$ , the skeletal breathing at  $1499$  and  $1452\text{ cm}^{-1}$ , and the aromatic substitution bands at  $750$  and  $700\text{ cm}^{-1}$ . These absorption bands can easily be seen in Scripto Tokai standard blue ink No. 903. According to the manufacturer, this ink contains relatively high concentrations of the RJ-101 resin.<sup>5</sup>

Low-temperature heating of the prepared DR sample to speed evaporation of the pyridine extracting solvent was found to cause changes in the spectra of some inks, as illustrated in Figs. 10 through 12. Figure 10 shows two spectra of Scripto Tokai standard blue ink No. 903 both before and after heating. The upper spectrum was obtained from analysis following air evaporation of the pyridine. The lower spectrum, with less intense bands, shows the same sample after exposure under a heat lamp for 1 h at approximately

<sup>5</sup>Massey, R. R., Anja Engineering, producers of Scripto Tokai, personal communication, 1990.

TABLE 1—*Listing of the original inks sampled showing reduction of samples for the creation of the Whole Ink Library.<sup>a</sup>*

Brand	Black Ink		Blue Ink	
	Library	Original	Library	Original
Berol	1	1	1	1
Bic	3	18	1	12
Cross	1	3	1	3
Eberhard Faber	1	1	1	1
Faber Castell	0	0	1	2
Fisher	4	5	4	6
Hallmark	1	1	1	1
Itoya	1	2	1	1
LevLite	1	1	0	0
Lindy	1	1	1	1
Major	1	1	1	1
Marvy Uchidi	0	0	1	2
Mont Blanc	2	3	2	2
Niji	1	1	0	0
Papermate	4	13	7	9
Parker	1	4	2	3
Pentech	2	2	2	2
Pentel	1	1	0	0
Pilot	3	6	2	5
Remmie Arnold	1	1	0	0
Schwan	1	1	1	1
Senator	1	1	0	0
Scripto Tokai	2	2	2	2
Sheaffer	8	12	2	2
Skilcraft	3	6	2	3
Staedtler	1	1	1	1
Waterman	1	3	2	4
Zebra	2	3	1	1

<sup>a</sup>NOTE: Not included in the library are 5 black advertising inks, 15 blue advertising inks, 1 black Erasermate, 1 green Skilcraft, and 2 ink resins.

75°C. Low-temperature heating was found to cause changes in this ink spectrum consistent with evaporation of the dye components. These changes may also reflect fading of dyes caused by the intense light of the heat lamp. Based on the remaining spectral absorptions after heating, the RJ-101 resin seems to be unaffected by heat. Some inks showed dramatic changes in peak ratios with heat exposure, such as the Scripto Tokai blue No. 903, shown in Fig. 10. Others showed less dramatic evaporative effects, such as Scripto Tokai black No. 202, shown in Fig. 11. This ink also contains the RJ-101 resin,<sup>5</sup> and the prominent peaks are clearly visible. Still other inks showed no effect from heating, such as the Pilot black ink, shown in Fig. 12.

Since heating resulted in a variety of spectral changes among the ink samples, a second library was created: the Heated Ink Library. This library was prepared with spectra from samples contained in the Whole Ink Library after 1 h of exposure under a heat lamp. Use of this library for searching extracted samples would be expected to provide closer hits, since heating is a means of artificially aging writing samples [13].

Seventy-one inks were extracted from paper as previously described and analyzed to test the search capabilities of the two libraries. The libraries were searched using the absolute derivative algorithm. Of the 71 samples searched, 38% came up as the first hit from the Whole Ink Library, and 42% came up as the first hit from the Heated Ink Library. Of the 71 samples tested, 74% were within the first five hits when searched against the Heated Ink Library (see Table 2).



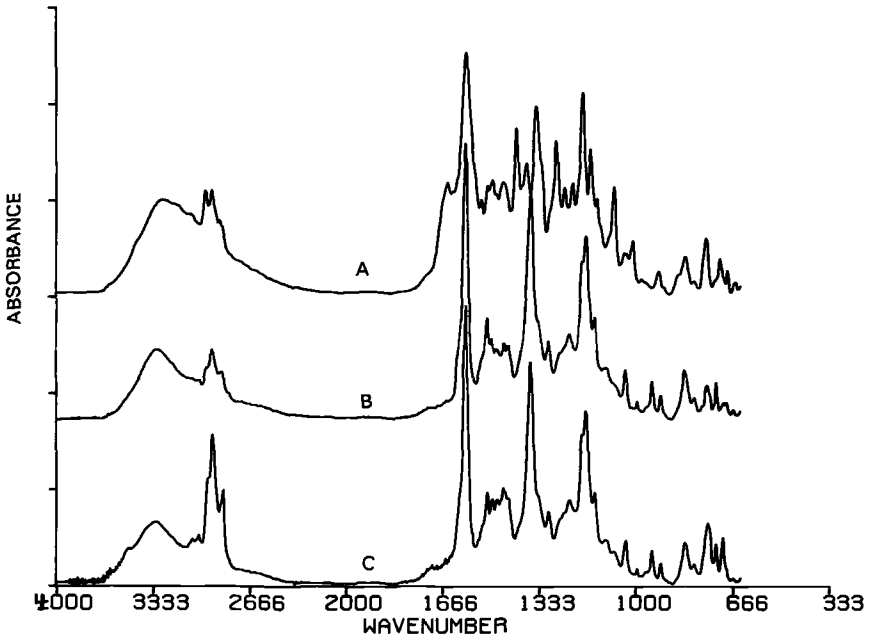


FIG. 7—Infrared spectra of three different formulations of Bic black ink: (a) Bic's European formulation, (b) and (c) both formulations made in the United States.

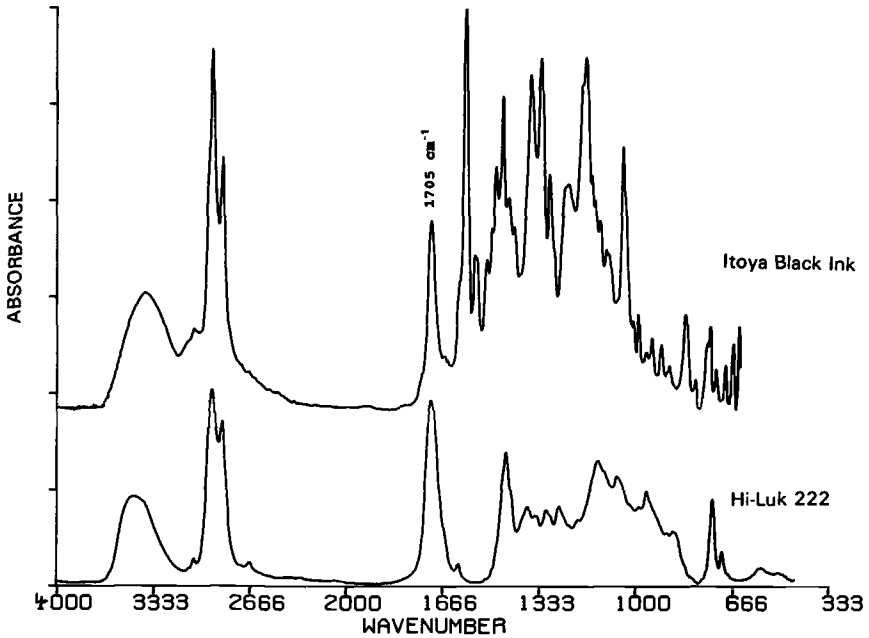


FIG. 8—Infrared spectrum of Hi-Luk 222, a ketone resin from Japan commonly seen in Japanese-made inks, shown with a spectrum of an Itoya black ink made in Japan.

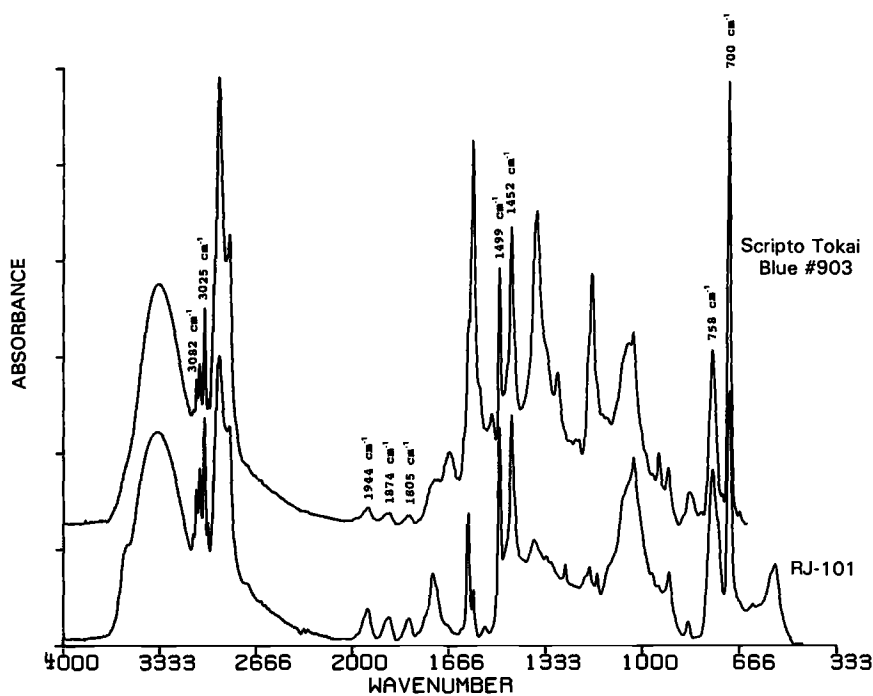


FIG. 9—Infrared spectrum of RJ-101 resin, a styrene allyl alcohol copolymer, commonly added to American-made inks, shown with a spectrum of a Scripto Tokai blue ink No. 903.

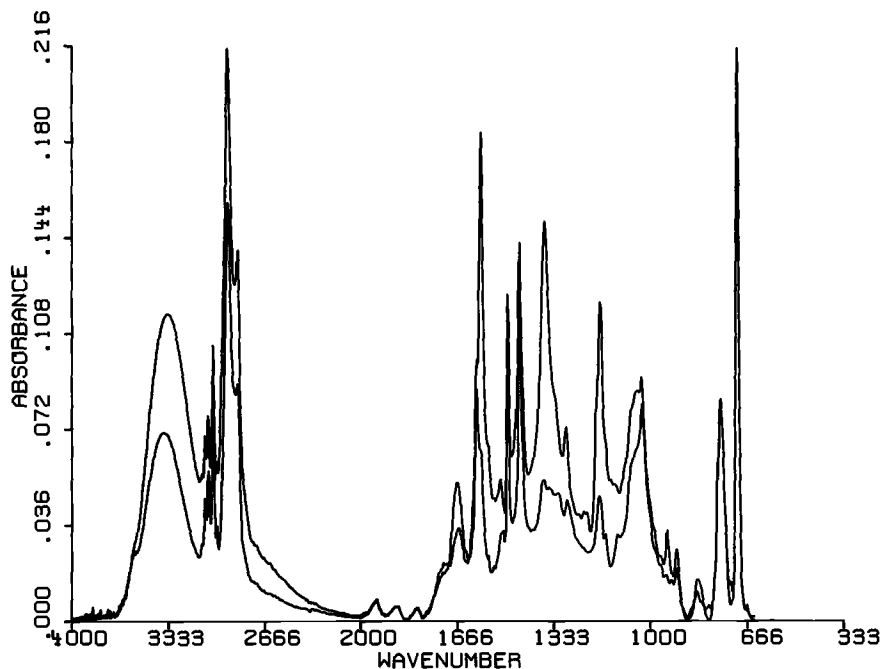


FIG. 10—Two infrared spectra of Scripto Tokai standard blue ink No. 903. The upper spectrum, showing more intense absorptions, is the result of analysis following air evaporation of the pyridine diluent. The lower spectrum, with less intense absorptions, is the result of heating under a heat lamp for 1 h.

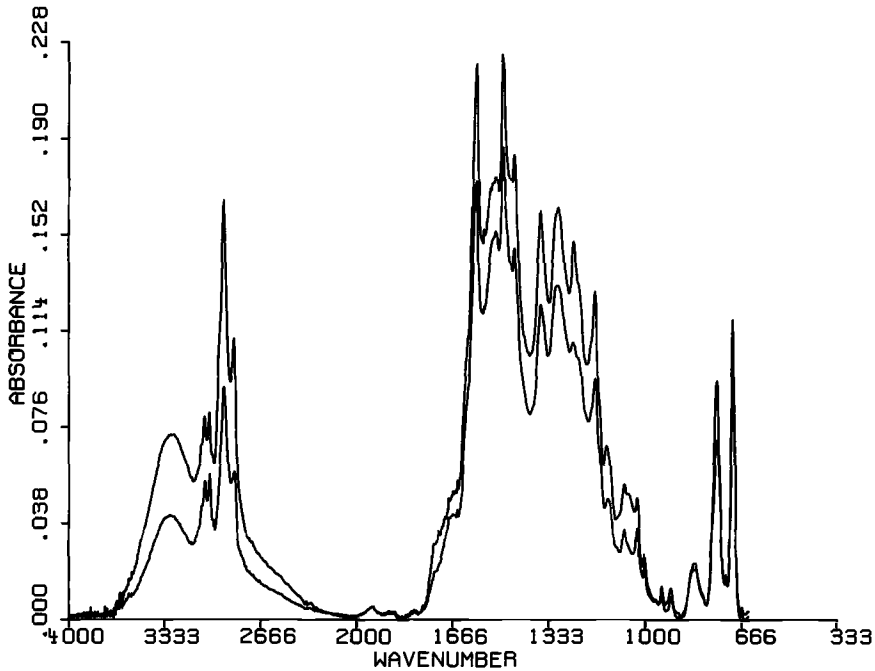


FIG. 11—Two infrared spectra of Scripto Tokai black ink No. 202. The upper spectrum, showing more intense absorptions, is the result of analysis following air evaporation of the pyridine diluent. The lower spectrum, with less intense absorptions, is the result of heating under a heat lamp for 1 h.

Comparison of library spectra must be done very carefully to avoid error. Figure 13 shows a paper-blank-subtracted spectrum of Pilot black ink extracted from paper and analyzed by diffuse reflectance. The spectrum was searched against the Heated Ink Library. The first hit was a black Papermate ink with a very similar spectrum. This hit was probably first because of the closeness of fit with the aliphatic C-H stretching region and the similarities in size of the peaks between 1700 and 1000  $\text{cm}^{-1}$ . The correct hit came up fifth. The differences seen between the extracted sample and the correct hit in the aliphatic C-H stretching region could be a result of paper interference and incorrect subtraction of the paper blank. To avoid searching this region containing paper-blank errors, a second search was done on the spectrum of extracted Pilot black ink after blanking the region from 4000 to 2000  $\text{cm}^{-1}$ . Results of the search on the remaining spectrum from 2000 to 650  $\text{cm}^{-1}$  once again showed a black Papermate ink as the first hit. The correct hit came up third this time. Upon comparison, it should be noted that even though the main envelope between 1700 and 1000  $\text{cm}^{-1}$  was closer to the first hit, the peak height ratios are more consistent with the third hit, especially in the areas of about 1500 and 700  $\text{cm}^{-1}$ . The single absorption band at about 1200  $\text{cm}^{-1}$  seen on the extracted ink spectrum compares well with that region from the third hit but differs from the same region of the first hit, which shows two absorption bands in its spectrum.

It is known that once an ink is applied to paper it begins to undergo numerous changes, including oxidation, cross-linking, polymerization, and solvent evaporation [14]. Because these changes occur, an additional spectral library was prepared—the Extracted Ink Library. This library consists of the same ink formulations contained in both the Whole and Heated Ink Libraries, extracted from paper as previously described. The spectra resulting from analysis of the extracted inks were baseline corrected, and the spectra of the paper blanks were subtracted prior to creation of the library. Spectra contained in

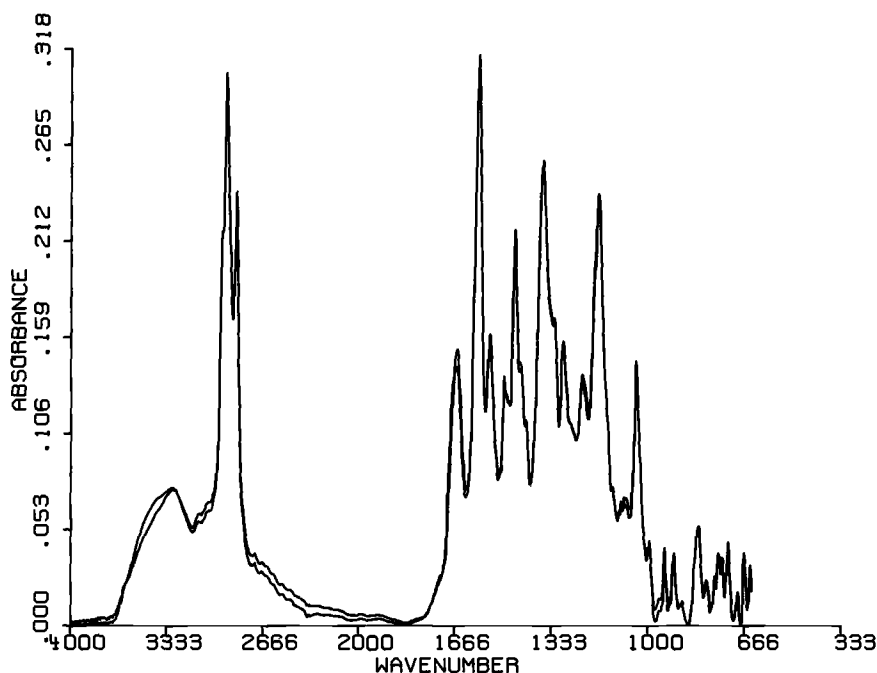


FIG. 12—Two infrared spectra of Pilot black ink. One spectrum is the result of analysis following air evaporation of the pyridine diluent. The other spectrum is the result of heating under a heat lamp for 1 h.

this library are better representations of extracted inks and thus provide a better comparison with an unknown ink extracted from a questioned document.

Twenty-nine inks were extracted from paper and analyzed to test the search capabilities of the three ink libraries. The spectra were searched both before and after the paper blank spectrum was subtracted in order to determine whether or not paper-blank subtraction is a necessary step prior to searching. The spectra were initially searched from 4000 to 650  $\text{cm}^{-1}$  against each of the three libraries. The spectra were then blanked from 4000 to 2000  $\text{cm}^{-1}$  using the Nicolet blanking routine, and the remaining region from 2000 to 650  $\text{cm}^{-1}$  in each spectra was searched against each of the three libraries. By limiting the region searched, variation in C-H stretching and noise interference should be removed, thus providing closer matches during a library search. Results of this study are shown in Tables 3 and 4.

It was determined through this study that for the analysis of extracted inks, the best

TABLE 2—Results of library searches of 71 spectra of extracted inks both with and without paper blank subtractions.

Ink Library	Paper Blank Correction	Library Search Hit Number			
		1	2-5	6-10	11 and up
Whole	unsubtracted	38%	30%	15%	15%
	subtracted	38%	32%	8%	21%
Heated	unsubtracted	42%	32%	11%	14%
	subtracted	39%	39%	6%	15%

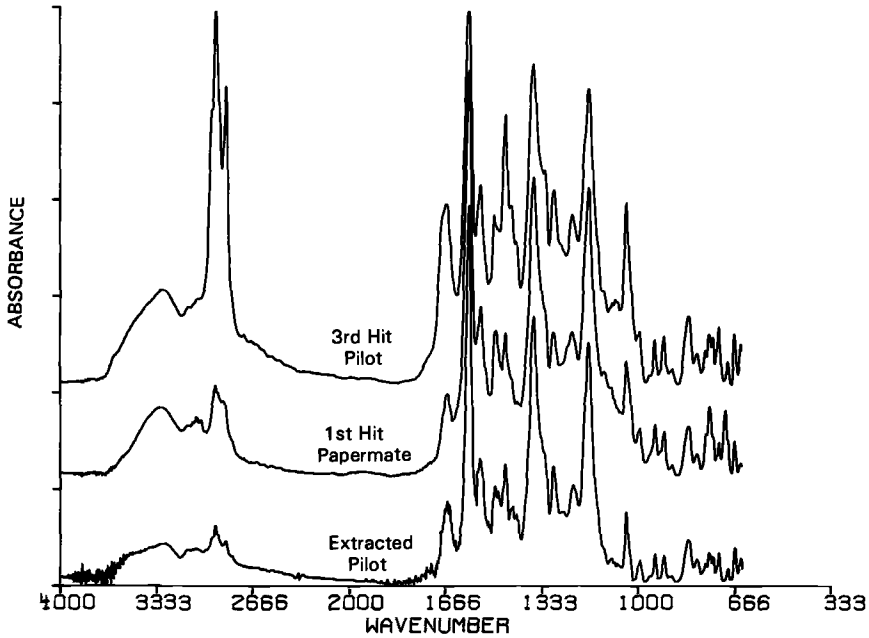


FIG. 13—Comparison of the infrared spectrum of extracted Pilot ink with the first and third hits from the library search of the Heated Ink Library.

TABLE 3—Results of library searches of 29 spectra of extracted inks searched from 4000 to 650  $cm^{-1}$  both with and without paper blank subtractions.

Ink Library	Paper Blank Correction	Library Search Hit Number			
		1	2-5	6-10	11 and up
Whole	unsubtracted	52%	24%	10%	14%
	subtracted	48%	34%	7%	10%
Heated	unsubtracted	41%	31%	14%	14%
	subtracted	45%	38%	3%	14%
Extracted	unsubtracted	66%	28%	3%	3%
	subtracted	66%	31%	3%	0%

TABLE 4—Results of library searches of 29 spectra of extracted inks searched from 2000 to 650  $cm^{-1}$  both with and without paper blank subtraction.

Ink Library	Paper Blank Correction	Library Search Hit Number			
		1	2-5	6-10	11 and up
Whole	unsubtracted	52%	34%	3%	10%
	subtracted	62%	28%	0%	10%
Heated	unsubtracted	45%	34%	10%	10%
	subtracted	48%	38%	3%	10%
Extracted	unsubtracted	79%	14%	7%	0%
	subtracted	86%	10%	3%	0%

search results are obtained by subtracting the paper blank spectrum from the ink spectrum and then blanking the region from 4000 to 2000  $\text{cm}^{-1}$  before searching against the Extracted Ink Library. It was determined that of the 29 ink samples tested, 86% of them appeared as the first hit, 96% appeared within the first five hits, and 100% appeared within the first ten hits.

### Conclusion

As a result of this work, we have determined that FT-IR is a valuable method for the analysis of inks. Use of the DR accessory and the direct deposit technique makes sampling reasonably convenient. Computerized searching of the composite spectra provides a rapid means of comparing the variety of inks currently on the market. Though the correct inks did not always appear as the first hits, the field of possibilities was narrowed considerably. Careful comparison by the examiner of the closest hits from the spectral search is important for the final evaluation of the results. This is especially important because of the limitations of the spectral libraries based on quantity of reference spectra. The additional information provided by the DR analysis combined with the complementary information from the TLC analysis provides enhanced value to the forensic science examination of inks.

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