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A New Approach to the Microscopical Examination and Comparison of Synthetic Fibers Encountered in Forensic Science Cases

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ABSTRACT: This paper describes a refined approach to the problems of identification and comparison of synthetic fibers encountered in forensic science case work. The necessary apparatus consists of a polarizing microscope equipped with a Mettler hot stage. Observations concerning the variation of a fiber's birefringence with temperature have been found to be very useful, both for determining the generic type of an unknown fiber and for detecting subtle differences among fibers of the same generic type from different sources. Data, including semilogarithmic plots of birefringence as a function of temperature, are given for nine different generic types of synthetic fibers.

KEY WORDS: criminalistics, synthetic fibers, birefringence

The evidential value of textile fibers encountered in forensic science investigations has long been recognized. Kirk and other authors of note have pointed out their value [*1-6*]. Several studies have been made to evaluate some of the methods used in the forensic science examination and comparison of textile fibers [*7-10*]. These articles have demonstrated the need for further evaluation of current methods employed in forensic science laboratories and the need for the development of more specific and more sensitive methods of identification and comparison. A recent paper that reviews the procedures used in the examination and comparison of hairs and fibers in forensic science laboratories calls for more study and research on these subjects [*11*].

Many of the procedures used in the forensic science identification and comparison of textile fibers are concerned primarily with the microscopical examination of physical morphology and optical properties of the fibers. Other methods are based on the instrumental analysis of textile fibers. In most cases these procedures, if used by an experienced criminalist, will afford positive identification of the generic type of textile fiber and will enable the criminalist to determine whether or not the questioned fibers "could have" originated from the source of the known fibers. This is often not enough. Methods that are capable of detecting subtle differences between questioned and known specimens are needed so

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that a stronger opinion regarding commonality of origin can be rendered. This need is most apparent when fibers from different sources that are of the same generic type and that exhibit very similar physical characteristics and chemical properties are to be compared. Some otherwise useful methods are inadequate when the sample sizes are quite small.

Several studies involving both instrumental methods and microscopical methods have been made in which the researchers have developed more sensitive procedures for the identification and comparison of textile fibers [*12-16*]. Although these studies have given the criminalist several new and useful techniques for the examination of textile fibers in forensic science case work, more research is needed.

If a synthetic fiber behaved as a typical crystalline substance one would expect a sharp transition to take place between the ordered solid state and the unordered liquid state. This first-order transition is known as the melting point. However, unlike wholly crystalline materials, synthetic fibers do not have sharply defined melting points. It is well known that the greater the degree of crystallinity (molecular order) of a given type of fiber, the greater is its birefringence. It is further known that as the degree of orientation of a fiber's molecules changes, so does its birefringence. A fiber may soften over a range of temperature before it melts. It is apparent that one of the best ways to monitor the loss of order in a fiber during heating is to observe changes that take place in the fiber's birefringence. Grabar and Haessly [*17*] have used these changes in birefringence with temperature as an aid in the identification of various synthetic fibers.

Because pure crystalline materials have sharp melting points, a study of their birefringence while they are being heated would yield no more information than could be ascertained from separate birefringence and melting point measurements. However, in the case of fibers, changes in birefringence may occur at temperatures below the melting point of the fiber, and such changes among similar fibers may be observed during heating. In the present study these premises are evaluated and their utility with respect to the forensic science comparison of synthetic fibers is tested.

The approach reported here involves the detailed observation of the changes in a synthetic fiber's birefringence and thickness during the time it is heated at a uniform rate. The purpose of this study is to present a new approach to the forensic science identification and comparison of synthetic fibers. The method was designed to handle microscopic samples of synthetic fibers and to detect subtle differences between questioned and known fiber samples of the same generic type.

Methods and Materials

Apparatus

A Leitz AM research polarizing microscope equipped with a modified Ehrenreich Photo-Optical Industries fiber optic light unit was used to observe the changes in each specimen. The specimens were heated on a Mettler hot stage, Model FP52, which was controlled by a Mettler control unit, Model FP5.

Hot Stage Calibration

The accuracy of the Mettler hot stage and control unit was checked daily with the melting point standards. It was accurate within $\pm 0.5°C$ for the 2°C/min program. This degree of accuracy was more than adequate for the purposes of this study. At the slowest program rate, 0.2°C/min, the hot stage is reported to be accurate to better than $\pm 0.1°C$.

Sampling Distribution

Synthetic fibers of nine generic types were obtained from 31 sources, encompassing 16 manufacturers and 15 independent dealers. The total of 85 samples was acquired at various times during the period from 1976 through 1978. The specimens were selected at random from the available samples. These samples of synthetic fibers are listed and classified in Table 1.

Sample Preparation

A specimen of each synthetic fiber to be examined was cut into 5-mm lengths. Beyond this treatment every precaution was exercised to avoid altering the fibers in any manner. After being cut to size each specimen was mounted in silicone liquid near one end of a microscope slide and covered with a cover glass. Excess silicone liquid was removed by blotting with filter paper. The silicone liquid proved to be a satisfactory mounting medium for several reasons. First, it was inert with respect to fibers. It did not dissolve them or cause them to swell. Second, the silicone liquid helped to improve the fiber's image, which made observing and interpreting the interference colors exhibited by the specimen much easier. Finally, the silicone liquid improved the thermal contact between the specimen and the hot stage, thereby lessening the magnitude of any temperature lag. In the case of fiber comparisons, both the questioned and the known specimens were mounted on the same slide parallel to each other approximately 1 mm apart so they could be observed simultaneously.

TABLE 1—*A tabulation of the synthetic fibers collected for use in this study, their source and physical characteristics.*

Fiber	Number of Samples	Producer-Dealer		Color		Style		Luster	
				Dyed	Undyed	Staple	Filament	Bright	Dulled
Acele®	2	2	...	...	2	...	2	2	...
Celanese®	3	...	3	3	...	...	3	3	...
Chromspun®	11	11	...	11	...	...	11	11	...
Estron®	2	2	...	...	2	...	2	1	1
Acrilan 16®	2	...	2	1	1	...	2	...	2
Creslan®	3	1	2	2	1	3	...	...	3
Orlon®	4	2	2	2	2	4	...	4	...
Zefran II®	2	...	2	2	...	2	...	2	...
Dynel®	2	...	2	2	...	2	...	2	...
Verel®	8	8	...	1	7	8	...	3	5
Nylon 6	1	1	...	1	...	...	1	1	...
Antron®	3	2	1	1	2	...	3	3	...
Nylon 66	2	2	...	...	2	...	2	2	...
Herculon®	12	12	...	10	2	6	6	12	...
Marvess®	11	11	...	10	1	...	11	11	...
Dacron®	6	2	4	4	2	2	4	...	6
Fortrel®	2	...	2	2	...	...	2	...	2
Kodel 211®	2	2	...	1	1	2	...	...	2
Vycron®	1	...	1	1	...	...	1	...	1
Lycra®	2	2	...	...	2	...	2	...	...
Arnel®	2	1	1	1	1	...	2	2	...
Saran®	2	2	...	...	2	...	2	2	...
Totals	85	63	22	55	30	29	56	61	22

Results

Sample Measurement

The mounted fibers were observed with the polarizing microscope. The specimen's thickness was first measured with a previously calibrated ocular micrometer. Next, the fiber's sign of elongation was determined. Finally, the amount of retardation was estimated and the fiber's birefringence was calculated by using the Michael Lévy interference color chart [*18*] and the equation that expresses the interrelationship of thickness, birefringence, and retardation.[4] Table 2 lists the synthetic fibers used in this study with their estimated thicknesses and birefringence measurements.

Heating of Samples

The prepared man-made fiber specimens were placed on the Mettler hot stage set to a constant temperature of 25°C, which was maintained by the Mettler control unit. The hot stage was then placed on the stage of the polarizing microscope, and each specimen of synthetic fiber was observed between crossed polars. The specimens were oriented at a 45-deg angle (diagonally) to the crosshairs, the position of maximum brightness. The heating rate of the Mettler temperature control unit was then preselected for the 2°C/min

TABLE 2—*A list of synthetic fibers employed in this study with their estimated thickness and birefringence measurements at ambient temperature.*

Brand Name of Synthetic Fiber	Generic Type	Thickness[a]	Birefringence[b]
Acele	acetate	25	+0.005
Celanese	acetate	25	+0.0055
Chromspun	acetate	25	+0.0055
Estron	acetate	25	+0.0055
Acrilan 16	acrylic	20	−0.005
Creslan	acrylic	20	−0.005
Orlon	acrylic	30	−0.002
Zefran II	acrylic	20	−0.0025
Dynel	modacrylic	40–50[c]	+0.002
Verel	modacrylic	40–50[c]	−0.002
Firestone	nylon 6	25	+0.060
Antron	nylon 66	15	+0.060
Du Pont	nylon 66	15	+0.060
Herculon	olefin	30	+0.032
Marvess	olefin	30	+0.032
Dacron	polyester	12–15[c]	+0.175
Fortrel	polyester	15	+0.162
Kodel 211	polyester	25	+0.102
Vycron	polyester	15	+0.147
Lycra	polyurethane	200–300[c]	+0.0003
Arnel	triacetate	20–25[c]	+0.0002
Saran	vinylidene chloride	170–180[c]	−0.006

[a] All thickness measurements are in micrometres.

[b] All birefringence values are estimated from retardation colors, not calculated from exact measurements of refractive indices. Therefore, these values may differ slightly from the values seen in the literature.

[c] A range of thickness measurements.

[4] Retardation in nm = 1000 × thickness in μm × birefringence. Course Manual, *Polarized Light Microscopy,* McCrone Research Institute, Chicago, 1976, frontispiece.

program for all the fibers except Dacron®. In the case of Dacron, the heating rate was set at the 1°C/min program because at the more rapid rate the changes in Dacron's birefringence occurred too rapidly to be observed and recorded accurately. The temperature of the hot stage was then increased at the preselected programmed rate by the control unit. All changes in the fibers' retardation colors and thicknesses were recorded at the temperature at which they were observed to occur. The isotropic point for each fiber was noted at the temperature at which the fiber no longer exhibited any retardation colors. The changes in the fibers' birefringence were then calculated from the retardation and thickness data as previously described.

This method was unsuitable for the examination of viscose rayon. Rayon, which is a regenerated cellulosic fiber, was found to char at temperatures approaching 300°C. Aramid fibers such as Nomex® and Kevlar® have such high melting points that no appreciable change in their birefringences or thicknesses was observed within the heating range of 25 to 300°C.

Discussion

Graphical Representation

There were strong resemblances among the various samples of man-made fibers having the same generic classification and significant differences among the fibers of different generic classification. Although these data could be used to identify, compare, and differentiate among the synthetic fibers included in this study, some alternative way of presenting the data needed to be found. The changes in birefringence with temperature suggested the possibility of graphing changes in birefringence as a function of temperature.

Four-cycle semilogarithmic graph paper was used because of the extreme range in birefringence exhibited by the various samples of man-made fibers (from 0.0002 to 0.175). The abscissa was marked off in temperatures from 0 to 300°C and the ordinate was marked off in birefringence ($N_1 - N_2$) from 0.0001 to 1.0. A birefringence of less than 0.0001 is considered negligible for the purposes of this study. The synthetic fibers' birefringences were plotted as a function of temperature. The points were then connected, starting with the initial birefringence value for each specimen and ending at the temperature at which the birefringence for each specimen was no longer detectable. Both retardation and thickness were observed during heating. Where it was noted that the thickness had actually changed, the new thickness was used with the retardation at that temperature to calculate the birefringence so that no error would be introduced from this source.

A graph, as described above, was drawn up for each of the synthetic fibers included in this study; these graphs are shown in Figs. 1 to 8. Each graph contains plots of chemically related synthetic fibers presented together to show the similarities or differences in their curves.

The graphs proved to be extremely useful in interpreting the data. The diagrams that were obtained made the identification of generic class and the forensic science comparison of the synthetic fibers both simple and direct. The graphs allowed one to distinguish easily among synthetic fibers of different generic class and to make forensic science comparisons between man-made fibers within the same generic classification to differentiate similar specimens of synthetic fibers originating from different sources.

Reproducibility

A statistical evaluation of the reproducibility was done with the five fiber types shown in Table 3. Each specimen was treated in the same manner as previously described, and the changes in each specimen's thickness and retardation were noted and recorded along with

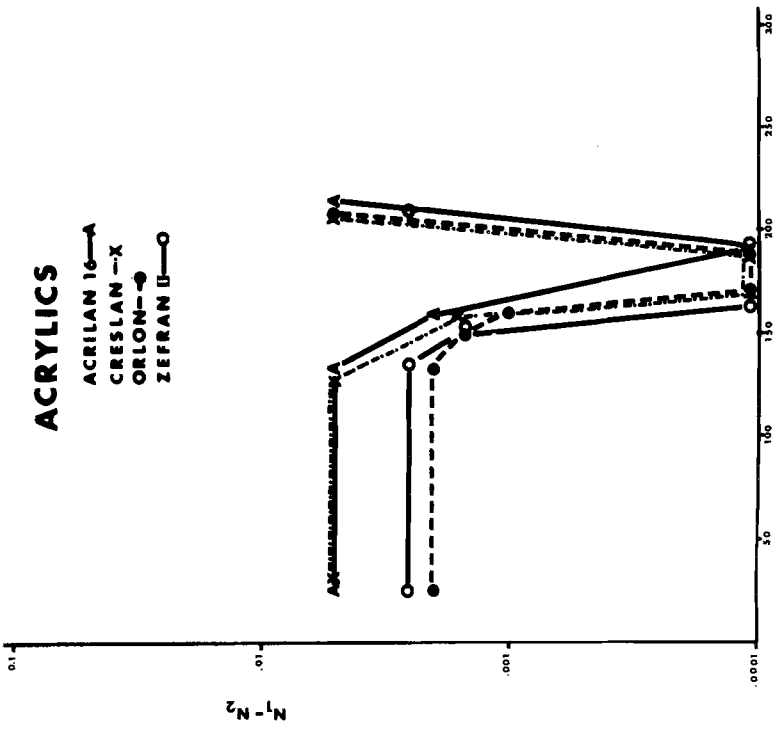

FIG. 2—*Birefringence versus temperature curves for the acrylic fiber specimens. Note that each fiber's sign of elongation changes from negative to positive at approximately 190°C.*

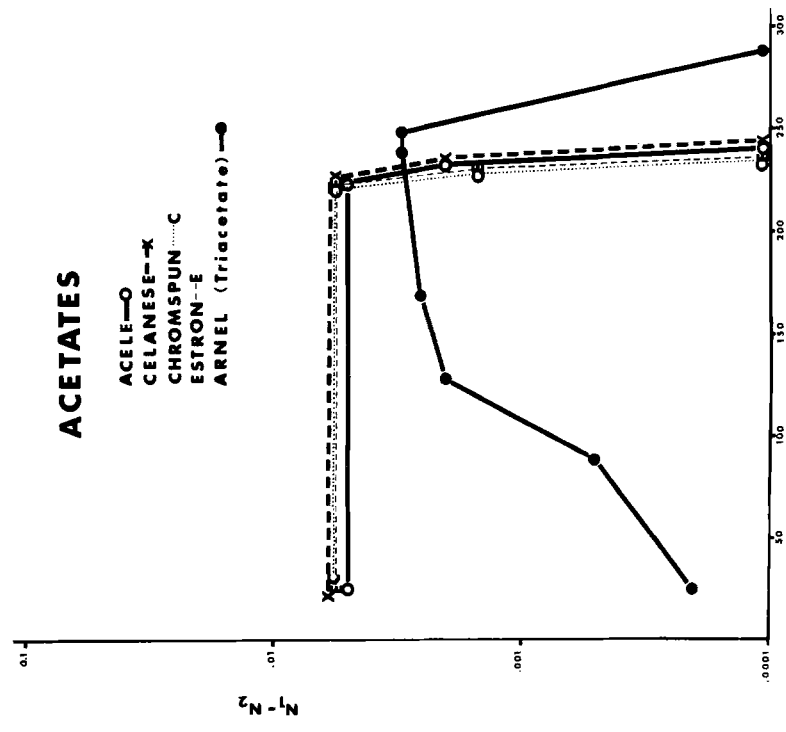

FIG. 1—*Birefringence versus temperature curves for the acetate and triacetate fiber specimens.*

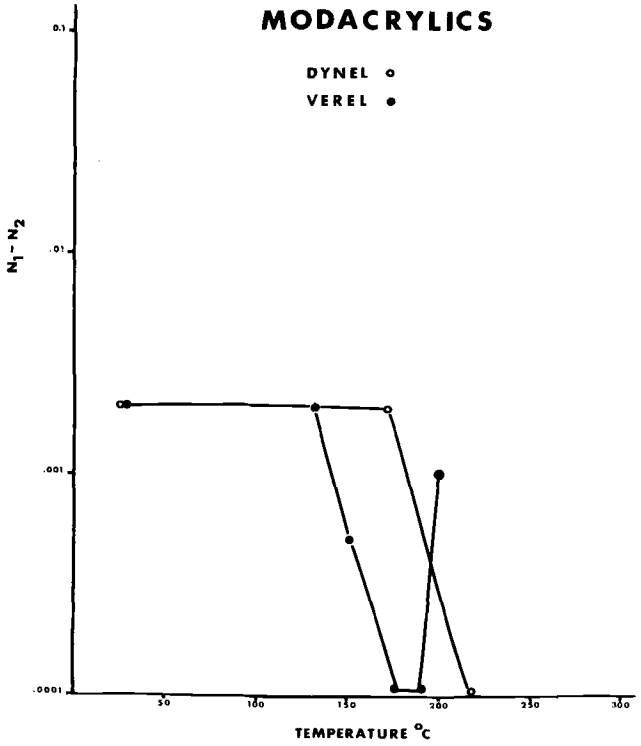

FIG. 3—*Birefringence versus temperature curves for the modacrylic fiber specimens. Note that Verel's sign of elongation changes from negative to positive, while Dynel's sign of elongation remains positive.*

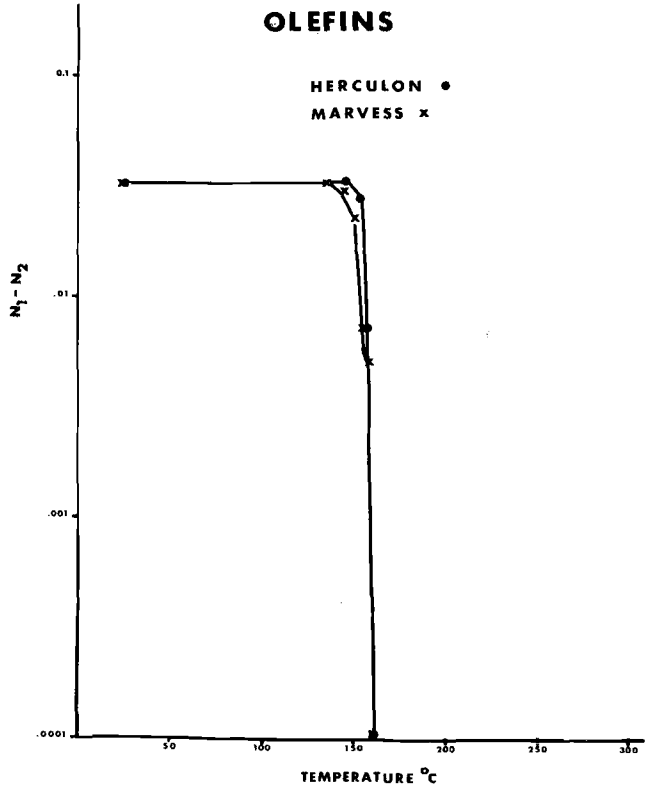

FIG. 4—*Birefringence versus temperature curves for the olefin fiber specimens.*

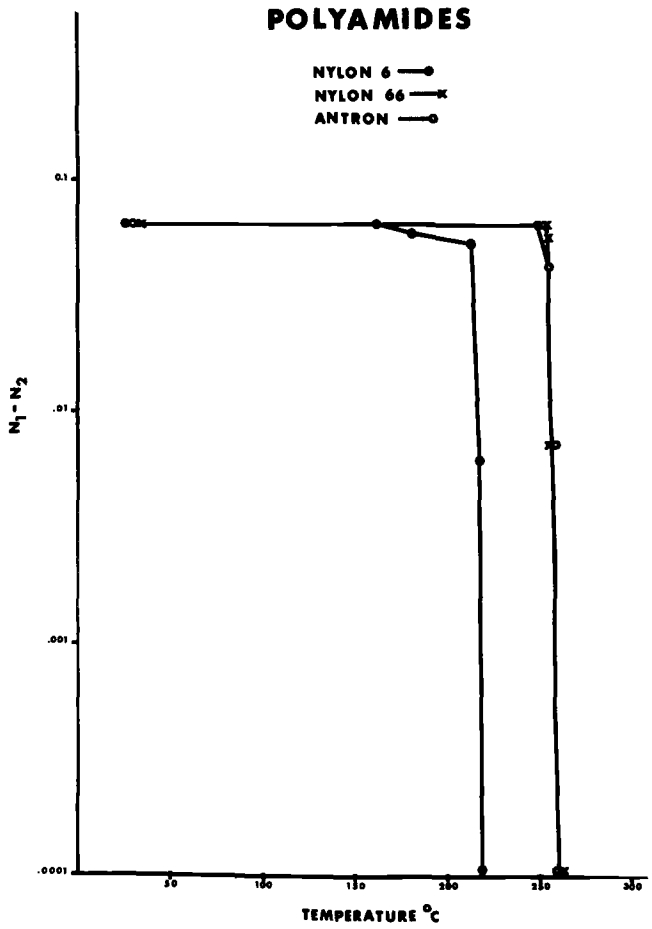

FIG. 5—*Birefringence versus temperature curves for the polyamide fiber specimens.*

the temperatures at which they occurred. The birefringence values for each sample were then calculated from the retardation. The standard deviation of the temperatures at the initial, intermediate, and final birefringence reading was calculated for each of the five generic classifications under study. These values are summarized in Table 3. Despite what appears to be a considerable range in some of the temperature values, the basic diagrams for each specimen were quite reproducible and recognizable.

Conclusions

Observations of changes in birefringence with temperature are useful in the forensic science examination of synthetic fibers. In this study, they have been used to assign synthetic fibers to generic classes. This research has also shown such observations to be extremely useful in the forensic science comparison of chemically similar synthetic fibers of the same generic class, as well as in the elimination of synthetic fibers of different generic types. The method could also be applied to the simultaneous analysis of fiber blends, a type of analysis often required in forensic science case work.

The graphs that show the changes in a fiber's birefringence by plotting birefringence as

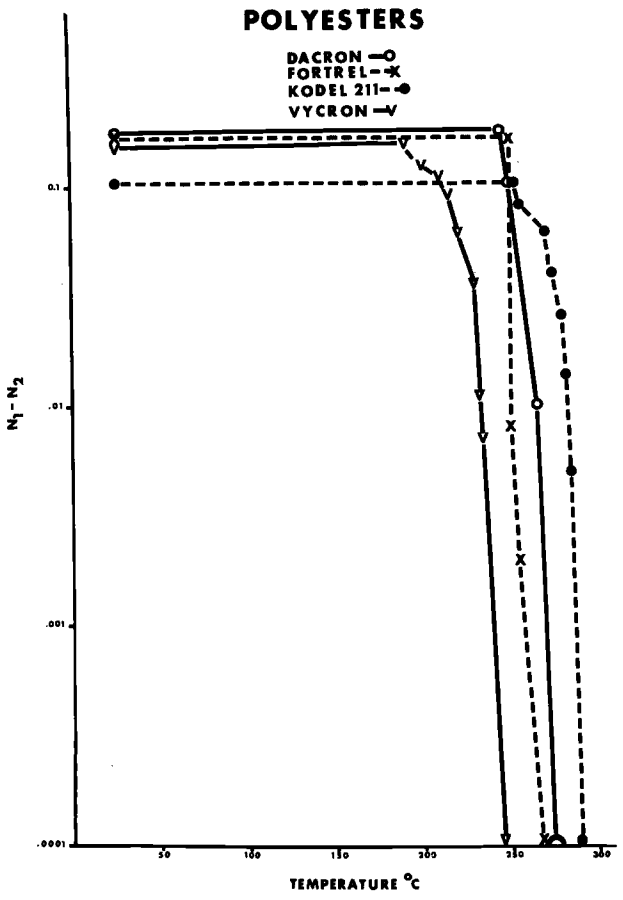

FIG. 6—*Birefringence versus temperature curves for the polyester fiber specimens.*

a function of temperature are useful in the forensic science identification and comparison of synthetic fibers. In most cases the generic class of the synthetic fiber could be determined from an examination of the plot. Only with modacrylic fibers was there a gross difference in the shapes of the curves obtained for two fibers of the same generic class, as might be expected. According to the established Federal Trade Commission definition of modacrylic fibers, the fiber must be composed of less than 85% but at least 35% by weight of acrylonitrile units, with the balance of the polymer being composed of some other monomer unit. Verel® contains 60% acrylonitrile units, while Dynel® is composed of 40% acrylonitrile units.

In the case of the triacetate fiber Arnel®, some confusion was experienced. As one examines the literature covering Arnel, one finds different values reported for its birefringence. The values have been reported as none [*19*], less than +0.001 [*20*], −0.0001 [*21*], and simply almost isotropic [*22*]. It is beyond the scope of this study to speculate as to why Arnel's birefringence seems to vary. However, to avoid confusion in the construction of the graph for Arnel only specimens of Arnel that exhibited a positive sign of elongation were employed in this study. Consequently, the data collected and presented on Arnel can apply only to the specific samples of Arnel examined in this study.

In the forensic science comparison of synthetic fibers, the curves have aided in demonstrating similarities and differences among fibers of the same generic type (see Figs. 1 to 6).

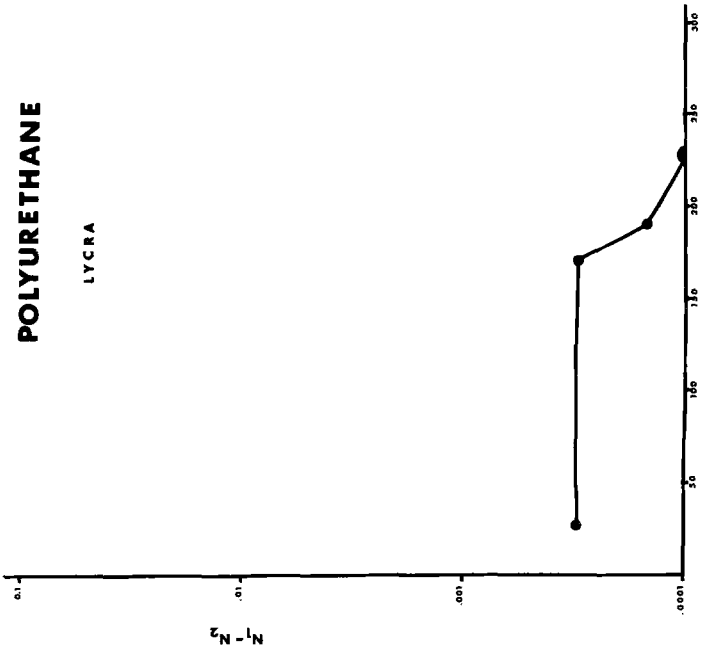

FIG. 8—*Birefringence versus temperature curve for the polyurethane fiber specimen.*

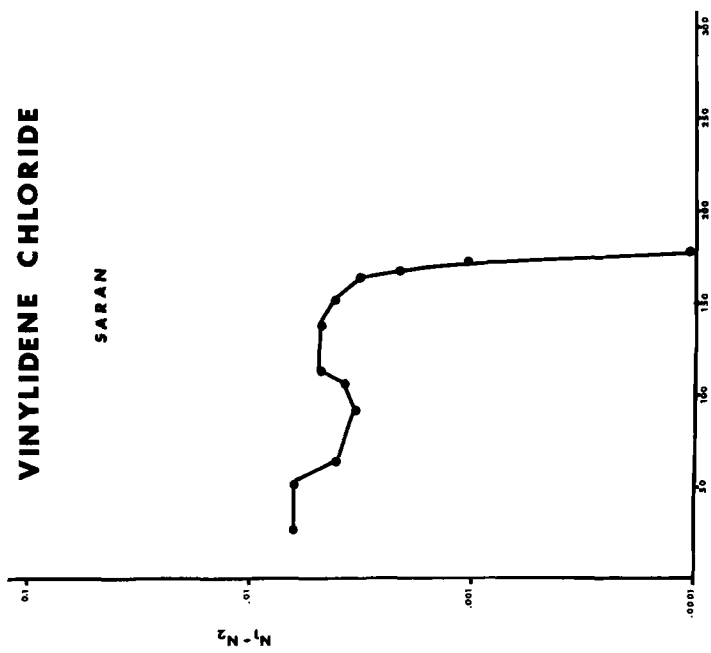

FIG. 7—*Birefringence versus temperature curve for the vinylidene chloride fiber specimen.*

TABLE 3—*Reproducibility study showing the standard deviations (SD) of the temperatures at which the birefringence started to change, an intermediate value, and the point at which the birefringence was no longer detectable.*

Proprietary Name	Generic Classification	Birefringence Value	SD Temperature, °C
Orlon	acrylic	−0.002	1.03
		I[a]	2.19
		+0.002[b]	2.40
Chromspun	acetate	+0.0055	0.50
		+0.0015	1.41
		I[c]	0.71
Antron	nylon 66	+0.060	1.22
		+0.040	0.83
		I[c]	0.68
Dacron	polyester	+0.175	1.58
		+0.010	2.20
		I[c]	3.60
Verel	modacrylic	−0.002	0.00
		I[a]	2.00
		+0.001[b]	1.25

[a] Fiber is almost isotropic; however, a slight birefringence is detectable.
[b] Fiber starts to char.
[c] Fiber is isotropic.

These graphs have also been extremely helpful in differentiating among man-made fibers of different generic classifications.

When this method for the comparison of synthetic fibers of the same class is employed, it is important that the questioned and known specimens be mounted side by side. This enables the criminalist to observe both specimens simultaneously and to detect subtle differences in the birefringence of the two samples during heating. These subtle intraclass differences in birefringence, which are not always evident in the curves, have been found to be of use in differentiating between closely related synthetic fibers of the same generic type originating from different sources.

This study has been limited to a number of the various types of synthetic fibers often encountered in forensic science case work. No attempt has been made to present data for all the hundreds of different synthetic fibers now available. However, data for a representative number of the various types of man-made fibers have been presented to demonstrate the apparent usefulness of this method. More study and data collection are needed to fully validate the usefulness of this method.

Summary

A method has been presented that promises to be of great use in the forensic science identification and comparison of synthetic fibers. The method incorporates two physical properties often used in the identification of man-made fibers into a procedure that is fast, easy to learn, and requires no equipment that is not readily available in most forensic science laboratories. This technique is presented as a different approach to the forensic science examination of synthetic fibers. It is not meant to replace the well-established methods now in use but, rather, to make a new, complementary method available to the forensic science community.

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