B170 Fiber Dye Identification by High Performance Liquid ChromatographyMass Spectrometry (HPLC-MS)

Min Huang, PhD, Jehuda Yinon, PhD, and Michael E. Sigman, PhD, National Center for Forensic Science, University of Central Florida, PO Box 162367, Orlando, FL 32816-2367*

The goal of this presentation is to present the progress on the development of a mass spectrometry-based method for the forensic comparison and identification of dyes extracted from textile fibers.

The results of this study offer an improved method for single source determination in fiber evidence comparisons. Molecular-level information from MS data opens the possibility for dye identification at the molecular level and the preparation of searchable libraries.

Textile fiber examination is frequently carried out in forensic laboratories to provide physical evidence in crime investigations. An important part of forensic fiber examination involves the characterization of textile dyestuffs. Compared with currently used methods, such as ultraviolet, visible, and fluorescence microspectrophotometry, infrared spectrometry, and high performance liquid chromatography (HPLC); high performance liquid chromatography-mass spectrometry (HPLC-MS) provides unique information about the molecular structure of fiber dyes and makes questioned and known comparisons and/or dye identification highly discriminating.

Determination of the elution times for extracted dyes is one of the key issues in applying HPLC-MS to the analysis of dyes. In the absence of a prior knowledge of a dye's elution time and/or the major ions formed from the dye, HPLC-MS analysis may be impractical, if not impossible, for the analysis of many dyes. This project utilizes tandem UV-visible absorption and mass spectrometry to facilitate the detection of dyes separated by HPLC. With visual information about the color of the textile fibers, the elution of the dyes can be efficiently pinpointed by a single wavelength UV-visible detector. Diode array detectors offer an even broader capability for detecting the elution of multiple dyes. In this way, even dyes that do not show an obvious peak in the total ion current (TIC) chromatogram can be analyzed. The method also allows extracted brightening agents and other non-dye components to be distinguished from the peaks of interest.

An Agilent 1100 MSD quadrupole mass spectrometer equipped with an electrospray ionization (ESI) source and an Agilent 1100 HPLC have been used for this study. The instrumentation allows both positive ions and negative ions to be monitored according to a dye's tendency to form negatively or positively charged species. Separation was carried out with a ZORBAX Eclipse XDB-C18 (2.1 x 150 mm) HPLC column. A mobile phase flow rate of 0.20 ml/min was utilized, and a solvent gradient was used to achieve better separation. Mass spectrometer parameters, which were optimized for sensitivity, included the drying gas flow rate for the electrospray interfase (ESI) at 12.0 L/min, and the ESI spray chamber temperature at 350 ºC.

A number of textile fibers were extracted and the dyes identified using the LC-MS method. Mass spectra of standard dyes and extracted dyes will be presented and discussed. Dye identification is based on multi-analyte ions, including various molecule-related ions and fragment ions. Other advantages of LC-MS, such as isotope information and the comparison of negative and positive ion spectra, will be discussed.

Textile Fiber, HPLC-MS, Dye Analysis

* *Presenting Author*