



## Questioned Documents Section - 2014

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### J3 Black Ink Analysis With Miniaturized UV-Vis Spectrometry and Capillary Electrophoresis

Victoria M. Bohme\*, 106-35 97th Street, Ozone Park, NY 11417; Courtney M. Grimes, BS, Chemistry Dept, 151 Hofstra University, Hempstead, NY 11549; Amir Mullick, BS, Chemistry Dept, 151 Hofstra University, Hempstead, NY 11549; Nicholas D. Petraco, PhD, John Jay College of Crim Just, Dept of Science, 899 10th Avenue, New York, NY 10019; and Ling Huang, PhD, Chemistry Dept, 151 Hofstra University, Hempstead, NY 11549

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After attending this presentation, attendees will understand how ink can be extracted from paper samples, studied with UltraViolet Visible (UV-Vis) spectroscopy, and separated with Capillary Electrophoresis (CE), all in one buffer solution with surfactant. Attendees will also learn how spectra and electropherograms can be analyzed using Principle Component Analysis (PCA) to find patterns and to match unknown samples with library ink profiles.

This presentation will impact the forensic science community by providing a simple, green, inexpensive, and rapid method for the effective extraction and spectral characterization of written ink. The streamlined protocol can be utilized to generate spectral and electrophoretic profiles from extracted ink for rapid ink comparison.

Conventional written ink analysis utilizes organic solvent extraction and Thin Layer Chromatography (TLC) separation which works well with organic-based ball pen ink.<sup>1</sup> The popularity of water-based ink such as gel pen ink and inkjet printer ink presented challenges to conventional methods due to the low solubility of the aqueous ink in organic solvent such as pyridine. The study proposes that the usage of surfactant-based buffer solutions can enhance the extraction efficiency of versatile ink samples while maintaining compatibility with downstream analytical methods.

Fifteen black ink samples from various sources, including ball-tip pens, gel pens, inkjet printer, and fountain pens, were prepared by writing or printing ink strokes on one type of white printer paper. Six holes of 1mm diameter were punched out of one stroke using the tip of a mechanical pencil before the ink was extracted in 20 $\mu$ L of 1% Sodium Laureate Sulfate (SDS) and borate buffer at a slightly basic pH, with agitation provided by a sonicator. Only 2 $\mu$ L of the extract solution were subsequently analyzed with a miniaturized UV-Vis spectrophotometer to generate a complete UV-Vis absorbance spectrum. The components of these remaining ink extracts, after dilution in the same SDS buffer, were separated using a CE method, particularly micellar electrokinetic chromatography with the same SDS borate buffer as run buffer. UV-Vis absorbances of separated components were used for signal detection with a Diode Array Detector (DAD).

After UV-Vis scanning and CE separation, the UV-Vis spectra and electropherograms were analyzed through PCA with open-source R program code. The 3D PCA plot indicated the clustering of similar type of ink samples. This novel ink analytical method utilizes the same SDS buffer for extraction, UV-Vis and CE, which facilitates the efforts for rapid profiling of various ink samples. The use of surfactant and water as solvent avoids toxic organic solvent and presents a greener alternative to conventional methods. Ink components from six hole-punches were effectively separated and spectroscopically probed with the miniaturized UV-Vis and the DAD on the CE system, which made this method a promising applicable option for practical question document examinations.

#### Reference:

1. ASTM Standard Guide for Test Methods for Forensic Writing Ink Comparison, E1422-05.
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#### Ink Analysis, UV-Vis, CE