

Y/DK-1185

Analysis of Dyes Extracted from Millimeter-Size Nylon Fibers by Micellar Electrokinetic Chromatography

July 30, 2001

Analytical Chemistry Organization
Y-12 National Security Complex
Oak Ridge, Tennessee 37831
Managed by
BWXT Y-12 L.L.C.
for the
U.S. Department of Energy
Under Contract No. DE-AC05-00OR22800

**Abstract Submission to the American Academy of Forensic Sciences
54th Annual Meeting in Atlanta, GA
February 11-16, 2002**

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

COPYRIGHT NOTICE

This document has been authored by a subcontractor of the U. S. Government under contract DE-AC05-00OR-22800. Accordingly, the U. S. Government retains a paid-up, nonexclusive, irrevocable, worldwide license to publish or reproduce the published form of this contribution, prepare derivative works, distribute copies to the public, and perform publicly and display publicly, or allow others to do so, for U. S. Government purposes.

Analysis of Dyes Extracted from Millimeter-Size Nylon Fibers by Micellar Electrokinetic Chromatography

Linda A. Lewis, Ph.D.;Gerald L. DeVault, Ph.D.; Samuel A. Lewis, Sr., M.S.; and Robert W. Smithwick, III, Ph.D.;
Development Chemists, Analytical Chemistry Organization, 113C Union Valley Road, Oak Ridge, TN 37830.

Learning Objective: to present to the forensic community a potential qualitative/quantitative method for trace-fiber color comparisons using micellar electrokinetic chromatography (MEKC).

Developing a means of analyzing extracted dye constituents from millimeter-size nylon fiber samples was the objective of this research initiative. Aside from ascertaining fiber type, color evaluation and source comparison of trace-fiber evidence plays a critical role in forensic-fiber examinations. Literally thousands of dyes exist to-date, including both natural and synthetic compounds. Typically a three-color-dye combination is employed to affect a given color on fiber material. The result of this practice leads to a significant number of potential dye combinations capable of producing a similar color and shade. Since a typical forensic fiber sample is 2 mm or less in length, an ideal forensic dye analysis would qualitatively and quantitatively identify the extracted dye constituents from a sample size of 1 mm or smaller. The goal of this research was to develop an analytical method for comparing individual dye constituents from trace-fiber evidence with dyes extracted from a suspected source, while preserving as much of the original evidence as possible.

The method outlined herein was found to be capable of detecting acid dyes, extracted from dark- and light-colored nylon fibers using as little as 1-mm of material. Large-volume stacking and polarity switching in a micellar electrokinetic chromatography (MEKC) system were employed to achieve the research goal. Anionic-acid dyes were extracted from individual nylon fibers using a 4:3 pyridine/water solvent. Trace amounts of pyridine proved detrimental to the separation, necessitating a solvent exchange with water. The optimized running-buffer system consisted of 50 mM SDS/10mM Borate/17% methanol at an approximate pH of 9.5. A sample volume of 500 nL was injected into a 75-micrometer column by pressure prior to applying a -25 kV reverse-polarity voltage. At this point, neutral and positively charged components were effectively backed-out of the column into a waste vial, while the anionic dyes were concentrated at the front of the column. Once the current reached -50 microamps, the voltage was changed to +25 kV for the anionic-dye separation. Using this injection technique, matrix interferences that affected reproducibility, such as water-soluble nylon monomers and low-molecular-weight polymers, were effectively removed and sample stacking was accomplished. When employing this method, acid-dye limits-of-detection were reduced from ~600 ppb (large-volume normal injection) to ~78 ppb. In addition, this injection technique resulted in a notable increase in separation efficiency and resolution.

Nylon-fiber samples and corresponding dye components were collected from manufacturers for validation purposes. Sample extracts were analyzed by the method described above using a capillary electrophoresis/diode array detection system. Extracted dyes were identified using both the dye-retention time and UV-Vis spectrum. The capability of evaluating the visible region, as well as UV, was crucial for distinguishing the dyes from other UV-absorbing components. An unexpected consequence of this on-column separation technique was the detection of other UV absorbing fiber-constituents, such as UV protectants, surfactants, binders, dye impurities, etc., that yield a potential fingerprint of the colored fiber material. Figure 1 illustrates an electropherogram obtained from the analysis of a 1-mm brown nylon fiber.

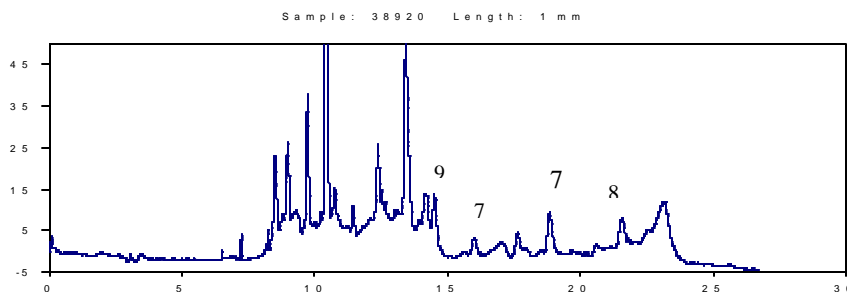


Figure 1. Electropherogram obtained from the extract of a single, 1-mm brown nylon fiber containing three dyes labeled 7, 8, and 9.

Peaks corresponding to known extracted dyes are labeled 7, 8, and 9. All other peaks were derived from extracted UV-absorbing constituents.

Nylon Fiber, Acid Dye, Capillary Electrophoresis