B101 Single Fiber Dye Analysis by Liquid Chromatography Mass Spectrometry (LC-MS) With SWGMAT Dye Extraction Protocol

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The objective of this presentation is to demonstrate that the dye from a single fiber can be extracted following the SWGMAT protocol and then subsequently analyzed by liquid chromatography mass spectrometry (LC- MS) with an electrospray interface in series with a single wavelength UV/VIS absorbance detector, monitoring at a wavelength previously deter- mined by microspectrophotometry.

This presentation will impact the forensic community and/or humanity by providing forensic laboratories with an additional technique for discriminating between single fibers when all other non-destructive methods of comparison fail to discriminate the two.

Textile fibers are encountered frequently in forensic casework and comparison of questioned and known fibers occurs regularly. A single fiber represents the smallest evidentiary unit for which robust analytical methods must be available. There are several non-destructive techniques (*e.g.* polarized light microscopy, fluorescence microscopy and microspec- trophotmetry) which are currently employed to discriminate between ques- tioned and known single fibers. When these methods fail to discriminate, alternative techniques such as dye extraction, ultraviolet-visible range (UV/VIS) spectroscopy and liquid chromatography-mass spectrometry (LC-MS) offer a different, yet destructive approach. LC-MS offers advan- tages over other separation techniques such as thin layer chromatography (TLC) because LC-MS will not only provide separation, but also mass fragmentation unique to the dye. An LC-MS can also be coupled in series with a UV/VIS detector to aid in the detection of conjugated compounds.

The UV/VIS detector can monitor a single wavelength (user defined) and will nondestructively detect compounds that absorb at the specified wavelength. This detection system can facilitate the analysis of single fiber extracts where the dye concentration may be very low. The signal to noise ratio for dilute dyes under LC/MS analysis may be below the limit of detection without the aid of UV-visible absorbance monitoring. If the dye absorbs at the specified wavelength, the UV/VIS detector will aid in deter- mining the retention time. Preliminary analysis by microspectropho- tometry will provide a viable maximum absorbance wavelength to monitor when performing the chromatographic separation. A SEE model 1100 microspectrophotometer was used to obtain a visible absorption spectrum for each fiber sample examined in this research.

Previous work has shown that fiber dyes can be extracted using methanol as a solvent and then analyzed by LC-MS (1); however, methanol extraction does not offer the dye classification information afforded by the SWGMAT protocols. Five fiber samples previously analyzed by methanol extraction (1) were reanalyzed following the SWGMAT dye extraction pro- tocol. Each dye was extracted from the fiber with high efficiency. A thread of each sample was analyzed separately to confirm the single fiber results retention time, mass spectral fragmentation). All separations were performed on a C18 reverse phase column and the instrumentation used was an Agilent 1100 Series LC-MS with an electrospray ionization source and a variable wavelength UV/Visible absorbance detector. Control blank samples consisting of the extraction solvent(s) were analyzed in each case.

Overall, LC-MS proves to be a convenient yet sensitive technique for the analysis of single fiber dye extracts, and is compatible with SWGMAT dye extraction protocols. All single fiber extracts were detected using both the single wavelength detector and the mass selective detector. Single fiber analysis would benefit by incorporating this technique into the investigative routine due to the techniques high discriminating power.

References

(1) Huang, M. PhD, Yinon, J. PhD, Sigman, M. PhD (2004) Forensic Identification of Dyes Extracted from Textile Fibers by Liquid Chromatography Mass Spectrometry (LC-MS). *J. Forensic Sci*., 49(2): 1-12.

LC-MS, Fiber Analysis, Fiber Dyes

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