# TECHNICAL NOTE

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# The Characterization of Small Quantities of Luminescent Invisible Detection Materials Using the Docuspec TM/1 Microspectrophotometer

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**ABSTRACT:** A simple method has been developed for the adaptation of a Docuspec TM/1 microspectrophotometer in obtaining emission spectra of minute quantities of luminescent materials. One of the forensic science applications of this method was demonstrated: its use for the characterization of luminescent invisible detection materials.

**KEYWORDS:** forensic science, luminescence, spectroscopic analysis

Luminescence techniques have many uses in forensic science examinations of various materials such as glass, fibers, gunshot residues, luminescent invisible detection materials, and so forth [*1-3*].

A microspectrophotometer has been found to be a valuable instrument in certain types of forensic science examinations such as the comparison and identification of small quantities of paints, inks, and fibers [*4-6*].

In our laboratory in the Criminal Identification Division of the Israel Police, a simple method was developed for adapting a Docuspec TM/1 microspectrophotometer for recording luminescence spectra. This method was applied for the characterization of minute quantities of invisible detection materials used by the Israel Police.

## Experimental Procedure

The system used was the Docuspec TM/1 microspectrophotometer which includes an Olympus BHT microscope with quartz-halogen lamps (50 W) for reflectance and transmission work. The system is equipped with a variable measuring aperture and its wavelength range is from 380 to 764 nm.

Through the coupling of the quartz-halogen illumination (originally provided for reflectance spectrophotometry) with a Kodak 18A glass filter, enough ultraviolet (UV) intensity

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was obtained to make possible the recording of emission luminescence spectra in the visible range. This is possible because the filter transmits near UV and some infrared (IR) radiation, while cutting off the visible range (Fig. 1 [7]).

With this small modification of the insertion of an 18A filter and an increase of the "gain" of the photomultiplier, the system is changed from a microspectrophotometric mode to a microspectrofluorometric mode. It should be emphasized that when this method is used, the emission is due to a broad excitation which is not the regular operational mode of a spectrofluorometer [*8*].

The software accompanying the Docuspec TM/1 dictates that either transmission or reflectance spectra of the samples can be recorded only relative to a reference in an optical density mode. The reference is recorded as an intensity of light versus wavelength. The obvious method used to obtain emission spectra of luminescent materials is the same used for recording a reference spectrum in the spectrophotometric mode of the Docuspec TM/1. The spectra obtained in this way are a convolution of luminescence intensity, diffraction efficiency of the grating, and the spectral response of the photomultiplier.

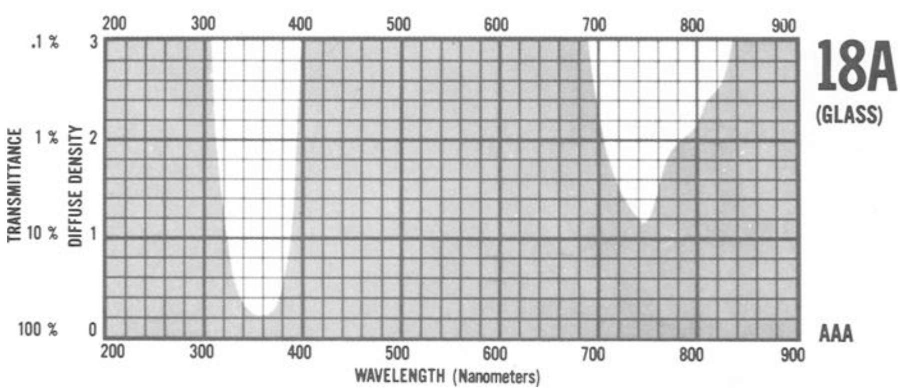
The luminescent invisible detection powders used for this study were Fargo Corporation's BB-20, BB-25, and BB-29. Several particles of the various powders were mounted on 1-in. (2.54-cm) stubs used in scanning electron microscopic (SEM) work that had been coated with double-sided adhesive (3M's #465).

Ordinarily, in real casework, the amount of luminescent invisible detection particles found is limited; it is therefore convenient that the same sample-mounted stub used for luminescence analysis can later be examined in the scanning electron microscope/energy dispersive spectroscope (SEM/EDS) for elemental composition.

A ×40 objective was used for observing the particles and recording their spectra. The variable aperture was adjusted for a measurement area of approximately 15 by 15 $\mu$m, which was the size of the single particles or clusters chosen for obtaining spectra. This size was sufficient to obtain satisfactory spectra without too much electronic noise.

## Results and Discussion

Figure 2 depicts the spectrum of the white standard used (supplied with the Docuspec TM/1) which does not exhibit luminescence in the visible range. This spectrum confirms that an 18A filter is indeed a good barrier for visible radiation. Although there is some trans-




Glass filter. Only transmits ultraviolet radiation between about 300 and 400nm (e.g., 365nm line of mercury spectrum) and infrared radiation. Used for ultraviolet reflection photography.

FIG. 1—*Transmittance characteristics of a Kodak 18A glass filter* [7].

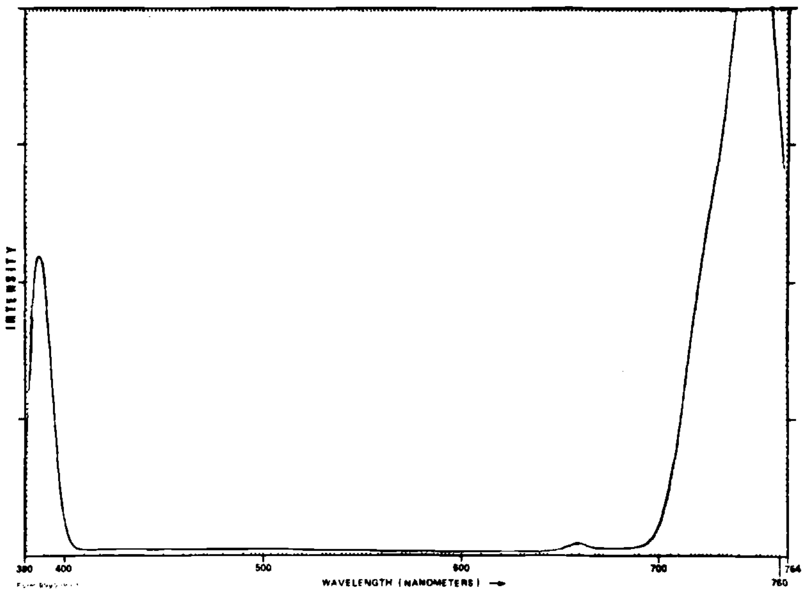



FIG. 2—*Reflectance spectrum of the white standard illuminated by filtered (Kodak 18A filter) quartz halogen lamp and recorded in the reference mode.*

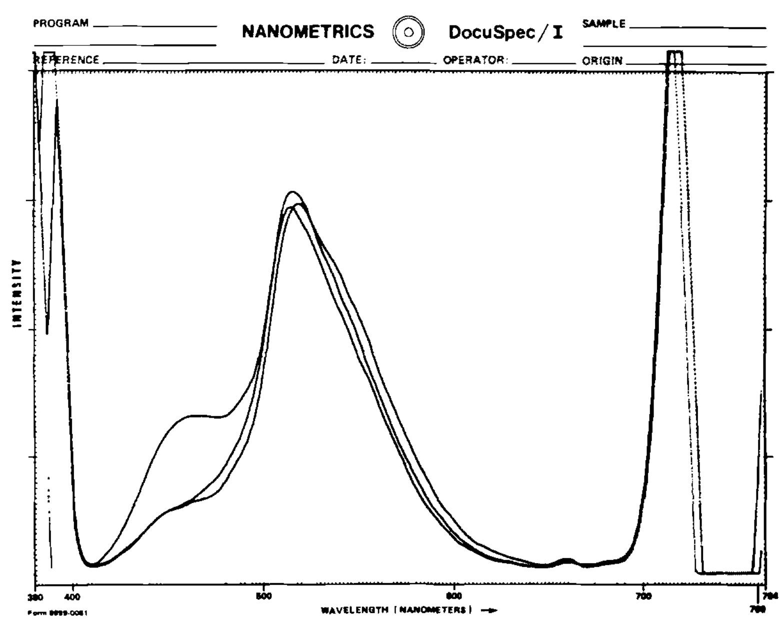



FIG. 3—*Emission spectra of three different luminescent particles from BB-20 powder.*

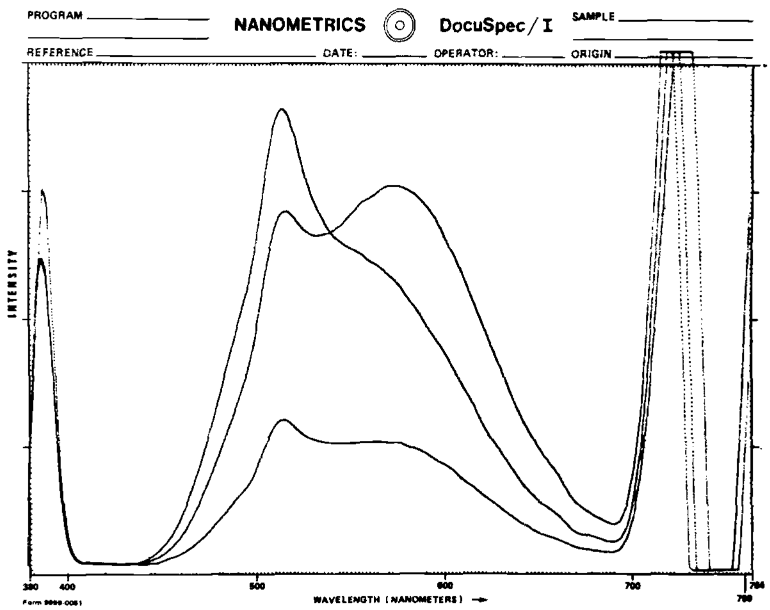



FIG. 4—*Emission spectra of three different luminescent particles from BB-25.*

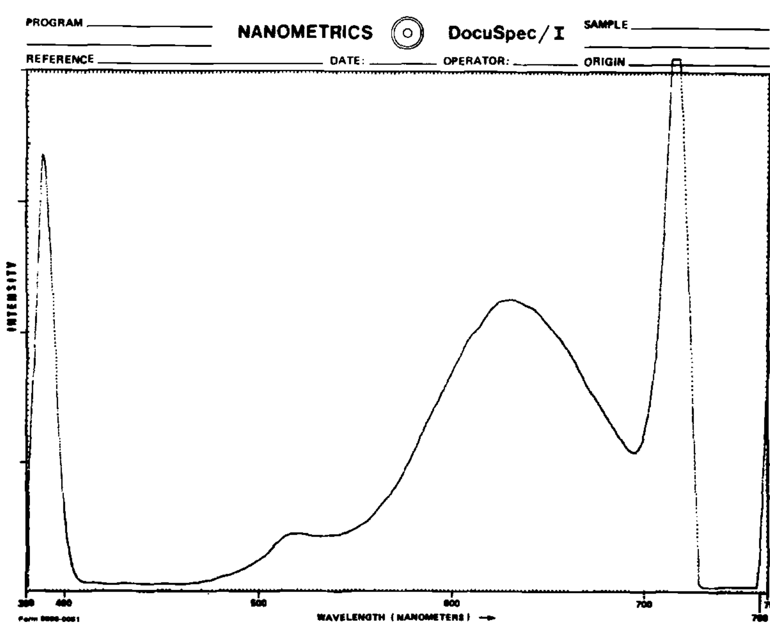



FIG. 5—*A typical emission spectrum of a particle from BB-29 powder.*

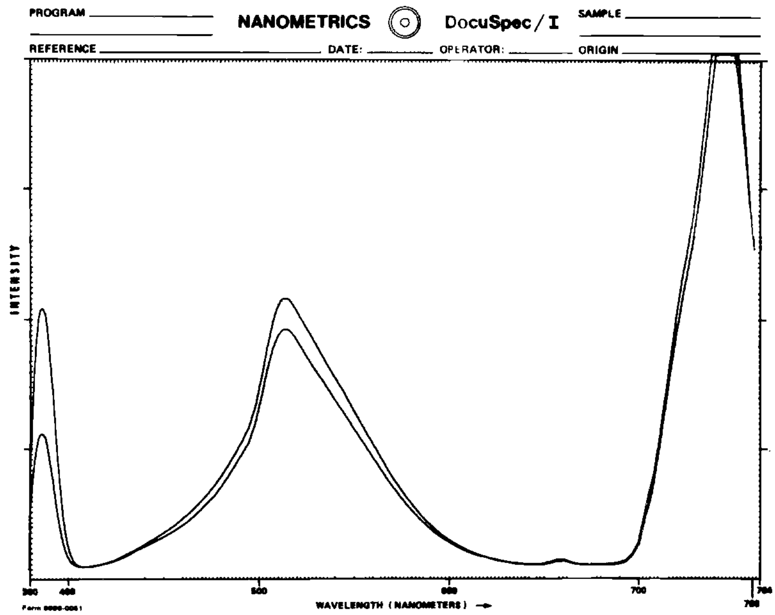



FIG. 6—*Comparison of spectra of a particle from clothes of a suspect and a particle from BB-20 powder used to prepare the "trap."*

mission in the region of 660 nm, it barely interferes with the comparison of luminescence spectra in the visible range.

While the colors of the luminescent invisible powders appear homogeneous to the unaided eye (BB-20 yellowish-green, BB-25 orange-yellow, and BB-29 red), examination with the microscope shows that they are really quite heterogeneous.

Figures 3 and 4 show spectra of three arbitrarily chosen particles which exhibit different colors that are found frequently in BB-20 and in BB-25 powders, respectively.

Figure 5 exhibits the typical spectrum of a BB-29 particle. In the B-29 powder, particles exhibiting blue luminescence were rarely found.

Figure 6 is taken from a real case in which a "trap" was prepared using BB-20 powder. When examining the clothes of the suspect, very few particles were found. As seen in the figure, a match was found between the spectrum of a luminescent particle found on the clothes and the spectrum of one of the BB-20 particles.

## Summary

Through a small modification, a Docuspec TM/1 was changed from a microspectrophotometric mode to a microspectrofluorometric mode. This method, having been proved efficient, was employed as a routine technique used in our laboratory for the characterization of invisible detection materials in addition to SEM/EDS analysis.

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