Timothy W. Sinor,[1] *B.S.; Jeffrey P. Wilde,*[1] *B.S.; Kathleen E. Everse;*[1] *and E. Roland Menzel,*[2] *Ph.D.*

Lasers and Optical Spectroscopy in Questioned Document Examination

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ABSTRACT: The use of in situ laser-luminescence and excitation spectroscopy for examination of questioned documents is investigated. It is found that such spectroscopy can at times discriminate between similar inks when conventional methods are unsuccessful. A particularly effective procedure for room temperature luminescence enhancement is presented and compared with luminescence at cryogenic temperatures.

KEYWORDS: criminalistics, questioned documents, lasers, absorption, emission, luminescence, excitation, thin-layer chromatography

The use of lasers, such as Ar-lasers, for detection of latent fingerprints was first reported in 1976 (E. R. Menzel, J. M. Duff, and B. E. Dalrymple, paper presented in October of that year in London, Ontario at the annual Michigan-Ontario Identification Association Conference) and has since amply proven its value in casework. A growing number of law enforcement agencies use lasers for fingerprint work. The most useful procedures for laser fingerprint development were recently reviewed [*1*]. Relatively little attention has been paid, however, to laser application to other areas of physical evidence analysis, such as fiber and document examination.

The combination of laser excitation with luminescence spectroscopic measurements has been used in criminalistics research laboratories for some time [*2*]. However, application of laser spectroscopy to evidence examination in order to enhance its probative value has not yet materialized. In this article, we investigate the use of lasers and spectroscopy in questioned document examination. A similar study dealing with fiber analysis will follow in a separate article.

Present Methods

The use of lasers in discrimination of inks that appear identical to visual examination in room light, as pertinent to detection of alterations on checks or documents, for instance, was demonstrated some time ago [*3*] and application to casework followed [*4,5*]. In these exami-

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[1]Research assistants and research associate, respectively, Center for Forensic Studies, Texas Tech University, Lubbock, TX.

[2]Associate professor, Physics Department, and director, Center for Forensic Studies, Texas Tech University, Lubbock, TX.

nations, inks could be distinguished visually via laser-induced fluorescence. In such situations, photographic documentation of the case suffices and there is no need to contemplate spectroscopic measurements. Here, however, we are primarily concerned with instances in which visual inspection is not able to discriminate between similar inks even under laser excitation. Three techniques one might consider in such cases are absorption microspectrometry [*6, 7*], thin-layer chromatography (TLC) [*7, 8*], and infrared (IR) luminescence [*8–12*].

Infrared luminescence, as practiced in forensic science laboratories, most often utilizes green light for illumination. Thus, the situation very much resembles photography of Ar-laser-induced fluorescence. The emphasis in this article is on occasions where such photographs are not able to distinguish between similar inks. Thin-layer chromatography is a rather good technique for ink analysis, provided that one has enough of the ink available to develop a TLC plate. This is not always the case. Also, it has been shown that the combination of laser excitation with TLC (that is, visualization of TLC bands by their fluorescence) can strongly enhance sensitivity [*3, 13*]. Significant variation of absorption microspectra of a given ink sampled from different fibers of the same paper item have been noted, and it was found that absorption microspectroscopy was less discriminating than TLC [*7*]. On the other hand, microspectroscopy does not require a substantial amount of ink, as TLC does. Unfortunately, only very few forensic science laboratories around the world possess absorption microspectrometers.

Laser Spectroscopy

To compare the use of laser-induced luminescence and luminescence-spectroscopic techniques with the above-mentioned three methods for discrimination of inks, we deliberately selected inks from pens that would tax the discriminatory ability of these current methods. Altogether, some 30 black, blue, and red ballpoint and porous tip pens were investigated. Table 1 lists the samples explicitly dealt with in this article.

We start out by examining the features of two blue inks (Samples 9A and 19). Solution absorption spectra (methanol solvent) obtained on a Perkin-Elmer 356 spectrometer are shown in Fig. 1. Such absorption spectra (which are not obtained on a microspectrometer) require sizeable ink samples and are, of course, not taken in situ. On the other hand, spectral dependence on the nature of fibers of the paper on which the ink is deposited does not play a role. An IR luminescence photograph of sample tracings with the same blue inks under 5145A Ar-laser excitation is shown in Fig. 2. Unlike many samples of blue, black, and red pen inks, which could be distinguished quite clearly by IR luminescence photography, the two blue samples could not thus be differentiated, as is seen in Fig. 2. A room-light photograph of a TLC plate on which the two blue inks were developed is presented in Fig. 3. This TLC plate shows that the two inks are indeed quite similar to each other in composition, but they are nonetheless distinguishable. With lesser ink amounts, as might occur when only a small alteration is made on a document, poorly or undeveloped chromatograms are obtained, as is shown in Fig. 4*a*. The plate of Fig. 4*a*, as developed by laser-induced fluores-

TABLE 1—*Pens used in the present document examination study.*

Sample Number	Ink Color	Pen Make
4	red	Pilot® BP-S Medium
9A	blue	Pilot Razor Point
11	black	Paper Mate® Prof. Fine Point
12	black	Bic® N-65
13	black	Bic B-C-3
14	red	Bic E-B-16
19	blue	UNI UBR-7 (Mitsubishi Pencil Co.)

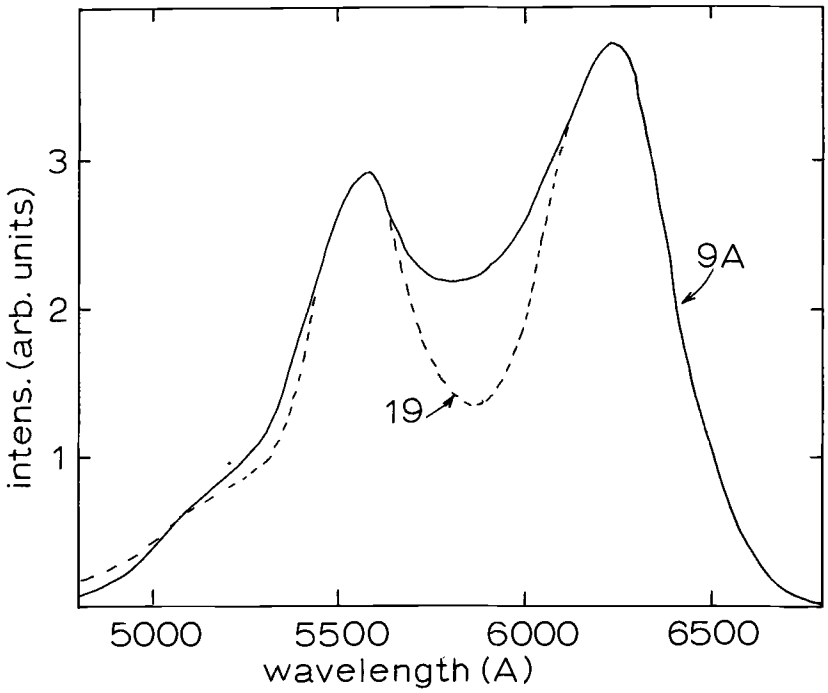


FIG. 1—*Room temperature solution absorption spectra of ink Samples 9A and 19 (see Table 1).*

cence, is shown in Fig. 4*b*. The chromatography used 100% C_{18}-silanized silica gel glass plates (Sigma Chemicals). The solvent was a mixture of equal parts (by volume) of acetone, methanol, and distilled water. Ink samples were applied by spotting. While it is clear from Fig. 4 that laser development of TLC plates can substantially improve sensitivity, the practical problem remains that one does not normally attempt TLC ink discrimination when only very small amounts of an ink are available in a suspected document alteration. In such instances, it becomes of interest to see whether luminescence spectroscopy is of any value in differentiating inks. The idea is to excite ink luminescence emission in situ by focusing a laser beam onto an area about the width of an ink trace (on the order of 1 by ½ mm) and to measure the ink luminescence spectrum using well-known spectroscopic techniques that have been described in detail [*14*]. At times, it may be valuable not only to measure luminescence spectra, but excitation spectra as well. This can be done using a white light source/monochromator combination [*14*] or a dye laser that can be tuned, and will be dealt with shortly in more detail.

Blue Inks—Emission Spectra

The in situ emission spectra of the two blue inks dealt with above are shown in Fig. 5 under Ar-laser excitation at 5145 A with power density of about 100 mW/cm^2. Figure 6 shows the block diagram of the experimental apparatus, which utilized a scanning monochromator equipped with a 1100 lines/mm grating blazed at 5000 A and a photomultiplier tube of trialkali spectral response. The luminescence was prefiltered with a Corning 3-67 filter. This is essentially the standard filter of Ar-laser safety goggles (orange in appearance). The spectra shown in Fig. 5 are not corrected for instrumental response versus wavelength. The described apparatus has a spectral sensitivity that decreases monotonically from the green to

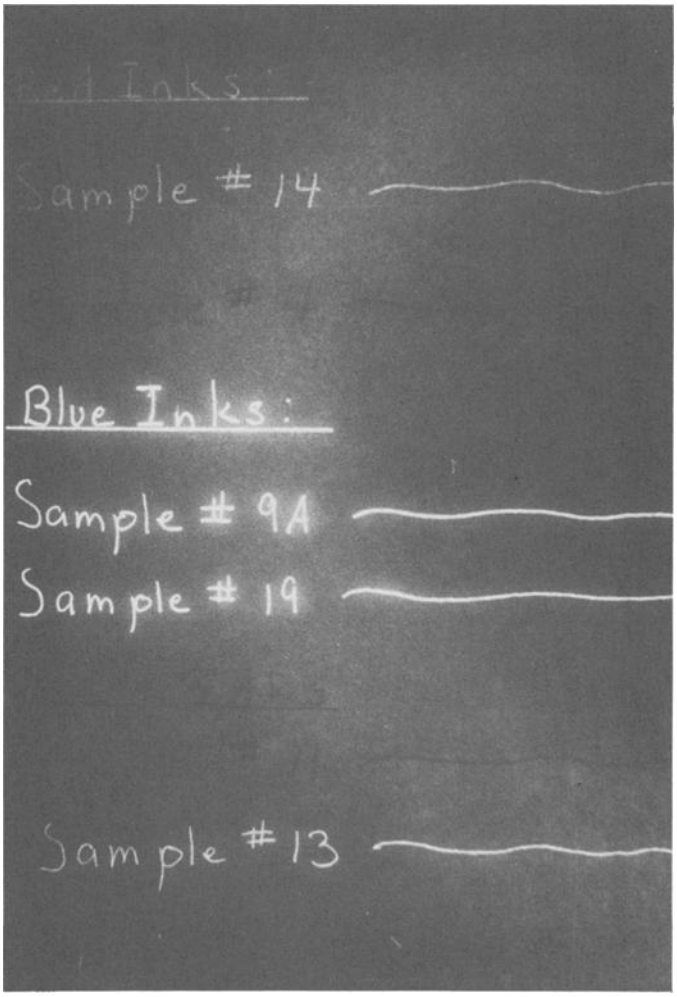


FIG. 2—*IR luminescence under 5145-A Ar-laser excitation for inks listed in Table 1.*

the near-infrared. The emission band at about 5800 A appears about five times stronger relative to the band at about 7200 A than it is in reality. For purposes of comparing inks, correction for spectral response versus wavelength is not needed since only differences in relative intensities are of interest. Indeed, it is perhaps preferable for court purposes to present actual, rather than massaged, spectra. The measurement of in situ excitation spectra of inks is difficult if luminescence yields are low. Accordingly, we have investigated in situ treatments to enhance the ink luminescence.

The most obvious and well-known way to increase luminescence efficiencies is to reduce the sample temperature. Figure 7 compares the room temperature and 77K (liquid N_2) spectra of ink Sample 19. Several comments are indicated regarding these spectra. The lower energy luminescence band (at about 7200 A) shows a much more pronounced intensity increase than the higher energy one when the temperature is lowered to 77K. This is to be expected. When molecules are in close proximity to each other, intermolecular energy transfer takes place in the direction from higher- to lower-lying excited states. The efficiency of

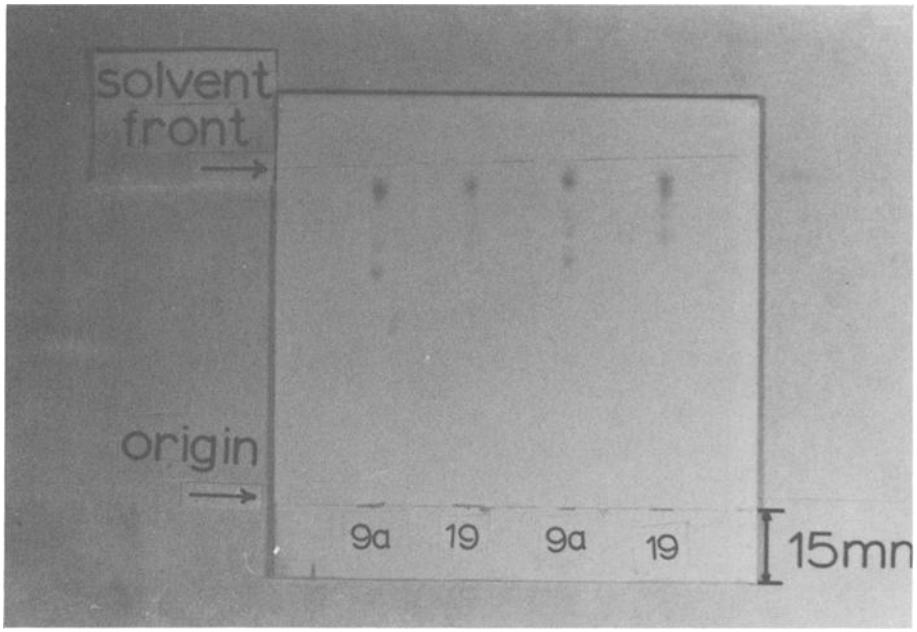


FIG. 3—*Room light photograph of TLC-developed blue Inks 9A and 19. The strengths and colors of the observed bands in direction from solvent front to origin are strong turquoise, weak turquoise, strong pink, weak purple and medium purple for Ink 9A, and strong turquoise, weak turquoise and strong pink for Ink 19. The film characteristics make the pink band appear weak in the photo.*

this energy transfer mechanism [*15*] is dictated not by temperature but by the distance between the interacting molecules. The generally observed decrease of luminescence with increase in temperature is due to molecular vibrations and to collisions between atoms and molecules. With rare exceptions, radiative decay (light emission) occurs at a considerably lower rate than nonradiative decay. Thus, the changes in the nonradiative deactivation channels dominate the luminescence intensity changes with temperature. For the species that gives rise to the higher energy luminescence (at about 5800 A), the two dominant deexcitation channels are the temperature-dependent radiationless decay and the intermolecular energy transfer route. As the temperature is lowered, the temperature-dependent decay route weakens. Since the intermolecular energy transfer route remains operative, however, the luminescence increase with decreased temperature is not as pronounced as it would be if there were no intermolecular energy transfer channel. For the species that gives rise to the lower energy luminescence (at about 7200 A) there is no intermolecular energy transfer decay route and the dominant mechanism is the temperature-dependent radiationless decay. Thus, the lower energy luminescence should increase in intensity more strongly as the temperature is decreased than the higher energy luminescence, as indeed seen in Fig. 7.

Comparison of Figs. 5 and 7 shows that the relative intensities of the luminescences at room temperature are not the same for ink Sample 19. This, too, is due to a temperature difference, but one arising from different laser illumination intensities (greater by about a factor of 4 for Fig. 5). If one uses a laser powerful enough for latent fingerprint development, one must keep the laser power low during document examination spectroscopic measurements, lest one risk charring the paper or destroying the ink by photodecomposition. Laser illumination can cause very substantial heating indeed. Thus, comparison of inks needs to be made under equal illumination and measurement conditions.

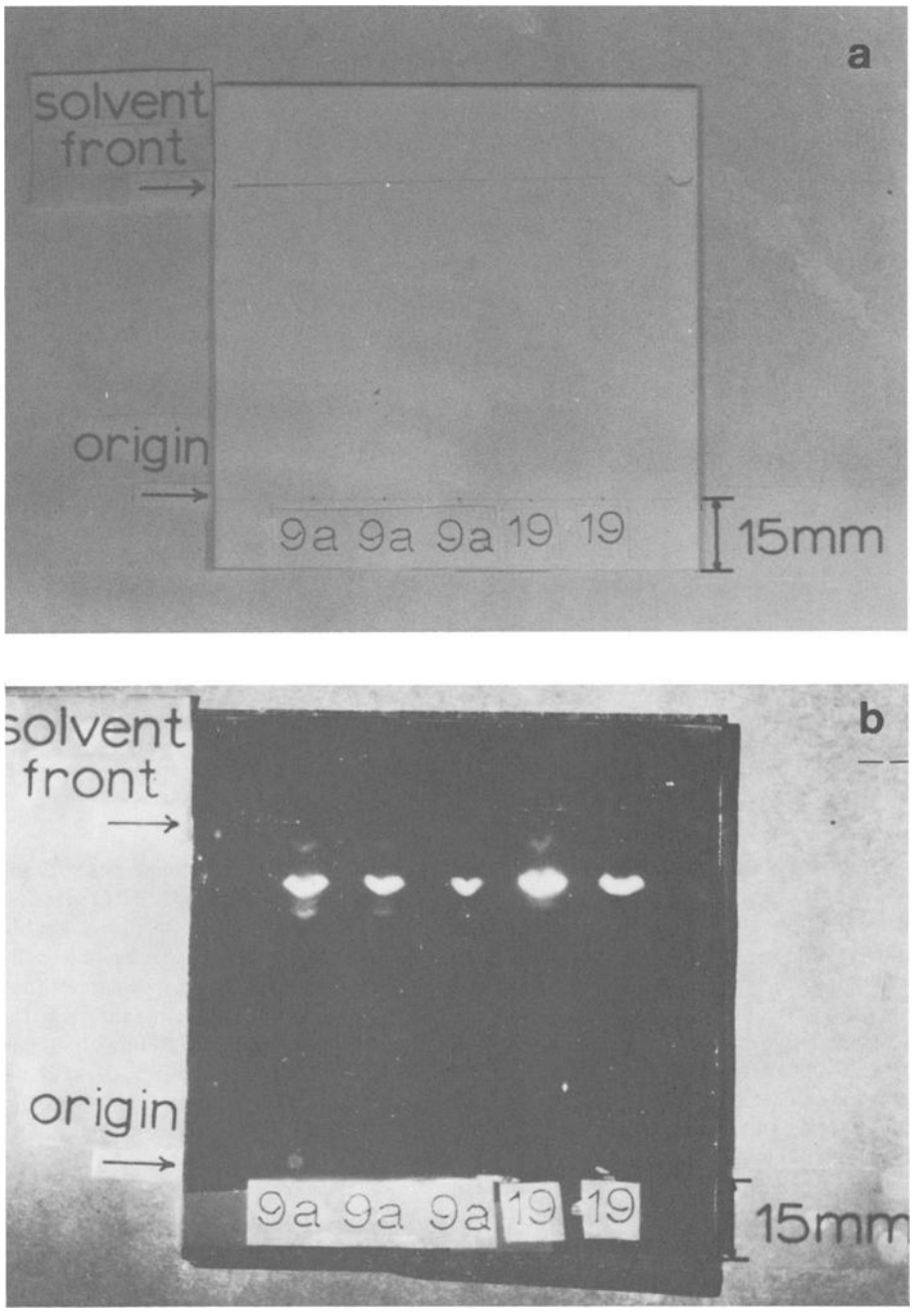


FIG. 4—*Room light* (a) *photograph of TLC-developed diluted samples of Inks 9A and 19; compared to Fig. 3, the dilutions in Fig. 4 are 1:100, 1:500, 1:1000 for 9A and 1:100, 1:1000 for 19. Visible luminescence* (b) *photograph of TLC plate of Fig. 4a under all-lines blue-green Ar-laser excitation. The strengths and colors of the observed luminescent bands (in direction from solvent front to origin) are medium red, strong yellow, weak orange, and medium red for 9A, and medium red, strong yellow, and weak yellow for 19.*

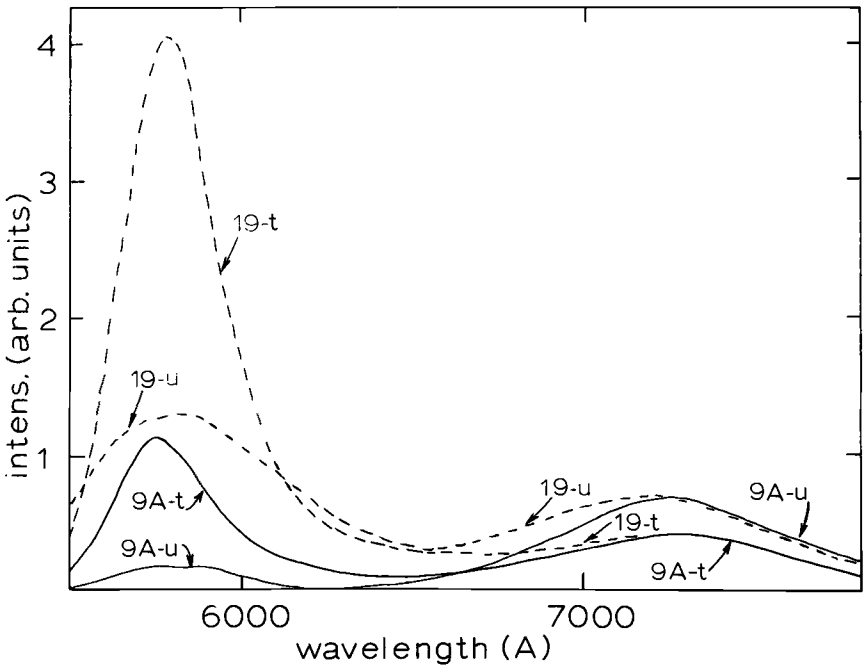
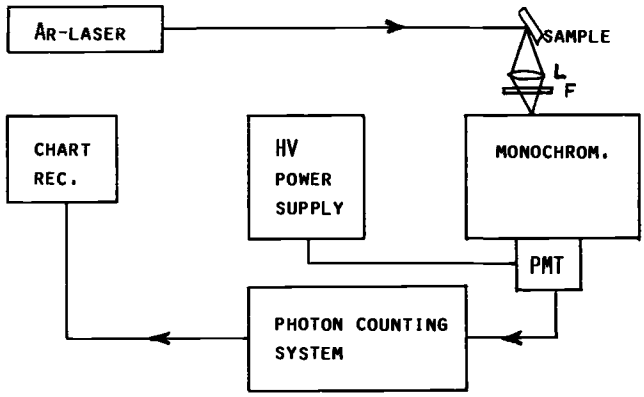


FIG. 5—*Room temperature emission spectra of Inks 9A and 19 under 5145-A Ar-laser excitation, untreated* (u) *and treated* (t) *as described in the text to enhance luminescence efficiency. Spectra are normalized for equal intensities of the untreated 7200-A emission band. The reduction of this band's intensity after treatment is as shown in the figure without any further normalization. See text for discussion.*



Ar-Laser = Spectra Physics 164-05, monochrom. = 0.3-m McPherson 218, PMT = EMI 9785B photomultiplier tube, high voltage (HV) power supply = Hewlett-Packard 6516A, photon counting system = PAR 1120 amplifier/discriminator + 1112 photon counter processor, chart recorder = Linear 1200, L = lens, F = filter.

FIG. 6—*Block diagram of experimental arrangement used to measure luminescence spectra.*

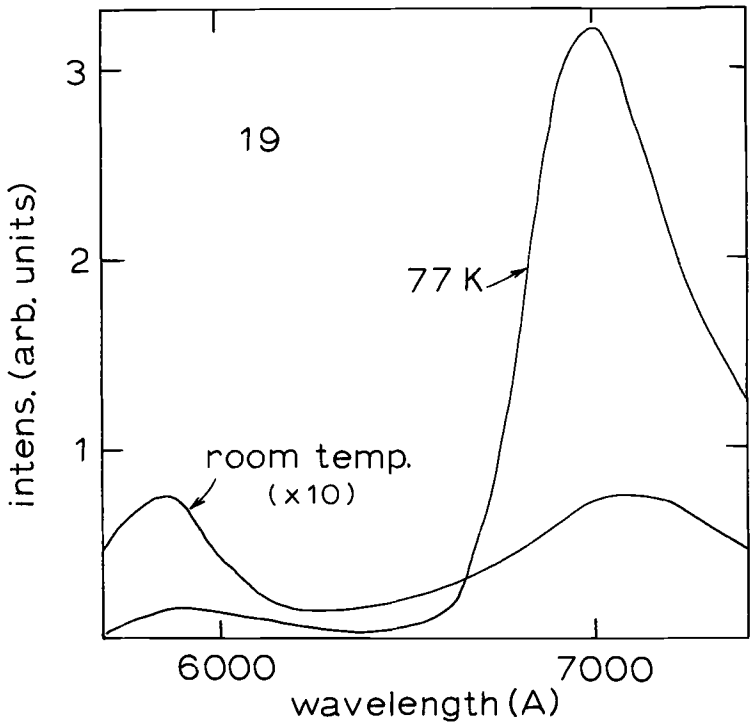


FIG. 7—*Room temperature and 77K emission spectra of Ink 19 (untreated) under 5145-A Ar-laser excitation. See text for discussion. The intensity of the room temperature spectrum is expanded by a factor 10. that is, the 5800-A band at room temperature is about 30% less intense than at 77K.*

Although spectroscopic measurements at cryogenic temperatures yield higher luminescence that at room temperature, they are somewhat cumbersome to perform. Indeed, the nature of the document under scrutiny may preclude low-temperature examination. Accordingly, we searched for means of in situ luminescence enhancement at room temperature. The foregoing discussion of the relative intensity changes of the luminescence bands of ink Sample 19 point toward a possible approach to achieve a luminescence enhancement. While one can do little about the temperature-dependent luminescence quenching as long as one remains at room temperature, one can increase intermolecular distances and thus reduce the luminescence quenching due to intermolecular energy transfer. If sufficient ink is on hand, one might consider extracting a sample of ink from the document and dissolving it in a suitable solvent to perform luminescence measurements. This would certainly lead to enhanced luminescence efficiency. However, the destructive nature of this procedure will often be unattractive. Luminescence enhancement for in situ room temperature examination can be achieved by coating the ink writing with a viscous liquid, containing a volatile solvent, with the following feature. As the solvent evaporates, a solid coating results, with some components of the ink dissolved into the coating. This increases intermolecular distances and thus leads to increased luminescence efficiency. Some bleeding of ink, that is, smudging of the writing, results from the migration of ink molecules into the coating, but this can be kept at an acceptable level by using a rapidly drying, but viscous, formulation. The use of white opaque correcting fluids (for example, Re-type®) represents a rather well-known instance of this situation. Sometimes these liquids are not very effective in covering writing because certain ink components (often red in color) dissolve into the correcting fluid, and thus the writ-

ing remains visible. Often, incidentally, the dissolved ink components also luminesce under laser excitation. However, since other components of the writing are covered by the correcting fluid, its use for our purposes leaves much to be desired. We thus investigated optically clear systems. We restricted our attention to commercially available formulations because of practicality considerations associated with technology transfer to the law enforcement user. An easily available and effective liquid is Cover Girl Nail Slicks®, which displays the requisite optical clarity, viscosity, ink solubility, and drying speed. Figure 8*a* shows a room light photograph of sample writings coated partly with this fluid. Figure 8*b* presents the visible luminescence of the same exhibit under blue-green Ar-laser excitation, showing the enhancement of luminescence at the coated locations. The luminescences for the red inks seen in Fig. 8*b* suggest substantial ink smudging. This is not the case. The smudged appearance is simply a matter of film overexposure as a result of the high luminescence intensity. For the blue ink Samples 9A and 19 dealt with so far, the enhanced luminescence spectra are shown in Fig. 5. For reasons already discussed, the higher energy luminescence band is enhanced, rather than the lower energy one, whose intensity decreases in fact because it is no longer fed as much by intermolecular energy transfer from the species which produces the higher-energy luminescence band and which is dissolved in the coating. The weaker yellow-orange luminescence intensity of Ink 9A relative to Ink 19 is evident in Figs. 5 and 8*b*. When feasible, a combination of the coating procedure with spectroscopy at cryogenic temperatures provides optimal luminescence efficiency enhancement. We point out that there is nothing very particular about the employed coating fluid. The important physical properties cited above can no doubt be satisfied by many formulations.

Blue Inks—Excitation Spectra

The excitation spectrum [*14*] of a luminescent compound reveals the same information as the absorption spectrum of that compound. Thus, excitation spectra are particularly valuable when compounds are located on opaque substrates such that absorption spectra cannot be measured. The excitation spectra corresponding to the species that produce the higher energy (about 5800 A) luminescence bands of Inks 9A and 19, enhanced by the foregoing coating procedure, were measured using an arrangement as shown in the block diagram of Fig. 9. The light source was a 150-W xenon lamp with nearly flat intensity between 4000 and 7000 A. The excitation monochromator was equipped with a grating blazed at 2500 A. As is seen from Fig. 10, the excitation spectra for the two samples are distinguishable even though they show similar general features.

Red and Black Inks

Our data on the foregoing blue ink samples give an indication that inks that are very similar to each other can indeed be distinguished by in situ luminescence and excitation spectra. It remains to be shown that inks that are somewhat more obviously different display very flagrantly different in situ luminescence and excitation spectra, and that such spectra are identical when inks of identical formulation are analyzed. We demonstrate this by considering a pair of red inks (Samples 4 and 14) and a pair of black inks (Samples 12 and 13) which have these features, respectively. Note that Sample 12 is not shown in Fig. 2.

Visual examination in room light of writing with red Inks 4 and 14 shows that these inks have slightly different shades of red. Thin-layer chromatography, solution absorption, and IR luminescence (see Fig. 2) showed clearly distinguishing features as well. Emission and excitation spectra of these inks (after enhancement via the coating procedure presented earlier) are shown in Figs. 11 and 12, respectively. The differences are seen to be very pronounced indeed.

Tracings of two black inks (Samples 12 and 13) were spectroscopically examined after

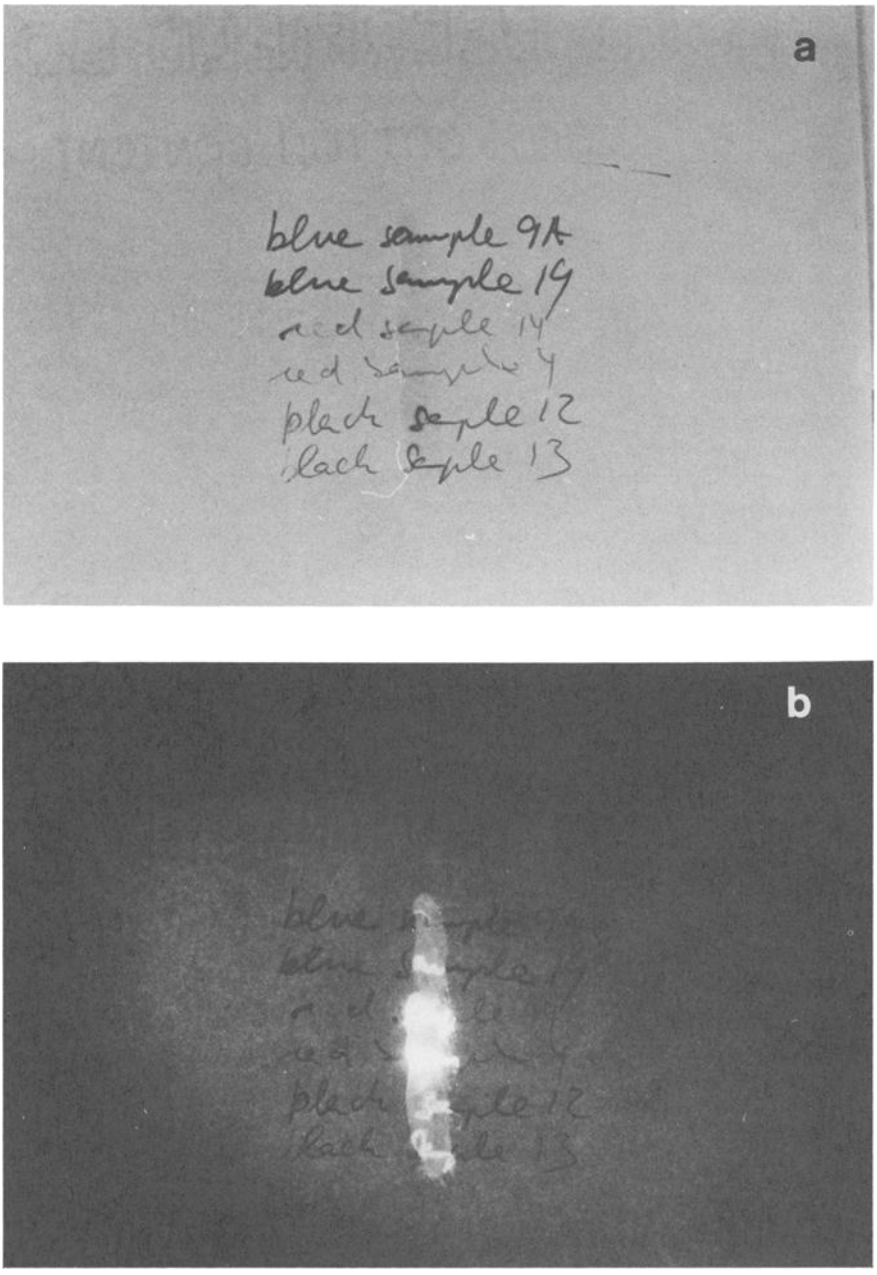
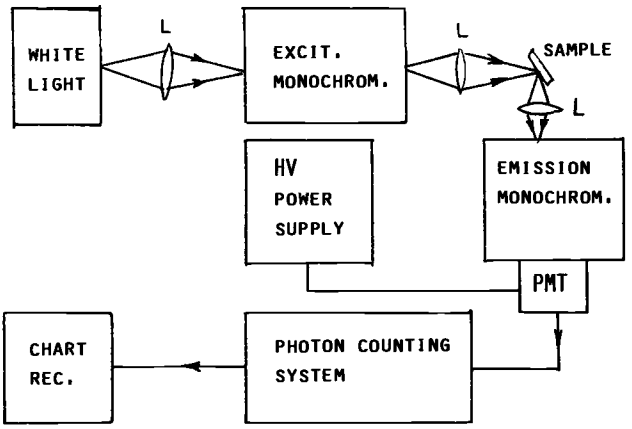


FIG. 8—*Room-light photograph* (a) *of writing with blue, red, and black inks, partially coated with Cover Girl nail Slicks and visible luminescence photograph* (b) *of same exhibit under all-lines blue-green Ar-laser excitation. The strengths and colors of the enhanced luminescences are, from top to bottom, weak yellow-orange, medium yellow-orange, strong orange, strong yellow-orange, medium red, medium red. The smudged appearance of the strong luminescences is due to film overexposure.*



White light = 150-W xenon lamp with Ealing 27-1031 lamp housing and 27-1015 power supply, excitation monochromator = Photochemical Research Assoc. 3102-1, 1/10 m, monochromator, remaining instruments as detailed in caption of Fig. 6.

FIG. 9—*Block diagram of experimental arrangement used to measure excitation spectra.*

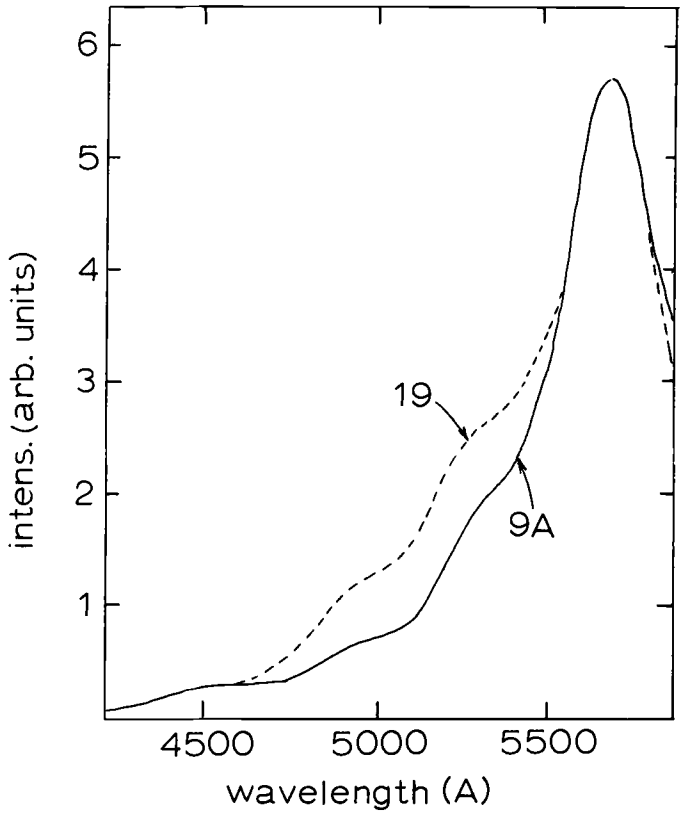


FIG. 10—*Room temperature excitation spectra of Inks 9A and 19 after coating to enhance luminescence. The emission monochromator was set at 6000 A.*

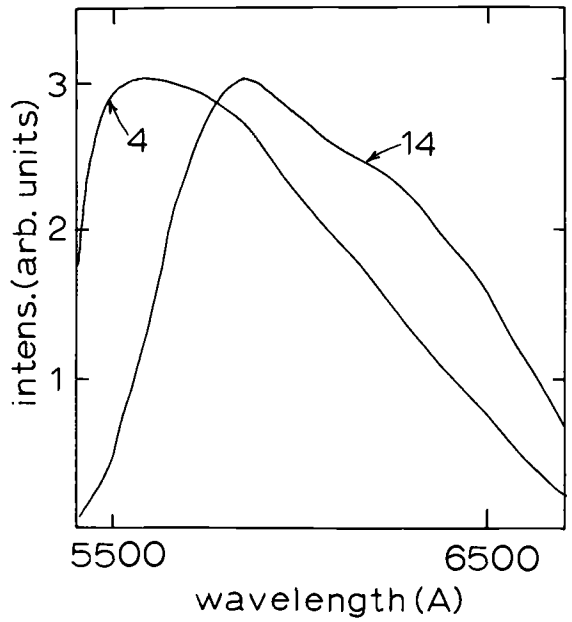


FIG. 11—*Room temperature emission spectra of coated red Inks 4 and 14 under 5145-A excitation.*

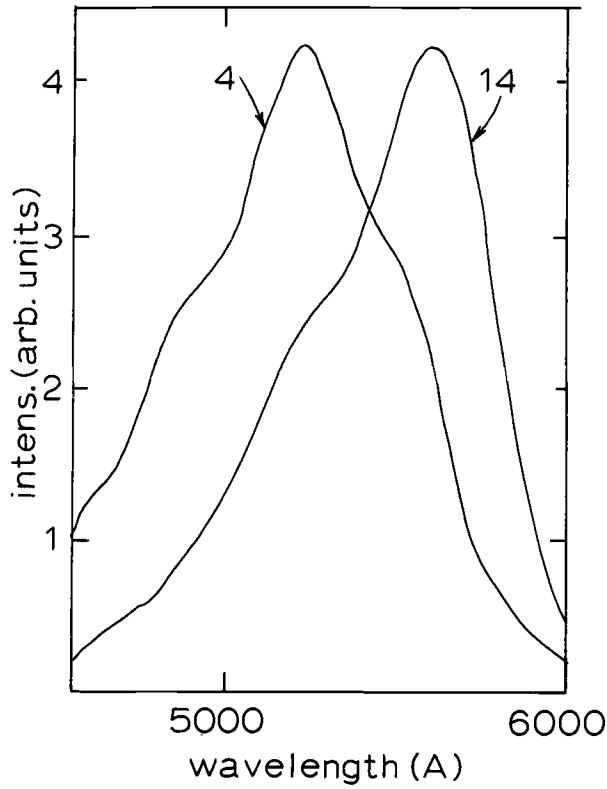


FIG. 12—*Room temperature excitation spectra of coated red Inks 4 and 14. The emission monochromator was set at 6200 A.*

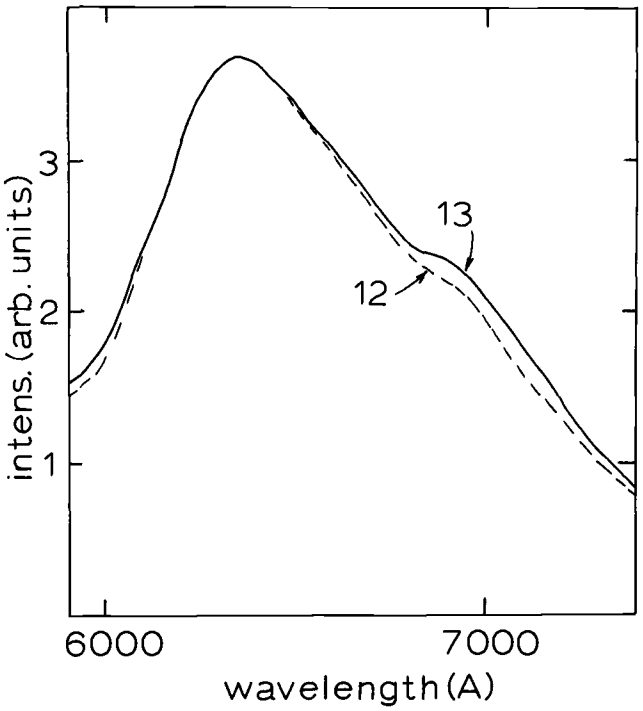


FIG. 13—*Room temperature emission spectra of coated black Inks 12 and 13 under 5145-A excitation.*

coating to enhance luminescence. The similarity of in situ luminescence and excitation spectra, as seen in Figs. 13 and 14, respectively, suggested to us that these inks (from different model pens made by the same company) are of the same composition. This was corroborated by TLC examination.

Discussion

The photographs of this article are necessarily black-and-white. Thus, differences in color shades or equality in color cannot be seen. Intensity differences do show up, however.

Since writing pressure can vary substantially, even within a letter or number, absolute intensities in spectra are far less significant than differences in spectral shapes, that is, differences in relative intensities at various wavelengths, when spectra are compared. Accordingly, we present spectral comparisons with normalization to equal intensities at a given peak.

Because inks tend to be suspensions, rather than true solutions, one can anticipate small composition fluctuations within a given sample. Accordingly, minor spectral differences should not be taken as a basis for concluding ink difference. The small differences in Figs. 13 and 14, therefore, are of no significance. Moreover, in instances in which spectra show considerable similarity, measurement of different types of spectra help to arrive at an unambiguous conclusion. Thus, the absorption spectra of Fig. 1 by themselves do not make for a very firm deduction that Inks 9A and 19 are of different composition. However, when the luminescence and excitation spectra of Figs. 5, 7, and 10 are added, the difference between the two samples becomes unambiguous. The difference between the two inks becomes particularly obvious when the room-light and laser-luminescence photographs after coating for

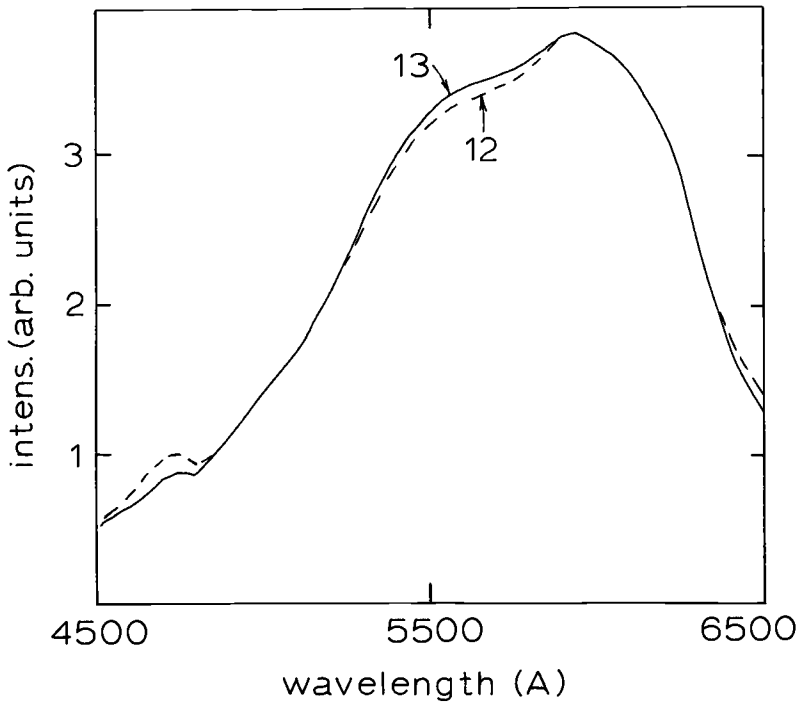


FIG. 14—*Room temperature excitation spectra of coated black Inks 12 and 13. The emission monochromator was set at 6700 A.*

luminescence enhancement are also examined (Fig. 8). While the amount of inks in the written samples is very comparable, the difference in enhanced luminescence intensity is flagrant. This demonstrates that the coating procedure can come in handy in permitting ink discrimination without need for spectroscopic examination in instances in which the untreated writings would be indistinguishable by visual examination in room light as well as visible and infrared luminescence. The coating procedure therefore adds to the discriminatory value of straightforward visual laser examination. Furthermore, the coating step to enhance luminescence is valuable in the sense of permitting one to measure in situ excitation spectra that otherwise would not be obtainable. There is no reason why a similar coating procedure should not be applicable to laser latent fingerprint development as well. We are currently engaged in exploring this possibility.

Laser spectroscopy for document examination is very expensive compared to conventional IR luminescence or TLC examination (but not compared to microspectrometry). However, when a laser is already on hand for fingerprint work, then spectroscopic examination can be implemented relatively inexpensively. Some laboratories are already equipped with fluorescence spectrophotometers, and these are easily adapted to laser use. Small monochromators suffice since wavelength resolution is not an issue, as is obvious from the presented spectra. An inexpensive photomultiplier tube and a photon counting system less sophisticated than that of our apparatus will give very adequate sensitivity. However, photon counting cannot be used with the pulsed Cu-vapor lasers that some agencies use for fingerprint work. This laser requires AC detection with, for instance, a lock-in amplifier. Because of the low pulse repetition rate, the frequency-doubled neodymium:yttrium aluminum garnet (Nd:YAG) lasers that are used for fingerprint work are of limited use in spectroscopic work. However, these lasers are intended for crime scene work, where spectroscopic measurements are not

pertinent. Even if one is not equipped with spectroscopic machinery, lasers, when on hand, should not be overlooked as useful tools for document examination, not only in the sense of direct visual examination (see Figs. 2 and 8), but also as a tool to enhance the sensitivity of TLC (see Fig. 4). Finally, we note that lasers are not essential for measurement of luminescence spectra of inks, particularly when cryogenic or coating enhancement are implemented. For visual examination and photography, however, lasers powerful enough to be suitable for fingerprint work are far superior to conventional light sources.

Acknowledgment

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Address requests for reprints or additional information to
E. R. Menzel
Center for Forensic Studies
P.O. Box 4270
Texas Tech University
Lubbock, TX 79409