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An Improved Technique for Preparing Solvent Cast Films from Acrylic Fibers for the Recording of Infrared Spectra

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ABSTRACT: Synthetic fibers which are microscopically indistinguishable could differ in chemical composition. The use of infrared spectroscopy to determine generic subtypes is standard forensic science practice. An improved technique is described for preparing good solvent cast films suitable for producing acceptable spectra from samples as small as 1 μg.

KEYWORDS: criminalistics, synthetic fibers, solvent cast film, spectroscopic analysis

The task of the forensic chemist to collect, characterize, compare, and identify fibers is often a long and tedious process. In transfer cases involving clothing, no methods are available that can show positively that found fibers originated from a particular garment. In order to enhance the suitability of fiber evidence in court as being suggestive of contact between garments, it is essential that a thorough examination be made. The characteristics of synthetic fibers that are generally compared in a forensic science laboratory include color, surface appearance, cross-sectional shape and diameter, fluorescence, and type. Microscopy provides the foundation for the major portion of fiber analyses and in this regard sample size rarely poses any obstacle. Dye comparisons can now be performed nondestructively and more objectively with a microspectrophotometer [*1*] even though metamerism [*2*] must still be considered. Main generic groups of synthetic fibers usually can be determined with polarized light [*3*]. Control and questioned fibers, however, that are microscopically indistinguishable, whose dye components compare in two or more thin-layer chromatography solvent systems [*4*] and also match photometrically, could still differ in chemical composition. In certain cases hot stage methods can provide additional information [*5,6*], however, they clearly are not applicable to some synthetics that decompose at elevated temperatures [*7*]. Birefringence measurements using the Michel-Lévy chart are not accurate enough to distinguish between generic subtypes; however, refractive index determinations to measure birefringence may provide valuable information leading to further discrimination [*6*]. Until

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a suitable optical method is presented for this purpose instrumental methods less sensitive to sample size remain necessary.

Acrylic fibers are one of the more commonly encountered synthetics in the forensic science laboratory. Smalldon has determined seven acrylic types by infrared spectroscopy [*8*]. With the exception of the lead foil technique [*9*] which requires a very high degree of skill, none of the sample preparation techniques in the literature is suitable for fibers with a mass of less than several micrograms [*10–12*]. The solvent cast method of Cook and Paterson [*9*] claims effectiveness on samples of not less than 3 μg. In our experience a solvent cast film prepared by their method usually forms very poorly and contains large voids. Often only a ring is formed or the diameter of the film becomes larger than that of the micropellet. This requires that the film be cut into fragments and fitted into the pellet disc. When the pellet is pressed, portions of the film are frequently forced to the edges of the hole and in some cases even beyond the salt "window" thereby not falling within the beam path. It was felt that if better quality films could be produced, greater sensitivity could be achieved.

Method

The fiber is placed into the neck of a melting point tube with tweezers and then pushed to the bottom using a fine wire or other suitable plunger. Dimethylformamide [*13*] is added using a finely drawn capillary tube. Care is taken to use the minimum amount of solvent, that is, just enough to cover the fiber. This may require cutting the fiber into several smaller fragments, especially if it does not compress well in the bottom of the tube. The tube is then heat sealed and placed into an oven at 100°C until the fiber is completely dissolved (approximately 10 to 30 min). Both the melting point tubes and the drawn capillaries are conveniently prepared from 50-μL disposable pipets (Corning 7099S). Rubber tubing and a mouthpiece are included. The tube is scored and broken just above the liquid and carefully sucked into a clean drawn capillary. A clean stainless steel anvil from a microdie assembly (Perkin-Elmer 061040) is placed for 0.5 min on a Thermolyne type 2300/series 242 hot stage at 125°C. It is then removed using forceps and the polished tip is focused under a stereoscope at a comfortable (approximately ×16 or lower) magnification. A microdroplet is gently blown by mouth onto the tip of the capillary (Fig. 1) and transferred to the anvil. With minimal practice small enough droplets can be formed so that they bead on the anvil with no overflow. The solvent dries quickly and successive droplets are added. Temperature is monitored throughout the process. If the anvil is too hot, splattering will occur causing some sample loss. If drying occurs too slowly the anvil is reheated accordingly.

Since the cast adheres firmly to the metal surface there is no fear of loss when the anvil is transferred back to the hot plate. After the entire sample has been added the cast is gently peeled off with a No. 11 scalpel blade and boiled in water for 5 to 10 min to remove any residual solvent. It is convenient to use a half filled 10-mL beaker for this to facilitate the subsequent recovery of the film.

A potassium bromide micropellet is prepared while the film is being boiled and placed into a desiccator. The microdie is adjusted with one disc in place so that the piston extends about halfway into the hole forming a well. The entire assembly is then placed under a stereoscope for the remaining steps. After the film has been removed and dried it is placed into the well and centered. The second disc containing the micropellet is now placed into the dye as was originally positioned. While focusing through the clear pellet onto the film, the height adjusting ring on the die is rotated slowly in a clockwise direction. Some slight repositioning of the upper disc may be necessary for the piston to move smoothly through the hole towards the pellet. The film should be brought up to the pellet but not touch it to prevent "popping" the pellet from its holder. The polished cover disc is now replaced, the screwcap attached and the adjusting ring then rotated completely clockwise to complete the "fusion" of the film onto the pellet.

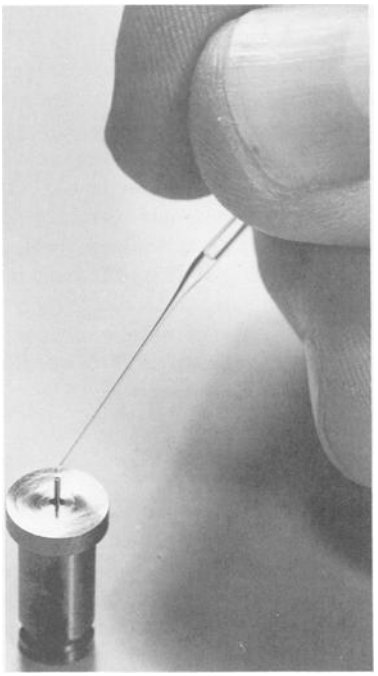

FIG. 1—*A microdroplet is blown onto the tip of the capillary tube and transferred to the heated anvil.*

Results and Discussion

Figure 2 represents spectra obtained on a Perkin-Elmer Model 283B infrared spectrophotometer using a 4:1 refracting beam condenser recorded at ×3 ordinate expansion. Solvent casts were formed on 0.5-mm diameter anvils using fibers 2 and 3 mm (1 and 1.5 μg) in length and pressed onto pellets of 1.0-mm diameter. Weights were estimated using the approximation provided by Cook and Paterson [9], that is, 2-mm length, 20-μm diameter = 1 μg. The fibers examined which were selected from actual casework had a round cross-sectional shape measuring 21 μm. Under crossed polars they displayed low birefringence and negative elongation which is characteristic of acrylic fibers and this was clearly confirmed by infrared spectroscopy.

Conditions for producing good spectra with this technique include the following:

(1) very thin, high quality pellets,
(2) films of uniform cross-sectional thickness, and
(3) exact centering of films on pellets.

Quality pellets are easily prepared with anvils maintained in good condition and following standard procedure. Production of good film casts may at first be challenging, however, with several repetitions, the skill is quickly acquired. Poor casts are frequently formed but can be detected if they are viewed during the drying process. Apparently, the solvent sometimes diffuses outwards carrying most of the polymer to the edges and thereby forming a donut-shaped film. It is advisable when first trying this technique to use dyed fibers since if a poor cast is formed it is more easily noticed. With colorless samples the anvil can be slightly tilted to allow for viewing the film at an angle more suitable for observing any surface unevenness. This condition usually can be corrected by reheating the cast and adding pure solvent in the normal manner, drop by drop, until the film is observed to be more uniformly formed (Figs. 3 and 4).

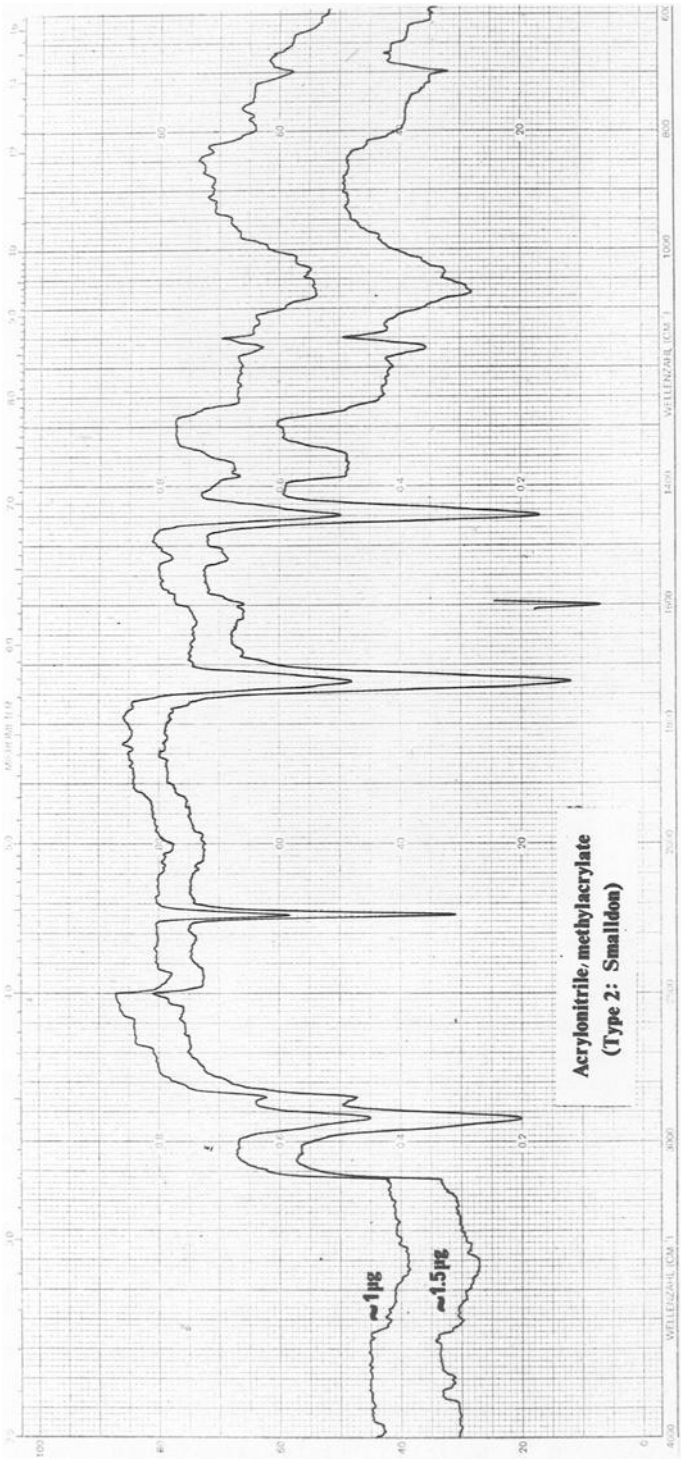

FIG. 2—*Spectra obtained from solvent cast films from acrylic fibers at* ×3 *ordinate expansion.*

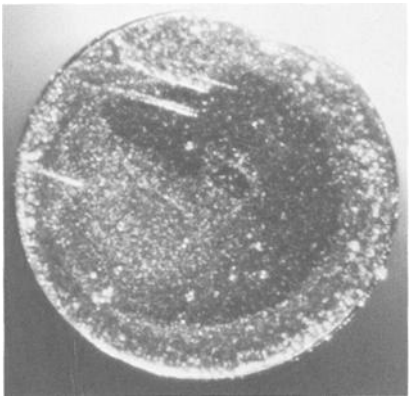

FIG. 3—*A cast that has formed poorly caused by diffusion of the polymer towards the edge.*

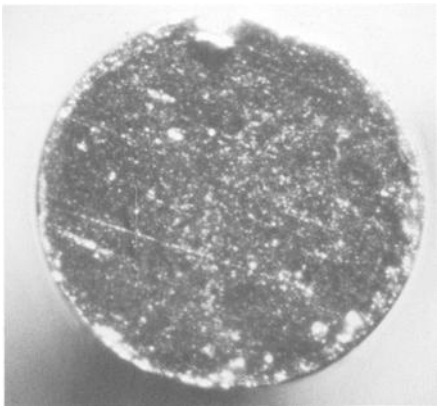

FIG. 4—*Same cast as in Fig. 3 after heating and addition of several droplets of pure solvent.*

Of course, excessively high heating should be avoided to prevent any decomposition. If the film initially does not transfer to the potassium bromide pellet it can be repressed using only slight pressure and thereby avoid any film damage which may affect spectral quality. Although this technique has been attempted with acrylics only, it is reasonable to expect comparable results with other synthetic fibers. With practice good samples can be prepared in less than 45 min.

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