TECHNICAL NOTE

Graeme W. White,[1] *B.App.Sc.*

A Simple High-Pressure Anvil and Template Device for the Production of Infrared Spectra from Microfiber Samples

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ABSTRACT: Infrared spectra were obtained from single microfibers using a specifically designed anvil and template to preflatten the fiber perpendicular to the infrared (IR) path. A series of IR spectra of fibers from the Collaborative Testing Service (CTS) reference Collection of Synthetic Fibers has been produced to demonstrate the technique and provide a basis for others in possession of CTS fibers to compare techniques.

KEYWORDS: forensic science, infrared spectra, fibers, synthetic fibers, anvil, microinfrared spectra, acrylic, modacrylic, nylon, polyester, polyethylene, rayon, acetate, polypropylene

Forensic fiber comparison usually requires that a series of tests be carried out on one fiber, that is, that the same individual fiber be compared microscopically, chemically, and spectroscopically. Therefore, there is a need to be able to recover the fiber for further tests, notably for dye comparison, by microspectrophotometry and thin-layer chromatography, after having obtained an infrared match.

A number of systems have been developed to produce infrared spectra from small single fibers. Systems such as solvent films [*1–3*], anvil cells [*4–7*], potassium bromide (KBr) micropellets [*8,9*], and microslits, which are commonly used in conjunction with beam condensers. Each has its merits; however, with the exception of the microdiamond cell, all are labor-intensive and require considerable dexterity in the manipulation of the fibers. Diamond cells have a large infrared (IR) opaque region that can present problems with acrylic fibers. Solvent films have the problem of being a destructive technique that inhibits further testing.

Producing a consistent infrared spectrum of a single fiber can present a number of problems to the forensic scientist. The circular profile of a fiber can induce diffraction and stray light effects [*7,10–12*], and the diameter of a highly colored fiber can affect the optical density of the fiber. The anvil and template described have been designed to allow up to ten fiber mounts, which are flattened perpendicular to the IR beam. The

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[1]Forensic chemist, Forensic Chemistry Section, Government Chemical Laboratory, Archerfield, Queensland, Australia.

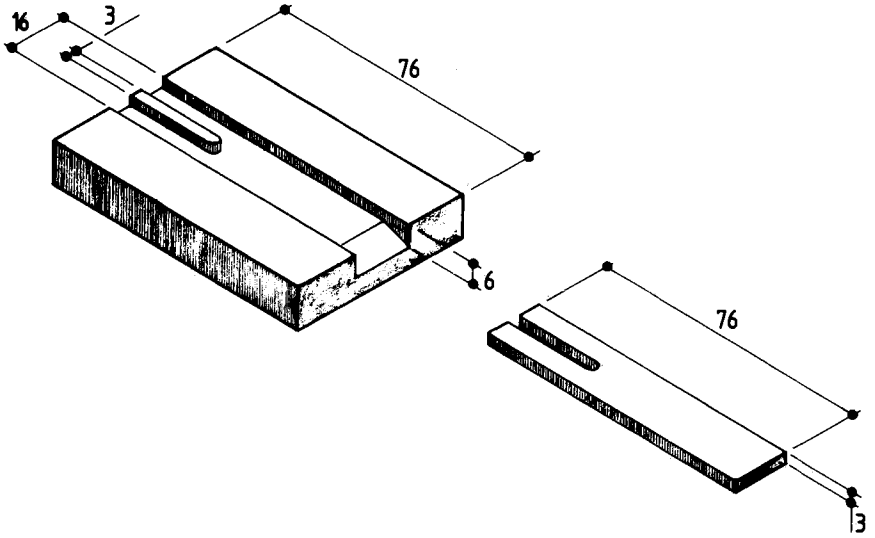

FIG. 1—*The anvil and template. The dimensions in millimetres.*

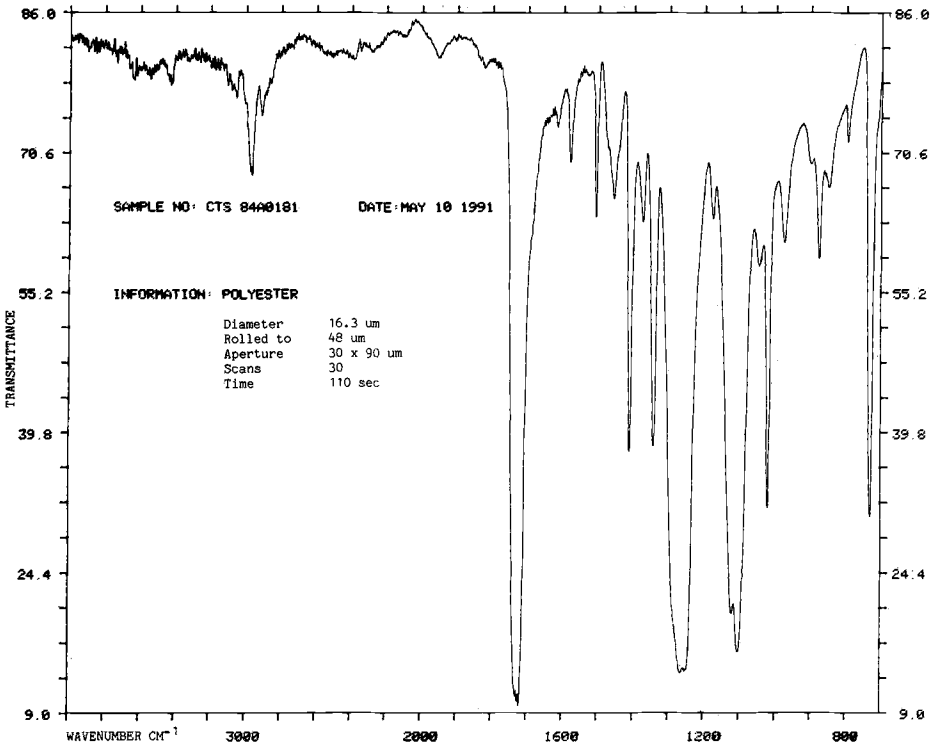

FIG. 2—*IR spectrum of a polyester single fiber.*

combination of the flattened profile and consistent orientation of the fiber to the IR beam minimizes diffraction and crystal alignment effects. The device also facilitates easy recovery of the fibers for further comparison.

Materials and Equipment

The template was designed to fit the stage of a Spectra-Tech Model 0036-033 microscope, attached to a Perkin-Elmer Series 1750 Fourier transform infrared spectrometer equipped with a Judson MCT detector.

Reference fibers were obtained from the Collaborative Testing Service (CTS) Reference Collection of Synthetic Fibers.

Description

The device consists of a plate 76 mm long, 16 mm wide, and 3 mm thick. A slot 3 mm wide and 25 mm long was cut into the center of the plate from one end. A mating anvil was machined into a base plate to match the slot in the plate (Fig. 1). A small chamfer was machined into the leading edge of the slot to guide a scalpel tip when incising the tape placed across the plate and anvil.

Procedure and Discussion

The plate is placed onto the anvil and base plate. A strip of double-sided tape is laid along the plate so that it covers both sides of the slot in the plate and the anvil. A scalpel

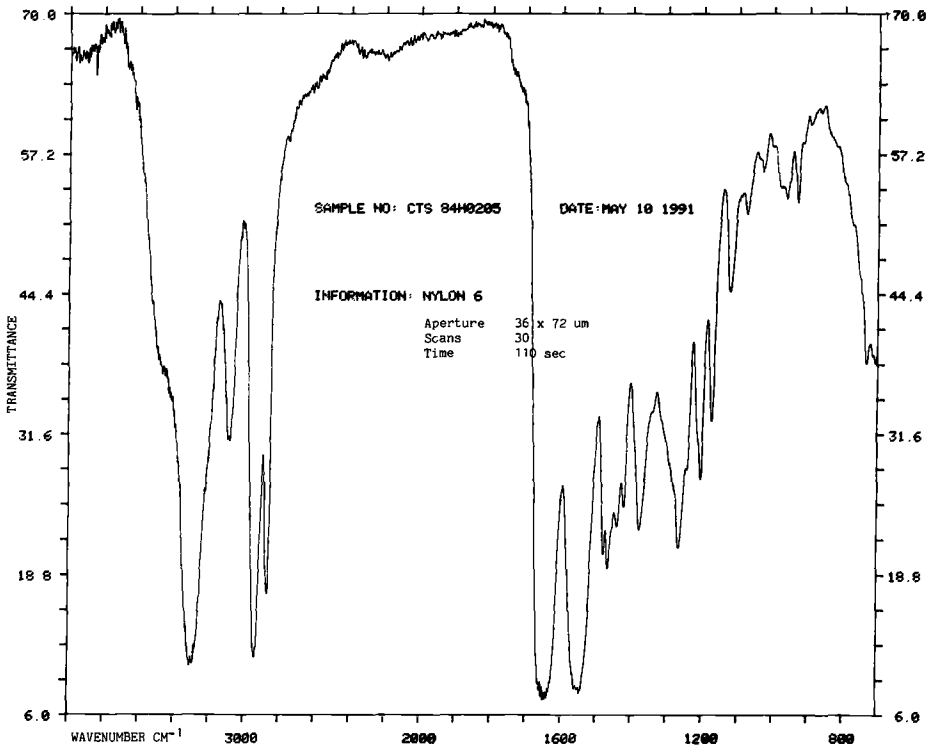

FIG. 3—*IR spectrum of a Nylon 6 single fiber.*

point is used to cut around the anvil, and that portion of the tape covering the anvil is removed.

Using a stereomicroscope, 5- to 8-mm-long sections of fiber are mounted across the slot. If the length of fiber is too short, a small piece of double-sided tape is placed from one side of the slot to the other as a bridge. The fiber is attached to the underside of the bridge so that it protrudes into the center of the slot.

After the fibers have been allowed to relax in the adhesive, they are then rolled, using a small ball-bearing race mounted in a yolk formed into one end of a pencil-sized handle. Only a light touch with an oscillating motion is required to flatten fibers up to 20 μm in diameter. Larger filaments are rolled vigorously on the anvil backing plate prior to mounting. The roller was first observed by the author in use at the South Australian Forensic Science Centre. There, acrylic fibers were rolled prior to mounting in a low-pressure diamond cell.

The plate is removed from the anvil and mounted on the microscope stage. The first fiber is located in the optical path, and the aperture is set to about 70% of the width of the flattened section being examined. With this microscope, the aperture is remote from the fiber and it is important to focus on the center of the fiber to minimize stray light effects [*10*].

The plate is then simply racked through, picking up each fiber in turn. To ensure that fibers are not missed or mixed, an odd-colored fiber is placed between selected fibers and recorded accordingly. Only fibers from one source are mounted at the same time.

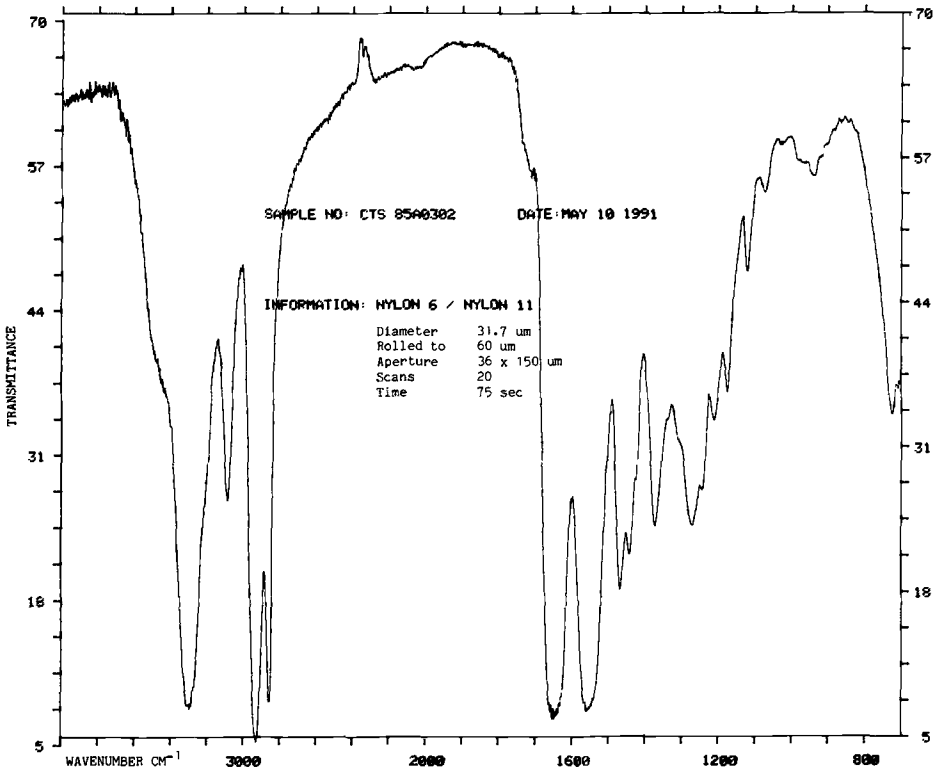

FIG. 4—*IR spectrum of a Nylon 6/Nylon 11 single fiber.*

Using this technique, infrared spectra were prepared from a selection of CTS reference fibers (Figs. 2 through 11).

Conclusions

Case work with the template and anvil has demonstrated its usefulness in routine single-fiber IR analysis. It has proven to be a rapid, reliable, and cost-effective technique, providing the following benefits for IR analysis of single fibers.

(1) rapid preparation of up to ten fiber mounts,
(2) maintenance of the security of the fiber,
(3) ease of recovery for further tests,
(4) presentation of a flattened fiber in a consistent orientation to the IR beam, and
(5) a reduction in diffraction effects and improved optical transparency.

Acknowledgments

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The skilful work done by Ron Arbery in the machining of the template and anvil is also acknowledged. Thanks are also due to Gary Golding for his invaluable assistance in the manipulation of the IR spectrometer and in the editing of this report.

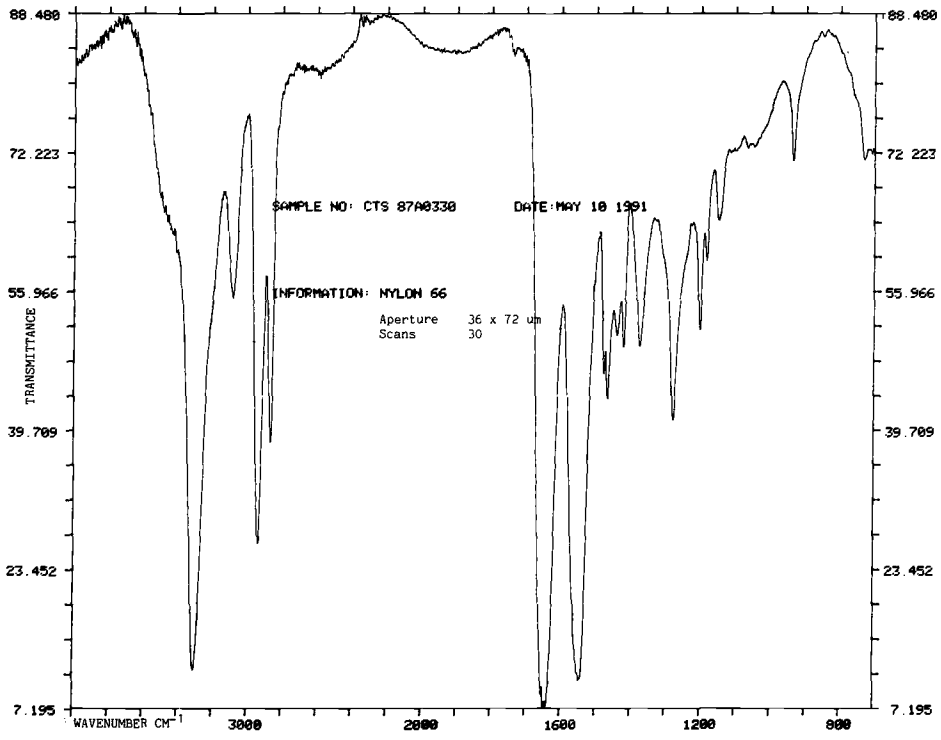

FIG. 5—*IR spectrum of a Nylon 66 single fiber.*

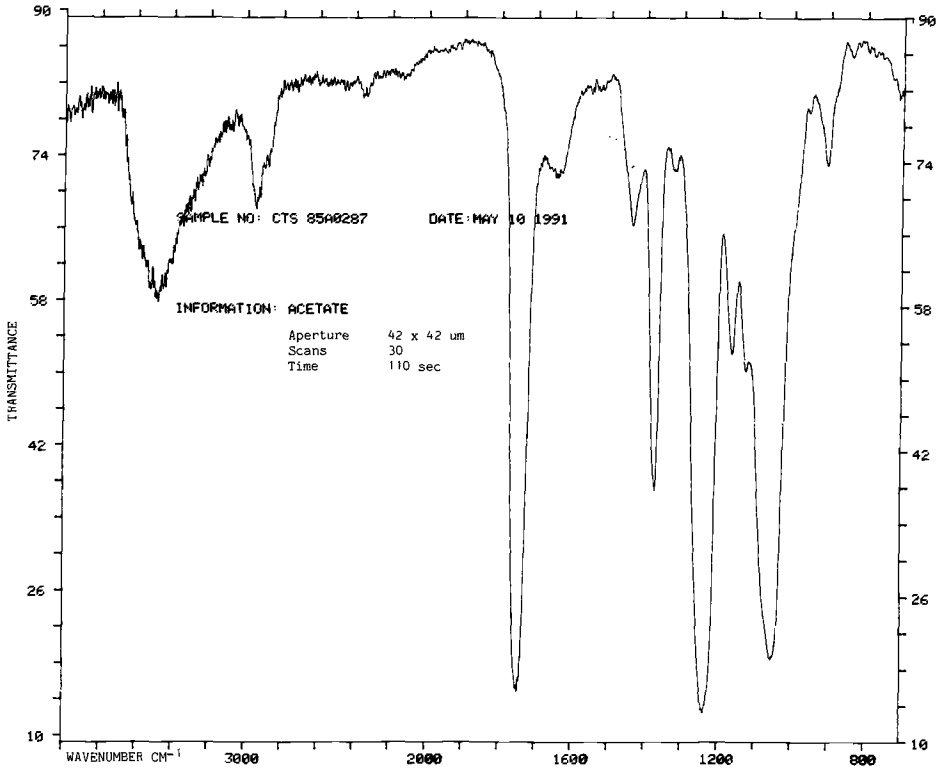

FIG. 6—*IR spectrum of an acetate single fiber.*

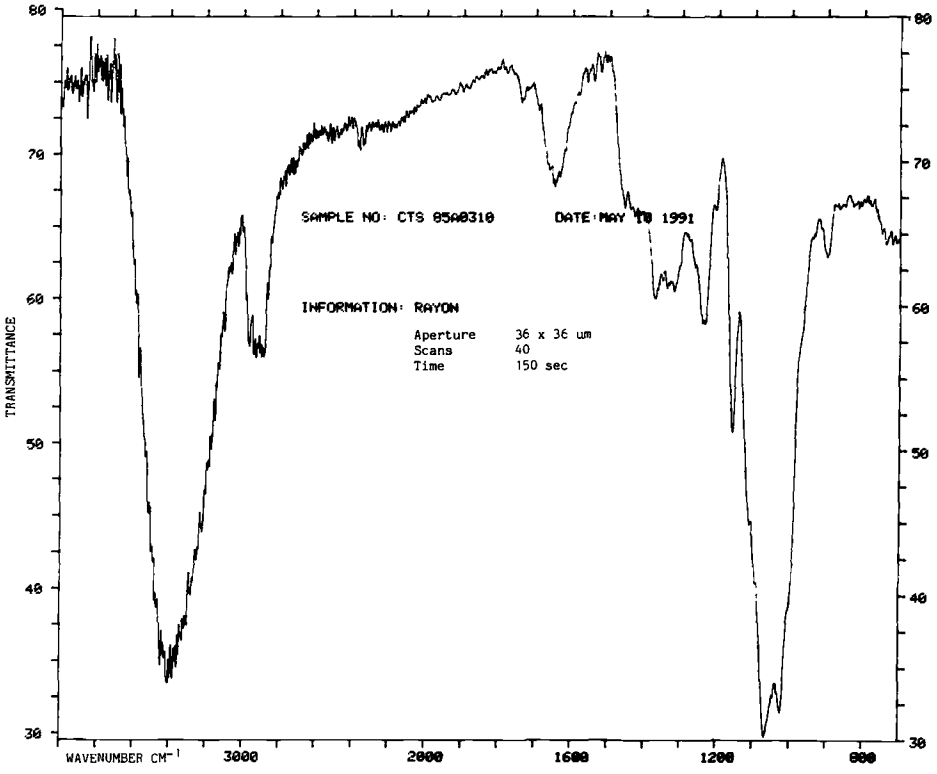

FIG. 7—*IR spectrum of a rayon single fiber.*

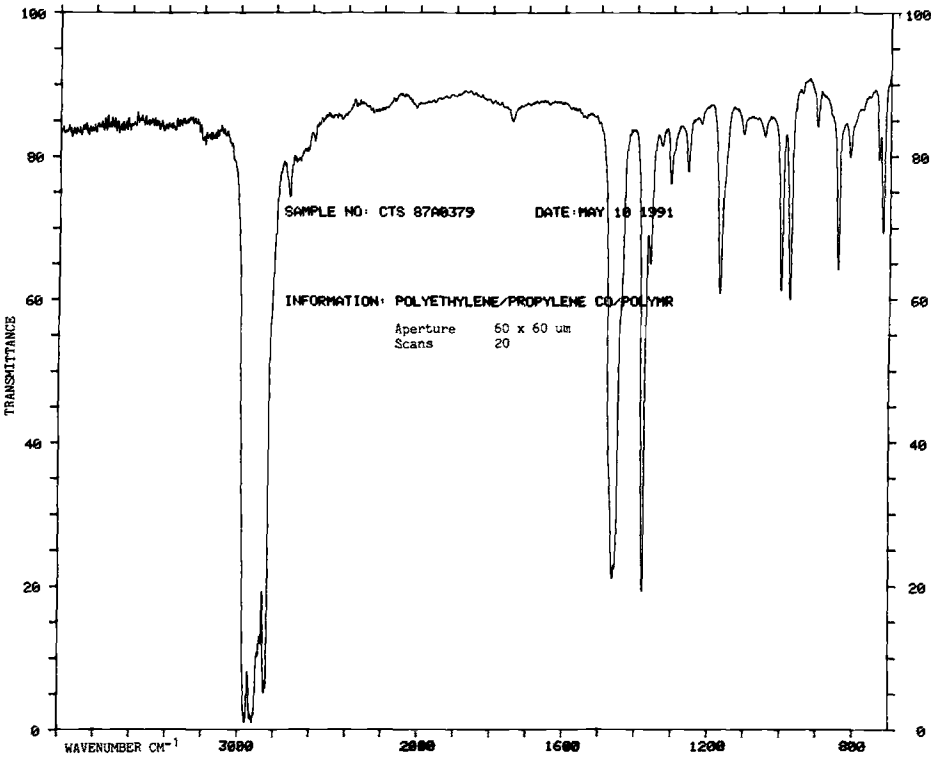

FIG. 8—*IR spectrum of a polyethylene polypropylene copolymer single fiber.*

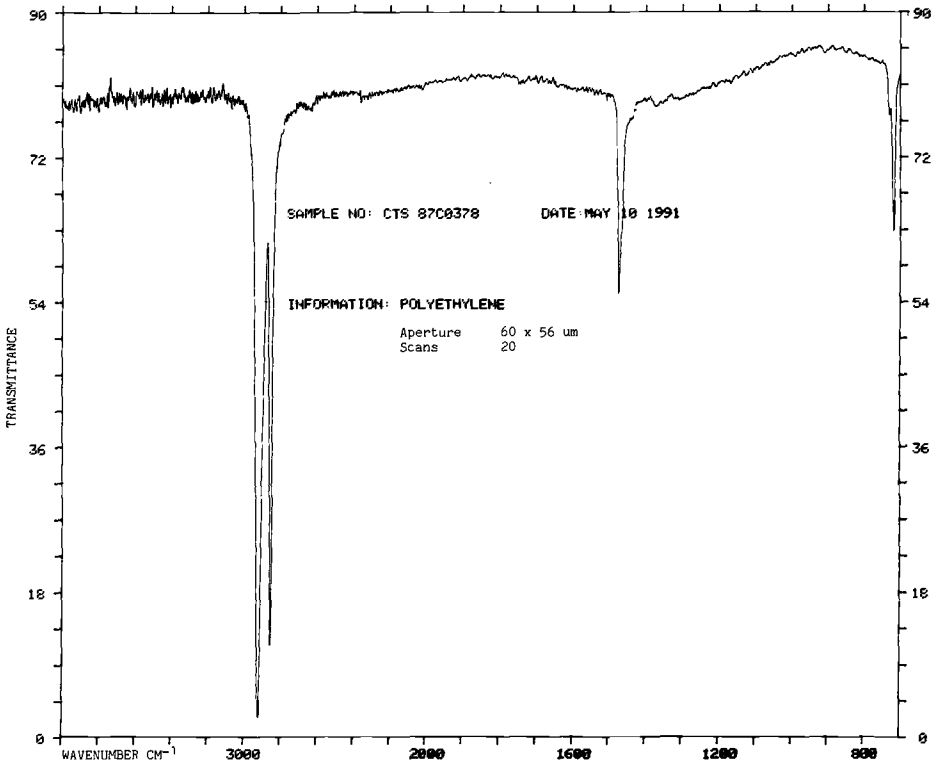

FIG. 9—*IR spectrum of a polyethylene single fiber.*

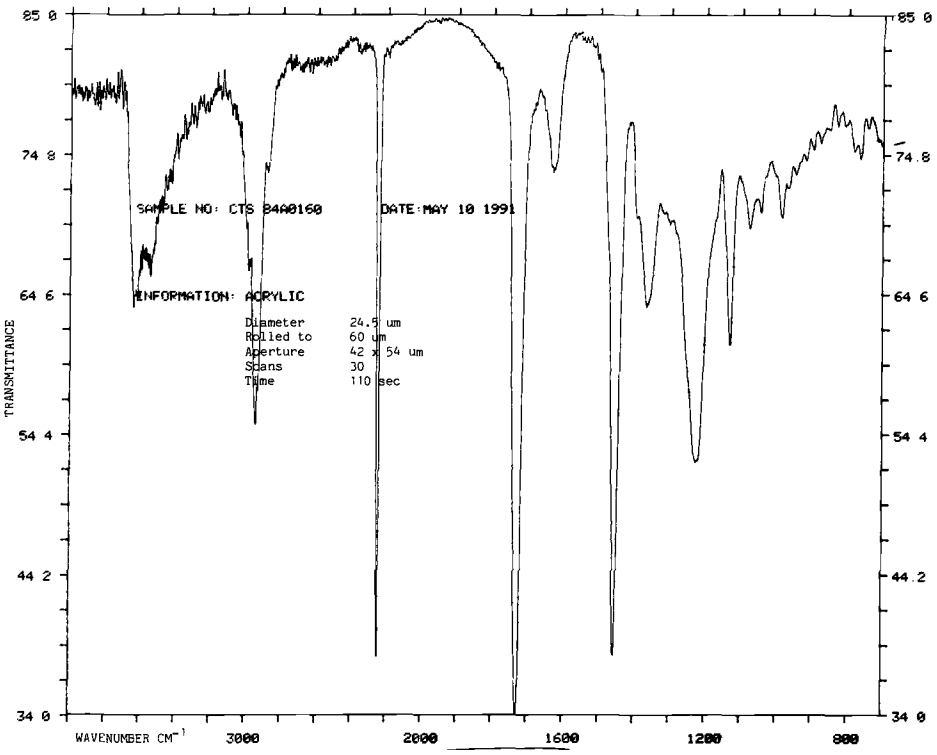

FIG. 10—*IR spectrum of an acrylic single fiber.*

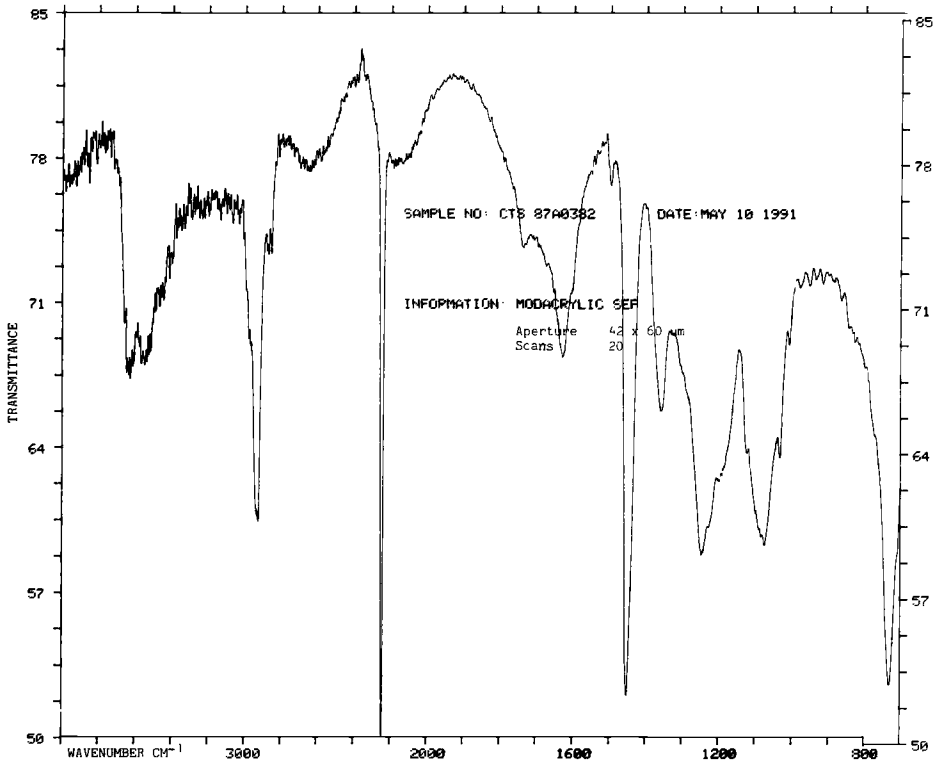

FIG. 11—*IR spectrum of a modacrylic single fiber.*

References

[1] Rouen, R. A. and Reeve, V. C., "A Comparison and Evaluation of Techniques for Identification of Synthetic Fibers," *Journal of Forensic Sciences*, Vol. 15, No. 3, July 1970, pp. 410–432.

[2] Grieve, M. C. and Kearns, J. S., "Preparing Samples for the Recording of Spectra from Synthetic Fibers," *Journal of Forensic Sciences*, Vol. 21, No. 2, 1976, pp. 307–314.

[3] Grieve, M. C., "Fibres and Their Examination in Forensic Science," *Forensic Science Progress*, R. L. Williams and A. Maehly, Eds., Springer-Verlag, Berlin, 1990, p. 77.

[4] Cournoyer, R., Shearer, J. C., and Anderson, D. H., "Fourier Transform Infrared Analysis Below the One-Nanogram Level," *Analytical Chemistry*, Vol. 49, No. 14, Dec. 1977, pp. 2275–2277.

[5] Curry, C. J., Whitehouse, M. J., and Chalmers, J. M., "Ultramicrosampling in Infrared Spectroscopy Using Small Apertures," *Applied Spectroscopy*, Vol. 39, No. 1, 1985, pp. 174–180.

[6] Bartick, E. G., "Applications of a New High Pressure Anvil Cell for IR Spectroscopy," *SPIE*, Vol. 553, 1985.

[7] Bartick, E. G., "Considerations for Fiber Sampling with Infrared Microspectroscopy," *The Design, Sample Handling, and Applications of Infrared Microscopes, ASTM STP 949*, P. B. Roush, Ed., American Society for Testing and Materials, Philadelphia, PA, 1987, pp. 64–73.

[8] Fox, R. H. and Schuetzman, H. I., "The Infrared Identification of Microscopic Samples of Man-Made Fibers," *Journal of Forensic Sciences*, Vol. 13, No. 3, July 1968, pp. 397–406.

[9] Anderson, D. H. and Wilson, T. E., "Novel Approach to Micro Infrared Sample Preparation," *Analytical Chemistry*, Vol. 47, No. 14, Dec. 1975, pp. 2482–2483.

[10] Chase, D. B., "Infrared Microscopy: A Single Fiber Technique," *The Design, Sample Handling, and Applications of Infrared Microscopes, ASTM STP 949*, P. B. Roush, Ed., American Society for Testing and Materials, Philadelphia, PA, 1987, pp. 4–11.

[*11*] Messerschmidt, R. D., "Photometric Considerations in the Design and Use of Infrared Microscope Accessories," *The Design, Sample Handling, and Applications of Infrared Microscopes, ASTM STP 949*, P. B. Roush, Ed, American Society for Testing and Materials, Philadelphia, PA, 1987, pp. 12–26.

[*12*] Griffiths, P. R. and de Haseth, J. A., *Fourier Transform Infrared Spectrometry*, Wiley-Interscience, New York, 1986, pp. 429–430.

Address requests for reprints or additional information to
Graeme W. White
Forensic Chemistry Section
Government Chemical Laboratory
P.O. Box 594
Archerfield 4108, Queensland, Australia