Chapter 6 Fibers

Objectives

After reading this chapter, you will understand:

- Why fibers are class evidence.
- How fibers can be used as circumstantial evidence to link the victim, suspect, and crime scene.
- Why statistics are important in determining the value of evidence.

You will be able to:

- Sample populations using statistical analysis.
- Distinguish and identify different types of fibers.
- Understand polymerization.
- Carry out an experiment in thin-layer chromatography.
- Judge the probative value of fiber evidence.
- Design and carry out scientific investigations.
- Use technology and mathematics to improve investigations and communications.
- Identify questions and concepts that guide scientific investigations.
- Communicate and defend a scientific argument.

"Wherever he steps, whatever he touches, whatever he leaves even unconsciously, will serve as silent witness against him. Not only his fingerprints or his footprints, but his hair, the fibers from his clothes, the glass he breaks, the tool marks he leaves, the paint he scratches, the blood or semen he deposits or collects—all of these and more bear mute witness against him. This is evidence that does not forget."

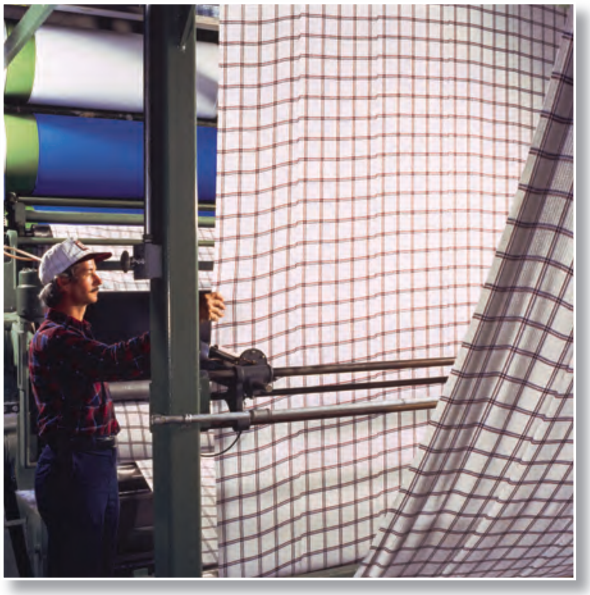
—Paul L. Kirk (1902–1970), forensic scientist

Using Fibers as Evidence

Teacher Note

The TRCD for this chapter includes a PowerPoint presentation, which is an overview of the chapter. It can be used as introductory material or at the end as a review.

Fibers are everywhere. Because **textiles** are mass-produced, it is difficult to trace a fiber back to a specific source, but fiber evidence is valuable because it creates links among victims, suspects, and places. Statistics and probability are important in narrowing the source. Investigators identify and compare fibers physically, using microscopy, as well as chemically.



Textile manufacturing

fibers: usually made up of many filaments twisted or bonded together to form a thread or yarn

textiles: fabrics woven in a distinctive pattern (although bonded textiles, such as felt, lack a pattern)

Reminder

Class evidence can be associated only with a group of items that share properties or characteristics. Individual evidence, such as fingerprints, can be related to a single source.

Probative value is the ability of evidence to prove something that is relevant to the crime.

More than 10 billion pounds of fibers are produced annually in the United States alone. Imports add billions of pounds more. For example, according to the American Apparel Manufacturers Association, more than 1.2 billion men's and boys' knit shirts were sold in 2003!

Like hair, textile fibers are among the most common items left at a crime scene. Trace evidence usually has no individuality; this is especially true of fibers, because they are mass-produced in huge quantities. Like hair, fibers are considered *class evidence*.

Fibers have *probative value* because they can create connections, or associations. For example, a suspect may deny he was ever in a particular place or ever in contact with a particular person. Yet questioned fibers (those found in the area or on the person) may be linked to the suspect.

Anything that can aid in narrowing down a fiber's origin to a limited number of sources makes that fiber much more valuable as evidence. Investigators compare physical and chemical characteristics of suspect (questioned) and known fibers. The more properties that are found to be similar, the stronger the case. Yet one inconsistency, one property that doesn't match, is sufficient to cancel out an association. That is why investigators must usually perform a great many tests to identify and compare fiber evidence.

Collection and Observation

Activity 6.1

Look at the manufacturer's label in your shirt, sweater, or jacket; at home, look at your curtains, rugs, and clothing. Do you know what those fiber products are? How can you describe them without a label? Most labels give the **generic** name of the fabric, like 100% cotton or 45% rayon–55% nylon. Some show only a trade or brand name.

Materials

For each group:

- 2-inch transparent tape
- clear acetate sheet
- stereomicroscope
- compound microscope

Procedure

Take several pieces of 2-inch-wide transparent sticky tape. Press one against your shirt, sweater, or blouse—whatever you are wearing above your waist. Rub it smooth, then pull it off slowly and smooth it out on a clear acetate page protector. Now repeat this procedure on your pants, skirt, or whatever you are wearing below your waist. Write in your lab book what fibers and hairs you think you will find. Label the source of each sample and look at it first under a stereomicroscope, then under a compound microscope. Sketch and label what you see. Classify the fibers and hairs you see and identify any you can. Did you predict the outcome correctly? What type of fabric is the best collector? What type is the worst?

generic: related to an entire group or class of products; not having a brand name

Teacher Note

Answers will vary.

Reminder

"When two objects come in contact with each other, material is transferred. The intensity and duration of the contact and the nature of the material determines the extent of the transfer."

—Edmond Locard (1934)

Suggested Assignment

The students should primarily see fibers from the fabric material. There probably will be hairs, both human and animal (pet), which can be identified according to the procedures outlined in Chapter 5, "Hair." The coarser the fabric, the better the chance of catching and

Sampling and Statistics

Activity 6.2

An expensive handheld calculator was taken from a student's hall locker and found later that day in a stairwell, completely smashed. Found with the pieces were two fibers of blue fabric. Approximately how many suspects are there in the student body of 1,500? Show how you arrived at your answer. Refer back to Chapter 2 for a method of approximation.

Rather than blue, suppose a green fiber had been found with the smashed calculator. How many students should be questioned? Show your work.

What are the odds of finding the responsible person based solely on the presence of the blue fibers? Based solely on the presence of the green fiber?

Suggested Assignment, *continued*

retaining fibers and hairs. Have the students share and compare their tapes. For homework, students could sample several areas of their home, such as a carpet, the kitchen floor, and a bed covering. The tapes should be labeled for inclusion in their notebooks; after portions of this chapter have been completed, identification should be easier.

Additional homework could include exploring the relationship between the composition of the

Suggested Assignment, *continued*

fabric and its use; for example, are most T-shirts cotton, and carpets nylon? Observations could be tabulated and summarized in class to deduce the most likely source of a particular kind of fiber. The point is that a type of fiber connected to a crime *may* lead to other areas of investigation.

Activity 6.2, *continued*

If there were two blue fibers and one green fiber found with the calculator, what are the odds of finding the perpetrator?

Statistical sampling can be a powerful mathematical method of approximation, and it is often the only method that applies to a forensic problem.

Believe it or not, knots and ligatures (something used to knot or bind) can be important to forensic scientists. Scientists can learn, for example, about a job, trade, hobby, or recreation of the person who tied the knots. Ligature marks left on a victim can sometimes be matched to the material that made them.

In the problem here, what fraction of students in your class are wearing something blue? Suppose that 10 students out of 30 are. If you assume that your class is typical of the entire student body, then you would expect 500 students to be wearing something blue ($^{10}/_{30} \times 1{,}500$). Suppose only one student in your class is wearing something green; then you would expect to see about 50 students wearing green ($^{1}/_{30} \times 1{,}500$). The odds of finding a combination of blue and green apparel on a student, based on this statistical sampling technique, would be 1 out of 90 ($^{10}/_{30} \times {}^{1}/_{30}$). You could expect approximately 17 students in the school to be wearing blue and green ($^{1}/_{90} \times 1{,}500$).

Here is an example of statistical sampling that should help in answering question 4 in the Checkpoint Questions on page 162.

School authorities at a high school wish to know student attendance at a football game. They give out 50 bright yellow caps to students randomly selected as they enter the stadium. At the end of the game, as students are leaving the stadium, spotters observe 2 yellow hats in a selection of 25 students, 1 cap among 35 students, and 4 hats among 40 students. How many students were at the game?

The first woven fabrics probably developed from ancient basket weaving. The oldest known woven cloth, found in Turkey, probably linen, is some 9,000 years old. Different textiles are typical of many ancient cultures: For example, linen was used in Egypt around 5000 BC; woolens were used in Bronze Age Scandinavia and Switzerland; cotton could be found in India by about 3000 BC; and silk was used in China by about 1000 BC.

$$\frac{2 + 1 + 4}{25 + 35 + 40} = \frac{7}{100} = \text{sampling total}$$

Let N equal the number of students at the game. The proportion of hats observed to those distributed should approximate the proportion of students in the total sampling group to those at the game:

$$\frac{7 \text{ hats observed}}{50 \text{ distributed}} = \frac{100 \text{ students sampled}}{N}$$

$$N = \frac{(50)(100)}{7} = 710$$

Sources and Types of Fibers

Fabric is made from fibers; fibers are usually made up of twisted **filaments** that can be classified as either natural or artificial. Natural fibers may come from animal, vegetable, or **inorganic** sources. Artificial fibers are synthesized or created from altered natural sources. Fibers are used to make textiles, such as cloth or carpeting; cordage, such as rope, string, or nets; brushes; filling materials for mattresses and upholstery; optical cables for transmitting information; and structural materials that are used in cars, tires, and airplanes. Each type of fabric has its own characteristics, which you can discover by using different tests. Fabrics are commonly made from fibers by weaving, although knitting, crocheting, knotting (macramé), braiding, netting, and felting are also employed. The three basic weave patterns are shown in Figure 6.1.

fabric: in this context, a cloth material made up of fibers woven or bonded together in a distinctive manner

filaments: single strands of material, usually twisted with other filaments to make a thread or fiber

inorganic: refers to substances not composed primarily of hydrocarbons, that is, carbon and hydrogen. Examples of inorganic fibers are asbestos and fiberglass. *Inorganic* is the opposite of *organic*.

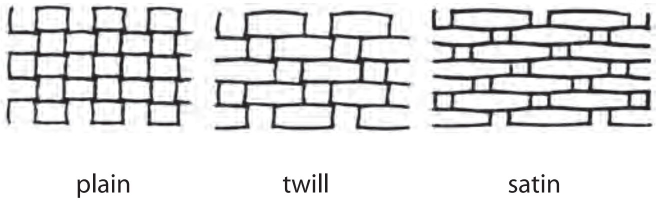


Figure 6.1 Types of weave

Fabric Observation

Laboratory Activity 6.1

Forensic scientists can identify fibers from different fabrics to help link evidence to a known source. This activity will acquaint you with different fibers comprising different fabrics in different weave patterns.

Materials

- Fiber samples for each group:
  - acrylic
  - nylon
  - polyester
  - rayon
  - linen
  - cotton
  - acetate
  - olefin
  - wool
  - silk
  - fiberglass
- stereomicroscope

Olefin is the term for a hydrocarbon containing a carbon–carbon double bond; for example, ethylene:

$$H_2C{=}CH_2$$

Advance Preparation

You can get samples of fabrics from a fabric or yarn store. Get white fabrics and wash them with a strong soap. Garage sales can yield a plethora of different types of fabric. All clothing sold in the

Advance Preparation, *continued*

United States must have a label listing all fibers that make up at least 5 percent of the fabric by weight. Fiberglass insulation provides fiberglass. A tarpaulin or rope may be made of a polyolefin. Use your imagination. Try to get pure material; blends make test results more confusing, although one sample of a 50/50 cotton/polyester blend is a good idea. Yarns and threads can also be used.

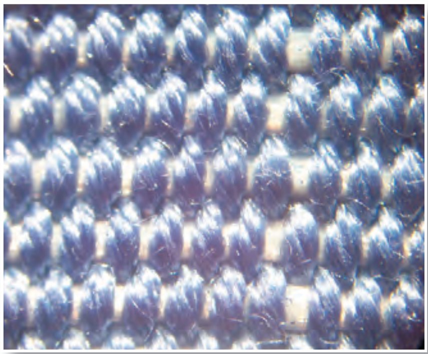
Cut samples into 1-inch squares or use a wad of yarn or thread. Allot only one sample of each known to each group of students. Also give each group an unknown from the set of samples. This should be all that they have to work with through the entire set of experiments. The object is to match and identify the unknown.

Laboratory Activity 6.1, *continued*

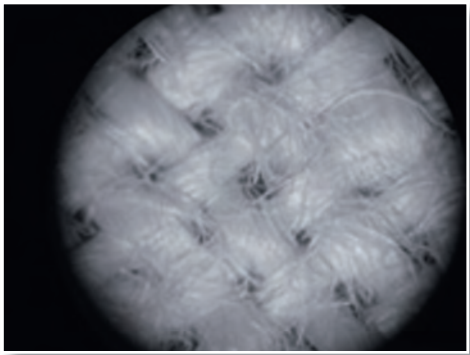
Procedure

Do not write in your textbook. Take notes in your science notebook.

1. **Get a set of labeled fabric samples. Tape the samples to a paper, label them, and keep the samples in your notebook. You will need to use these samples for several experiments.**
2. **Examine each sample with a stereomicroscope. Draw the different patterns.**
3. **Name the weave pattern observed in your fabric samples.**



Micrograph of a T-shirt



Micrograph of jute

yarn: a continuous strand of fibers or filaments, either twisted or not

warp: the lengthwise yarn or thread in a weave

weft or **woof**: the crosswise yarn or thread in a weave

blend: a fabric made up of two or more different types of fiber, usually as warp and weft

Figure 6.2 shows a good student example of fabric observation from Laboratory Activity 6.1.

In a weave, the lengthwise **yarn** is called the **warp**. It is usually stronger, smoother, and more even, with a tighter twist to it, than the **weft** or **woof**, which is the crosswise yarn. The warp need not be the same material as the weft (as in a **blend**), nor the same color. Sometimes, the warp and the weft have different diameters in order to produce special effects, such as ribbing. Can you tell the difference between warp and weft in the fabric samples you examined? Are there any blends? Note your observations on your drawings.

List examples of natural fibers. Remember that they can be derived from animals, plants, or naturally occurring inorganic material.

All fibers are *polymers*, long chains made up of simple molecules. Although there is a limited number of natural fibers, there are thousands of different

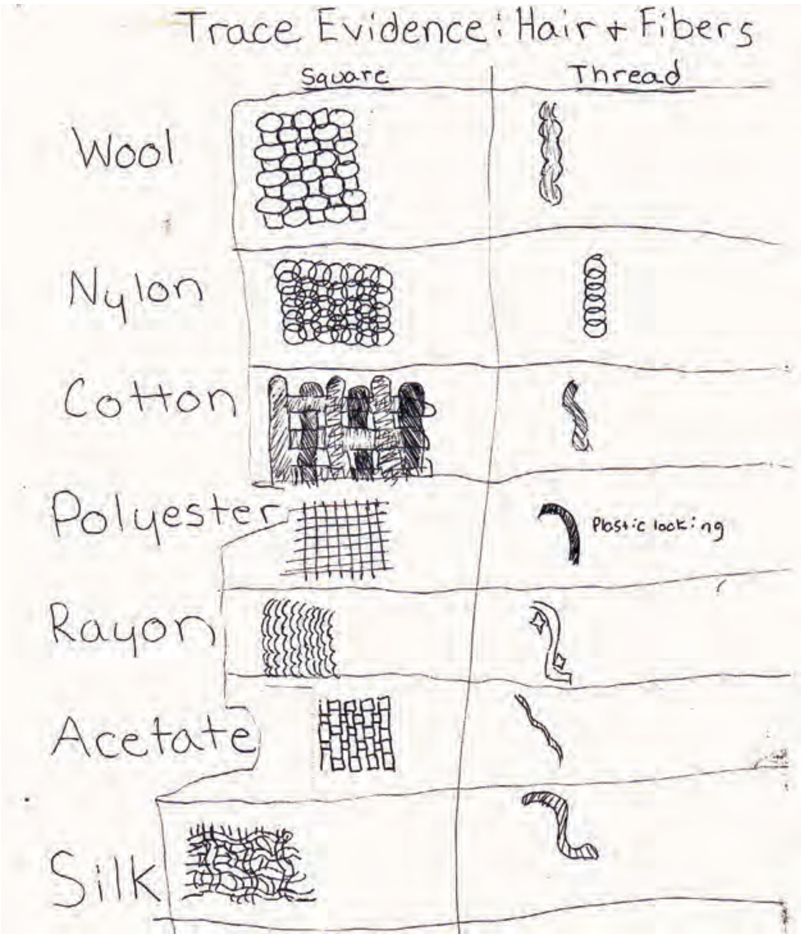
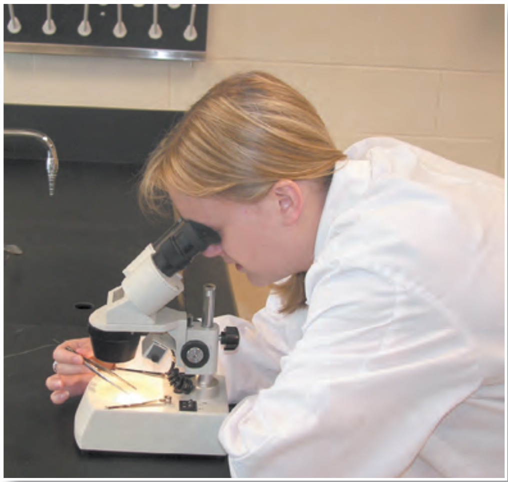


Figure 6.2 An example of a student worksheet



Student examining fibers under a microscope

artificial fibers produced today. Natural products such as cellulose can be chemically changed to form rayon, for example. Artificial fibers are polymers treated chemically to have particular characteristics that work well for specific textile applications. Examples include nylon, polyesters, and acrylics; these are sold under a bewildering array of trade names, such as Orlon, Dacron, Dynel, and Gore-Tex.

Reminder

polymers: giant molecules created when simple molecules, called monomers, are chemically joined together; also called macromolecules. Examples of polymers include mineral silicates, DNA and RNA, cellulose, starch, and keratin. The word *polymer* is derived from the Greek words *poly*, meaning "many," and *meros*, meaning "parts."

Fiber Morphology

World textile production is well over 100 billion pounds per year. There are hundreds of different types of natural and synthetic fibers, and thousands of colors, shapes, sizes, and treatments. What are the important characteristics that can be used to identify and compare fibers as forensic evidence?

Laboratory Activity 6.2

Microscopic Examination of Fibers

One of the major characteristics of a fabric is the fibers it is made of. Of course, the fibers have their own characteristics. Often a crime scene investigator will have just a few threads to examine.

Materials

For each lab group:

- fabric samples
- compound microscope
- glass slides
- glycerin or mineral oil
- cover slips

SAFETY ALERT! CHEMICALS USED

Always wear goggles and an apron when working in the labaratory

Procedure

Do not write in your textbook. Take notes in your science notebook.

1. Pull a fiber from each of your fabric samples; pull a fiber from both warp and weft if the fabric is a blend. Mount the fibers on a microscope slide, using mineral oil or glycerin as the medium. A cover glass will help keep them flat. Be sure to label each one so you don't get them mixed up.
2. Draw and describe features such as color, the number of strands or filaments, twist, and kinkiness.
3. Estimate the diameter of the fiber as well as that of a single filament if possible (use the methods described in Chapter 5, "Hair").
4. Can you classify the different fabric samples according to what the fibers look like?

Teacher Demonstration

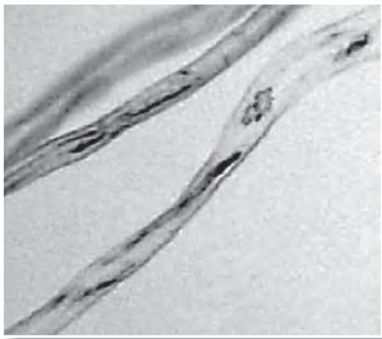
Examples of natural fibers include silk, wool, mohair and cashmere (goat), cotton, linen (flax), jute, sisal, asbestos, and fiberglass.

Use paper clips to demonstrate polymerization. A single clip is a monomer; two linked together, a dimer. Link a dozen or so to form a short straight-chain polymer. A copolymer is composed of two or more different monomers; it can be illustrated by using colored paper clips along with the regular ones. Cross-linking involves chemically linking polymer chains to each other. Show this by linking two chains in several places with extra clips. Two paper clip polymer molecules side by side can slip by each other; however, cross-linking prevents this.

Teacher Note

Cotton is about the easiest fiber to identify microscopically; it has a ribbonlike shape with twists at irregular intervals. Synthetic fibers are usually composed of very smooth filaments. Animal fibers can be identified using the criteria described in Chapter 5, "Hair."

Micrograph of cotton fiber



Cotton filaments

Fiber Cross Sections

Synthetic fibers are extruded when they are hot; this means that they are forced out of a spinneret, which is a nozzle like a showerhead; and then they are wound or woven. The holes in the nozzle need not be round, so the cross-sectional shape of a fiber filament may be one of its characteristics (see Figure 6.3). Making slides and observing the cross section can be fussy work, but it can also be very informative.

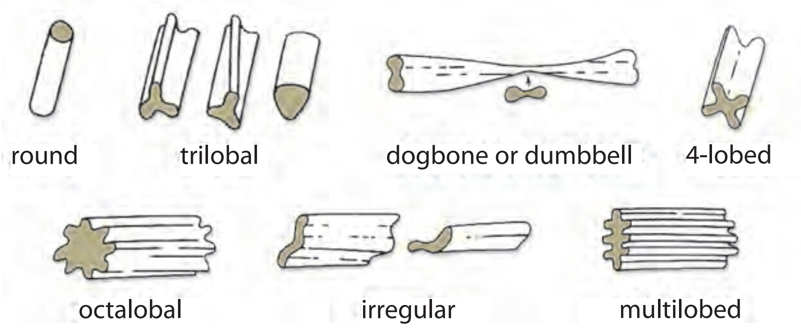


Figure 6.3 Synthetic fiber cross sections

Spinneret

Preparation of Fiber Cross Sections

Laboratory Activity 6.3

One important characteristic of a fiber is its shape in cross section.

Materials

For each lab group:

- fabric samples
- Beral pipette
- mounting medium, such as Paraplast or Norlands
- scissors or single-edged razor blade
- compound microscope

SAFETY ALERT! CHEMICALS USED

Always wear goggles and an apron when working in the labaratory

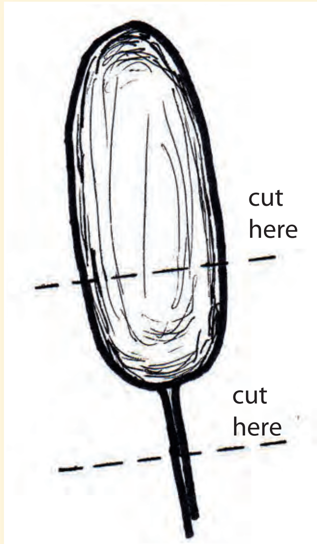
Procedure

Do not write in your textbook. Take notes in your science notebook.

1. Cut the top and most of the tip off a Beral pipette, as in the diagram.
2. Cut about 2 cm of fiber, untwist it to unravel the strands, and push it into the narrow part of the pipette.

Laboratory Activity 6.3, *continued*

3. Add a drop or two of a mounting medium so that it flows down the capillary.
4. After it has dried, slice sections as thin as you can. Mount them flat and observe in transmitted light under the microscope at high power. Water will make the contrast sharper. Record your observations in your science notebook.



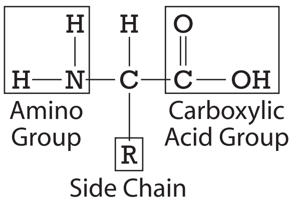
Beral pipette

polypeptide: a biochemical polymer formed by linking amino acids; the longer chains are called proteins

helix: a spiral arrangement, like a corkscrew, of a long-chain molecule

keratin: the main protein in all animal hair, including human hair

Amino acids contain nitrogen. Generally, compounds containing nitrogen can break down to form ammonia (NH_3), which is basic.



The Chemical Structure of Fibers

As you have noted, the physical characteristics of fibers and cloth provide only a little information for classification. However, the chemical structure of a fiber defines many of the properties that can be used to identify and further classify it.

Fibers are made up of polymers. Wool, for example, is composed of **polypeptide** chains, the monomers being amino acids (see Figure 6.4). The polypeptides form a complex, **helix**-shaped protein called **keratin** (see Figure 6.5). Keratin contains 19 different amino acids along its polymer chain; one of them, cystine,

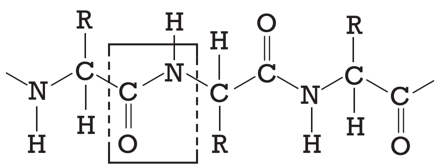


Figure 6.4 Polypeptide chain (R is the C—H part of any amino acid)

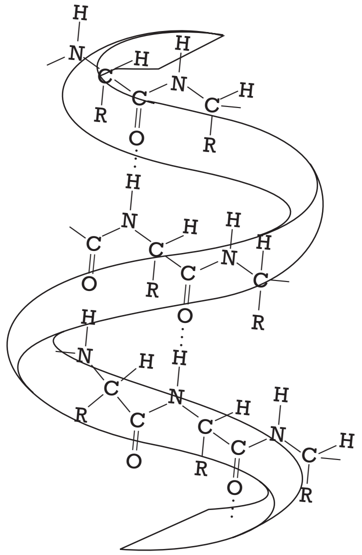


Figure 6.5 A wool helix

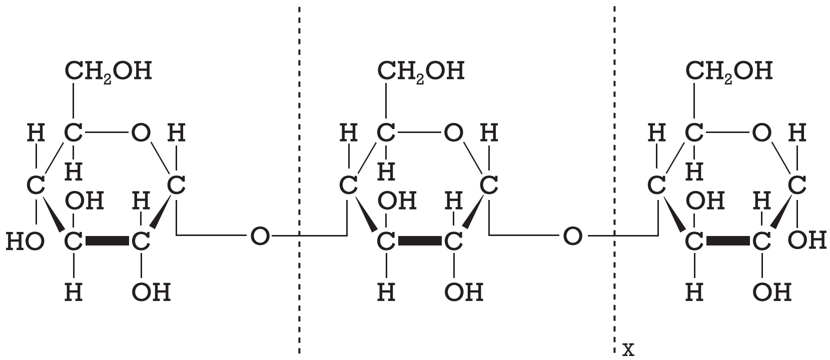


Figure 6.6 Cellulose molecule

contributes a relatively large amount of sulfur from its —S—S— bond. This accounts for the distinctive smell of burning wool.

Silk is also a protein; it is simpler in structure than wool because about 60 percent of the protein chain in silk is made up of only two different amino acids. Raw silk is produced by the larva of a moth, commonly called a silkworm. A moth lays 300 to 500 eggs a year. Each egg develops into a silkworm about 1 inch thick and 3 inches long. The worm spins the raw silk around itself to form a cocoon. The silk is processed by killing the larva, unwinding the cocoon, washing it in hot soapy water to dissolve the glue the worm produces to hold the silk filaments together, and collecting the white strands of pure silk.

Cotton is a plant fiber made up of cellulose, just like wood (see Figure 6.6). Cotton burns much the same way and smells like burning leaves. It is a straight-chain polymer of glucose. This polymer can be designated as $—(C_6H_{10}O_5)_n—$, where the formula is that of glucose, the monomer, and n refers to the number of monomer units that make up the polymer chain.

More textile products are made of cotton than of any other single fiber, although only about 40 percent of the fiber market is cotton. Approximately 60 percent of all clothing and home furnishings are made up of cotton. Therefore, statistically, cotton has low probative value without any other peculiar characteristics; it is too common for a sample to be uniquely linked to a known source.

Linen is a plant fiber, made from the flax plant. Like cotton, it is composed of cellulose, but the fibers are longer. Flax fibers are somewhat brittle

> Sericulture (the raising of silkworms) began in China in 2640 BC. Raw silk was exported, but the export of silkworm eggs was punishable by death. Silkworm eggs and seeds of the mulberry tree, on which the worm feeds, were supposedly smuggled to Constantinople in about AD 550. The Byzantine Empire became famous for its fine silk products.

Cotton plant

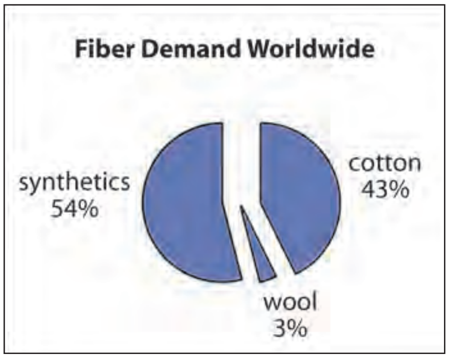
and so are often blended with other fibers. Linen is used in some clothing, tablecloths, napkins, and handkerchiefs. Jute is another cellulosic plant fiber used to make coarser products than cotton, like burlap, rope, and carpets. It is grown mainly in China, India, and Bangladesh.

The fibers described above are natural fibers. Rayon and acetate are natural fibers that have been chemically altered. Rayon is a very pure cellulose fiber; it also burns like cotton. Rayon can be found in all sorts of apparel, as well as in bedspreads, blankets, curtains, and upholstery.

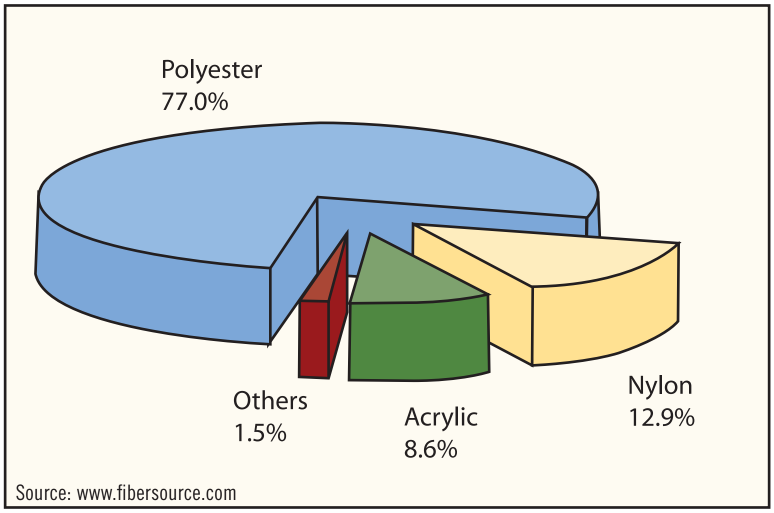
Acetate fibers are also made from cellulose through a reaction with acetic acid. Generally, when a polymer contains carbon–oxygen bonds in a *carboxyl group*, it will break down into an acidic product. Acetates are used in making blouses, dresses, draperies, upholstery, and cigarette filters.

plastics: substances that flow under heat and pressure and can, therefore, be molded into various shapes. All plastics are polymers, but not all polymers are plastics.

Many, many different human-made (that is, synthetic) materials are used to make fibers and are included, alone or in combination, in textiles. Some of the same materials, as well as many other polymers, are used in **plastics**.



Nylon was the first truly synthetic fiber to be discovered and sold. In 1935 a chemist working for DuPont prepared a polymer from adipic acid and hexamethylenediamine to create a polyamide called Nylon 66. This material could be extruded and was stronger and more chemically inert than natural fibers—that is, it didn't react chemically with other materials as easily as do natural fibers. Nylon is used in clothing, underwear, raincoats and windbreakers, bedspreads, carpets, upholstery, and rope.



Worldwide synthetic fiber production, 2004

Probative Value of Fabrics

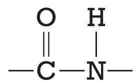
Activity 6.3

Which of the following fabrics would have the most probative value in an investigation? Which would have the least? Explain.

Answer

The particular content and percentages of #5 are unusual, whereas #3, 100% cotton, is the most common in the world.

Synthetic Polymers



an amide functional group

Polymers can be broadly classified by generic names that describe their chemical type. For example, wool and silk are polypeptides (a biochemical term). The peptide bond is boxed in Figure 6.4. Chemically, it is called an amide functional group or link, and it is the weakest point for chemical breakdown in the polymer chain. Monomers that make up nylon are also linked by an amide group. Polyamides (a chemical term) constitute one such generic classification of fibers and plastics.

A typical polyester is produced from a dialcohol and a diacid. If ethylene glycol and terephthalic acid are used as the monomers, the product is commonly called PET. Many beverage containers are made of PET, as are audio- and videotapes, paints, food packaging, and film. Fibers of PET, such as Dacron and Terylene, make up 40 percent of the synthetic fibers market.

Another generic classification of fibers and plastics is the polyesters. Dacron is an example. Its chemical structure is shown in Figure 6.7. The ester functional

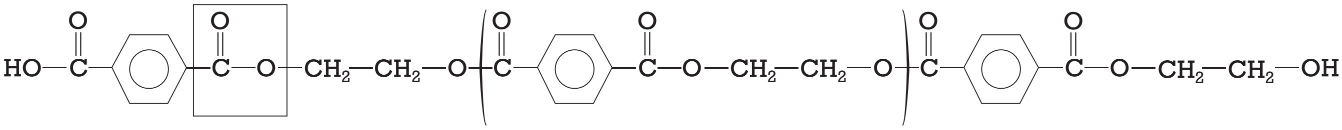
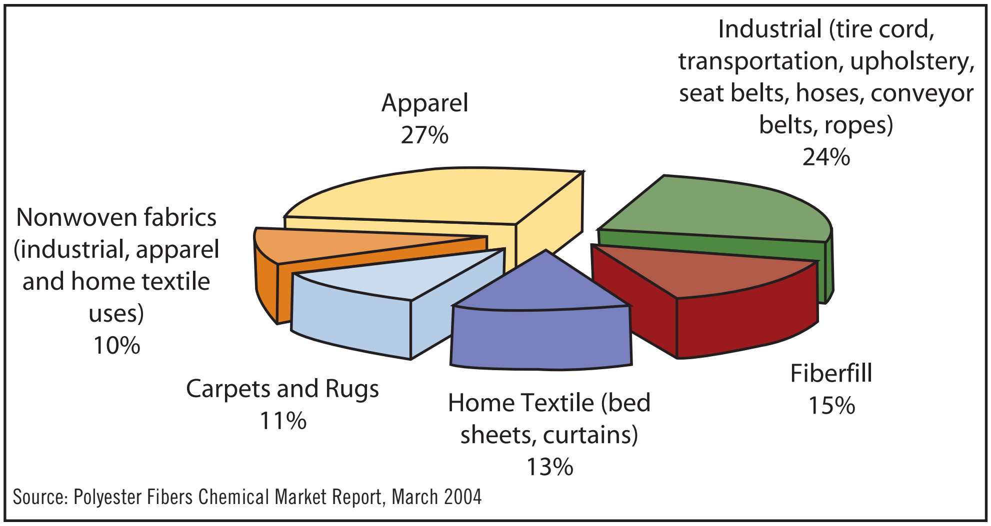


Figure 6.7 Structure of Dacron

Plastic Recycling Symbols	
1 PETE	**Polyethylene terephthalate -** Fizzy drink bottles and oven-ready meal trays.
2 HDPE	High-density polyethylene - Bottles for milk and washing-up liquids.
3 V	**Polyvinyl chloride -** Food trays, cling film; bottles for squash, mineral water, and shampoo.
4 LDPE	**Low-density polyethylene -** Carrier bags and bin liners.
5 PP	**Polypropylene -** Margarine tubs, microwaveable meal trays.
6 PS	**Polystyrene -** Yogurt pots, foam meat or fish trays, hamburger boxes and egg cartons, vending cups, plastic cutlery, protective packaging for electronic goods and toys.
7 OTHER	**Any other plastics** that do not fall into any of the above categories. An example is melamine, which is often used in plastic plates and cups.



Polyester end uses, 2004

group is boxed. This group is the weakest link, chemically, in polyester chains, yet it is stronger than the amide group.

The preparation of another polyester is described by the equation in Figure 6.8.

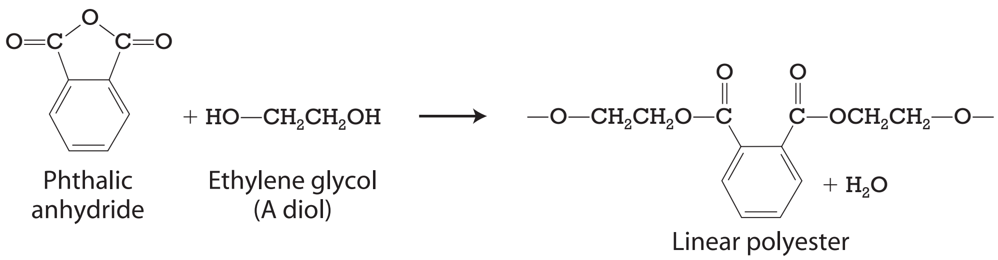


Figure 6.8 Preparation of a polyester

Note that the dialcohol, ethylene glycol, combines with the diacid, phthalic anhydride, to form a straight-chain polymer, such as Dacron. Glycerol, on the other hand, is a triol and has three hydroxyl groups to react with the diacid to form a cross-linked, three-dimensional polymer. Write the chemical equation and draw a portion of the cross-linked product (see Figure 6.9).

SCILINKS NSTA
GO TO www.scilinks.org
TOPIC polymers
CODE forensics2E140

The properties are quite different: A linear polymer is flexible, as you might imagine, whereas cross-linked material is so rigid and hard that it is used as a coating in paint.

We will prepare a simple polyester in the next laboratory activity.

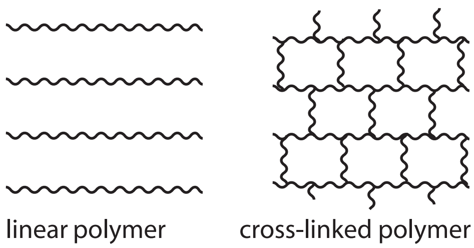


Figure 6.9 Linear and cross-linked polymers

Preparation of a Polyester

Laboratory Activity 6.4

Materials

For each lab group:

- safety goggles
- phthalic anhydride
- sodium acetate
- ethylene glycol
- glycerol
- test tubes
- Bunsen burner
- plastic film

SAFETY ALERT! CHEMICALS USED

Always wear goggles and an apron when working in the labaratory

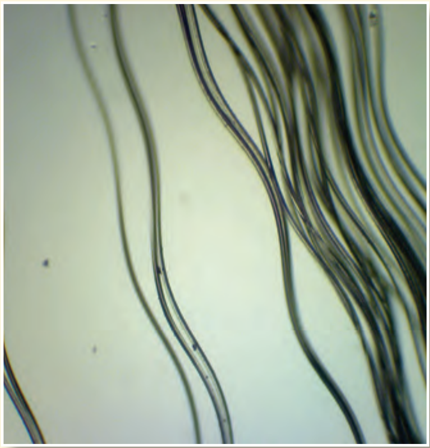
Procedure

Do not write in your textbook. Take notes in your science notebook.

Put on your safety goggles.

1. Place 2 g of phthalic anhydride (mp = 131°C) and about 0.2 g of sodium acetate (a **catalyst**) in each of two 15-ml test tubes.
2. To one tube add 0.8 ml of ethylene glycol, and to the other add 0.8 ml of glycerol (glycerin).
3. Clamp both tubes so that they can be heated simultaneously with a flame. *Never heat a test tube pointed toward another person.*
4. Heat the tubes gently until the solutions appear to boil (water is eliminated during the reaction), then continue the heating for 5 minutes (at about 160–180°C).
5. Continue heating the test tube containing the glycerin vigorously for an additional 3 to 5 minutes (up to 200°C).
6. Pour the contents of the test tubes onto aluminum foil or a plastic film and let them cool. Compare the **viscosity** of the two polymers as you pour.
7. Compare the physical properties of the two polymers. Note your observations. Explain.

   The cross-linked polymer is called Glyptal and is used in alkyd (oil-based) paints and resins. Why wouldn't it be used for fibers?



Polyester filaments

Answer

View the cross-linked polymer in three dimensions. It is too rigid to form a filament with enough flexibility to make fabric. On the other hand, the linear polymer chains can be bent.

Advance Preparation

Phthalic anhydride, sodium acetate, ethylene glycol, and glycerin can be purchased from a science product supplier, or check with your Kendall/Hunt representative.

catalyst: a small amount of a substance that increases the rate of a reaction without being used up in the process

viscosity: the resistance of a fluid to flow. Water has a low viscosity relative to syrup. Heating generally lowers the viscosity of a liquid.

A chemistry student
named Chester
Spent hours in the lab
each semester.
He discovered one week
A synthetic unique
Which he named for his
wife, Polly Esther.

Procedure

The polyester should be clear and somewhat flexible; the Glyptal should be hard and rigid, and may be discolored. Both may feel a little "greasy" because of unreacted glycol which can be cleaned off with soap and water. It may be easier to discard the test tubes, although acetone will clean up the polyester; the Glyptal is more difficult to clean up. The time involved for this experiment is about one-half hour.

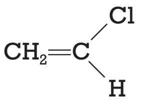
homopolymers: polymers made up of one type of repeating unit. Each is made from one monomer only.

polyethers: a series of carbon atoms connected by oxygen atoms, such as:

$$-[O\text{-}CH_2\text{-}CH_2]_n$$

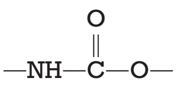
The subscript *n* refers to the number of ether units in the polymer.

This monomer makes polyvinyl chloride (PVC), used in many plastics such as pipes, floor tile, and raincoats.



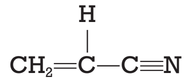
Vinyl chloride

Polyurethanes are used in making foam rubber, rigid foams, and coatings.



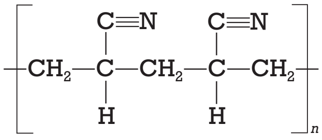
Urethane functional group

Polyester fibers are used in nearly every form of clothing, in many home furnishings such as carpets and curtains, in ropes and nets, and as fiberfill for various products.

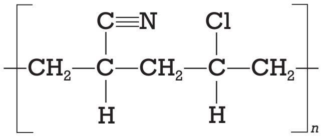


Acrylonitrile

The acrylics are yet another class of synthetic fibers. **Homopolymers** of acrylonitrile were first marketed by DuPont as Orlon. Acrylic fibers are used in apparel such as sweaters and sportswear and in home furnishings such as blankets and area rugs.



Structure of Orlon

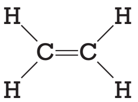


Structure of Dynel

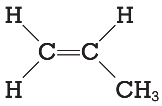
Another type of acrylic fiber, trademarked as Dynel, is made from acrylonitrile and vinyl chloride. Dynel may be found in fake fur, wigs, sleepwear, blankets, carpets, curtains, and stuffed toys.

The chloride in this polymer causes blue litmus paper to turn red when broken down by heat.

Spandex is a generic class of fibers primarily made up of segments of a polyurethane connected to long sections of polyesters or **polyethers** (see Figure 6.10). It is structurally similar to a polyamide. Spandex fibers can be stretched up to 600 percent and recover their original shape. They are always blended with other fibers and are used in articles of clothing where stretch is wanted, such as athletic apparel, bathing suits, and underwear.



ethylene



propylene

Filaments of nylon

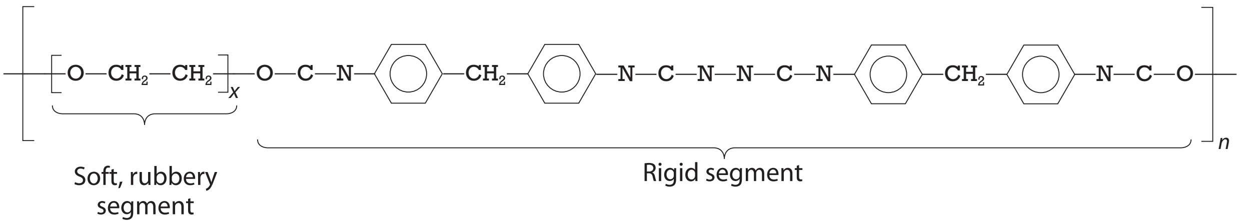


Figure 6.10 Structure of Lycra, DuPont's spandex fiber

The olefins are one last class of fibers you may come across in your investigation. These fibers are generally made from ethylene or propylene. Because there are no weak functional groups, such as amide or ester, this type of polymer is very resistant to weathering and chemicals. Polypropylene (pp) is used more in textiles than is polyethylene (pe) and is found in carpeting, upholstery, automotive interior fabrics, and rope.

Teacher Demonstration

Now is a good time to perform Teacher Demonstration 6.1, found on the Teacher Resource CD.

Fiber Analysis

As with most forensic evidence, the characteristics of a material are used as a basis for comparison. The following sections describe properties of fibers that are useful in forensic examinations.

Burn Tests

Laboratory Activity 6.5

How a fiber burns, its odor, and the appearance of the ash or residue can help an investigator identify it.

Materials

For each lab group:

- fabric samples and unknowns
- forceps
- Bunsen burner or alcohol burner
- burn test table handout

SAFETY ALERT! CHEMICALS USED

Always wear goggles and an apron when working in the labaratory

SAFETY NOTE *Do not smell the burning samples directly. Instead, use your hand to waft the fumes toward your nose.*

Procedure

Do not write in your textbook. Take notes in your science notebook.

1. Pull a bundle of fibers from each of your labeled fabric samples.
2. Hold the fibers with tweezers or forceps and bring them *slowly* into the open flame of a Bunsen burner or alcohol lamp.
3. Note any odor, whether the fabric continues to burn when you *slowly* remove it from the flame, the color of the flame, the type of ash or residue, and the color of the smoke.
4. Make a table in your notebook like that shown to describe your observations, or use the one given out in class. Fill in your table using words such as *scorches, smolders, fuses, melts, glows, shrinks, sizzles, flickers, flares,*

Look closely at your blue jeans. The blue yarn makes up the warp (vertical rows in the fabric), and the white yarn makes up the woof (horizontal filling). Denim is a three-on-one twill weave, where the warp passes over three strands of filling, then under one, over three, under one, and so on. So every fourth strand is white. The back side of the fabric, consequently, is one-fourth blue and three-fourths white. Take a look.

Laboratory Activity 6.5, *continued*

Procedure Notes

Table B6.1, Burn Test Results, provided on Blackline Master 6.2 on the Teacher Resource CD, may be used as a handout. It is provided and filled out in Blackline Master 6.3 for the teacher's benefit. The unknown can be selected from any one of the fabrics under study. Your students should perform microscopic observation first because burning is obviously a destructive test.

sputters, burns fast or slow, smoky, sooty, and the like. The ash or residue can be light gray, black, dark gray, shiny, clumpy, beady, sticky, feathery, and so on.

5. If you think you are dealing with a blended fabric, that is, one where the warp threads are a different generic material from the woof threads, unravel and separate the crosswise threads from the lengthwise ones. Twist each group into a bundle and check its burning characteristics.
6. Based on your microscopic observations and the results of the burn tests, what is the unknown fabric?

Student performing burn test

Laboratory Activity 6.6 Thermal Decomposition

Student performing thermal decomposition test

When polymeric materials are gently heated, they often break down, or decompose, to their monomer building blocks or other simple characteristic products (see Figure 6.11). For example, acetate fibers decompose to form acetic acid, which turns blue litmus paper red. Predict what each fabric type will do to red and blue litmus paper. Write your predictions in parentheses in a table in your notebook or the table handed out in class.

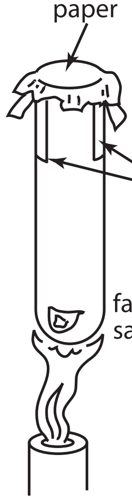


Figure 6.11 Thermal decomposition

Materials

For each lab group:

- fabric samples and unknown
- Bunsen burner
- red and blue litmus paper
- filter paper
- 5 to 10 percent lead acetate solution
- 13-mm test tubes
- thermal decomposition table handout

SAFETY ALERT! CHEMICALS USED

Always wear goggles and an apron when working in the labaratory

SAFETY NOTE *Also wear disposable lab gloves. Perform lab in a well-ventilated area.*

Procedure

Do not write in your textbook. Take notes in your science notebook.

1. Place a ½-cm-square piece of known fabric or a bundle of fibers in the bottom of a 13-mm test tube.
2. Wet pieces of red and blue litmus paper and stick them to the inside of the neck of the tube. The strips must not touch each other.
3. Cover the top of the test tube with filter paper cut to size; then moisten it with a drop of lead acetate solution.
4. Gently heat the base of the test tube. Record all your observations in your notebook or in the table provided.
5. Compare the observed litmus test to your predictions. What is happening? Why do silk and wool decomposition products turn lead acetate paper brown or black?

Procedure Notes

Table B6.2, Thermal Decomposition Results, provided on Blackline Master 6.4 on the Teacher Resource CD, may be used as a handout. It is provided and filled out in Blackline Master 6.5 for the teacher's benefit. The unknown can be selected from any one of the fabrics under study. Microscopic examination should come before the thermal decomposition tests. The column headed "Other" should be used for observations thought of by the students, such as type of smoke.

Use disposable test tubes, because the residue is almost impossible to clean. Lead acetate paper may be purchased instead of the filter paper and lead acetate solution.

The sulfur in the silk and wool polymers is liberated and reacts with the lead ions to form black lead sulfide.

Laboratory Activity 6.7

Chemical Tests

Advance Preparation

Do not use polystyrene well plates for the chemical tests, because acetone will dissolve them. Reagents: 6 M NaOH (add 24 g sodium hydroxide to about 80 ml water; when dissolved, add water to make 100-ml solution), 6 M HCl (dilute concentrated acid 50:50; always add acid to water), 6 M H_2SO_4 (dilute concentrated acid, one part acid to two parts water), household bleach (5.25 percent sodium hypochlorite, NaOCl), and acetone. Note the hazards of each of these (see Flinn's catalog).

In crime labs the reagents used are far more hazardous than these, are used at different temperatures, and are more difficult and expensive to obtain.

Table B6.3, Chemical Tests, provided on Blackline Master 6.6 on the Teacher Resource CD, may be used as a handout with answers on Blackline Master 6.7.

"M" is an abbreviation for molar, which is a concentration based on atomic or molecular weight of a substance.

The chemical composition of a polymer largely determines its chemical reactivity. With this series of tests, you will look into solubility and chemical decomposition of different fabrics to classify and identify them.

The chemicals you will be using in this activity involve strong acids, bases, and solvents, all of which can irritate the skin, cause irreparable damage to your eyes if not immediately washed, and dissolve your clothing, as you will see. Wear safety glasses, gloves, and an apron. Clean up spills, even a drop, at once. Report any accidents to your teacher. Wash your hands after the lab.

Student performing chemical tests

Materials

For each lab group:

- fabric samples and unknown
- 24-well plates: one for each group
- NaOCl
- acetone
- 6 M NaOH
- 6 M HCl
- 6 M H_2SO_4
- toothpicks
- chemical tests table handout
- safety goggles

SAFETY ALERT! CHEMICALS USED

Always wear goggles and an apron when working in the labaratory

SAFETY NOTE *Also wear disposable lab gloves. Perform in a well-ventilated area—strong acids!*

Procedure

Do not write in your textbook. Take notes in your science notebook.

1. **Pull a few fibers from your different fabric samples and place them in the 24-well plate.**
2. **Arrange your samples so you can test each one with the five reagents: acetone, NaOCl (household bleach, 5.25 percent sodium hypochlorite), 6 M NaOH, 6 M HCl, 6 M H_2SO_4.**
3. **Add enough reagent to cover each sample. You may use a toothpick or stirring rod to mush the fabric into the liquid. In some samples (those that are blends), only the warp or weft may dissolve. Try to keep them separate.**
4. **After 15 minutes, record your observations in a table in your notebook or in the table handed out in class.**
5. **It may help to look at your results under a stereomicroscope with light transmitted through the bottom of the well plate. Would you be able to identify each fabric from such tests? If not, what could these tests be used for?**

Having used the four procedures (microscopic observation, burning, thermal decomposition, and chemical testing), you should have unequivocally determined the composition of your unknown fabric. What is it? Why do you think so? Could you defend your conclusions if you were called to testify?

Teacher Note

Using microscopic observations and the results of the three tests described above, students should be able to identify any of the fiber samples. Each has a unique set of characteristics.

You can still perform some other tests that could further determine fiber type; these include tests for density, refractive index, and fluorescence. All are valuable because they can be used on single fibers and are not destructive.

Density

Often **density** can be used to confirm a type of fiber. Tests for density are especially useful for single fibers and are nondestructive. Table 6.1 on page 148 lists density ranges of common generic fibers.

Note that different types of fiber are similar in density. Olefins are unique in their ability to float on water.

density: a physical property of matter:

$$D = \frac{\text{mass of object}}{\text{volume of object}}$$

Density is usually expressed in g/cc. It can change with temperature (and pressure, in the case of gases).

Table 6.1: Density and Refractive Index of Fibers

Fiber	Density, g/cc	Refractive Index
cotton	1.45–1.60	1.53
silk	1.20–1.28	1.54
wool	1.28–1.35	1.54
acetate	1.28–1.35	1.47
acrylic (Orlon)	1.1–1.2	1.51
Dynel	1.28–1.35	1.53
nylon	1.1–1.2	1.53–1.54
polyester	1.35–1.45	1.57–1.60
rayon	1.45–1.60	1.52–1.54
olefin (pe + pp)	0.90–0.95	1.50 (pe); 1.54 (pp)
fiberglass	2.56	1.54
water	1.00	1.33

Advance Preparation

The first experiment is obvious: Drop each fiber into water. The second requires preparation of a density column such as that used in Chapter 10, "Soil and Glass Analysis," but in water because the organic solvents would dissolve most fibers. The NaCl solution in the "Density of Saturated Solutions" table is more practical.

Create a density experiment to tell nylon apart from polypropylene (pp). Now create one that tells nylon apart from polyester.

You might wish to experiment in comparing fiber density using an aqueous density column, using solutions shown in Table 6.2. Add 20 to 30 g of K_2CO_3 to the bottom of a 100-ml graduated cylinder. Fill the cylinder to the top by pouring water gently down the side. Let it sit undisturbed for days, allowing the carbonate to partially dissolve, with the greatest density at the bottom of the cylinder. Gently poke samples of fiber into the water. The samples should be immersed in a soap solution to lower their surface tension. You can obtain relative densities. Shining the beam of a flashlight into the column in a darkened room will make observation easier.

Table 6.2: Density of Saturated Solutions at 20°C

Salt	Weight Required, g	Water Required, cc	Density, g/cc
NaCl	31.7	88.0	1.20
KI	59.8	69.1	1.72
K_2CO_3	52.9	73.5	1.56
$CaCl_2 \cdot 6H_2O$	46.1	79.2	1.47

Refractive Index

Refractive index is the measure of the bending of a ray of light as it passes from air into a solid or liquid. When light passes from a medium of low density into a medium of higher density, such as from air into glass, the light rays are slowed down by the denser medium. The degree to which the medium changes the speed of light is the refractive index. For example, the speed of light in air is almost 300 million meters per second, and the speed of light in water is 225 million meters per second. Therefore, the refractive index of water is:

$$\frac{300{,}000{,}000}{225{,}000{,}000} = 1.333$$

Refractive index is a physical property of any substance that will transmit light. In a plastic or fiber, it depends on the physical composition and the alignment of the molecules. If a clear substance such as a fiber is put into a liquid that has the same refractive index, the substance becomes invisible because it bends the light at the same angle that the liquid does. If the liquid has higher or lower refractive index, the substance can be seen. The visible substance appears to have a "halo" of a dark boundary. This boundary is known as the **Becke line**, as shown in Figure 6.12.

Becke line: a halolike shadow appearing around an object immersed in a liquid of a different refractive index

A. The Becke line appears outside the fiber edges; the liquid has higher refractive index than the fiber.

B. The edges of the fiber cannot be seen; the refractive index is the same.

C. The Becke line appears inside the fiber image; the liquid has lower refractive index than the fiber does.

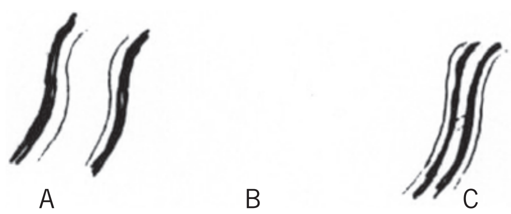


Figure 6.12 Illustration of the Becke line

Distinguishing Fiber Type by Refractive Index

Laboratory Activity 6.8

The intensity of the visible boundary around a substance depends on the difference in refractive index between the substance (fiber) and the liquid in which it is placed. Investigators measure refractive index of unknown samples in the laboratory with liquids of known refractive index. They put the samples in the different liquids until they can no longer see the Becke line. The refractive index of the known liquid is the same as that of the unknown sample when the sample is virtually invisible in the known medium.

Laboratory Activity 6.8, *continued*

Materials

For each lab group:

- fiber samples
- refractive index liquids with eyedroppers
- glass slides
- compound microscope
- tweezers

SAFETY ALERT! CHEMICALS USED

Always wear goggles and an apron when working in the labaratory

Procedure

Do not write in your textbook. Take notes in your science notebook.

1. Place a 1-cm length of known fiber on a microscope slide.
2. Add one drop of a liquid with a refractive index matching that of the fiber (see Table 6.2).
3. View the sample with a compound microscope at an appropriate magnification. You may have to pull a few filaments from the fiber so that they are transparent in transmitted light.
4. If the refractive indices match exactly, the Becke line disappears and the fiber is invisible.
5. Remove the fiber with tweezers and place a different fiber sample, one that has a different refractive index, in the liquid. Note the Becke line.
6. Draw what you have observed. What is the refractive index of some of the fibers?

Fluorescence

Some fibers will fluoresce when exposed to ultraviolet radiation. *Fluorescence* can be caused by the chemical and crystalline properties of the fiber itself, by dyes, or by **optical brighteners** and other agents added to the fabric. Some laundry detergents and bleaches have optical brighteners. Washing and wear can also reduce fluorescence. Nevertheless, fluorescence is useful for comparing fibers of a common origin as well as spotting fibers for collection.

Reminder

fluorescence: the absorption of light at one wavelength (often in the ultraviolet range) and its reemission at a longer wavelength (often in the visible part of the spectrum)

optical brighteners: colorless dyes that cause blue light to be reflected, thereby making an object look whiter

Advance Preparation

You can use commercial refractive index oils, or liquids of different refractive index can be made up from olive oil, castor oil, and clove oil as described in Chapter 10, "Soil and Glass Analysis."

Refractive index fluids are available in a 19-liquid "Series A" set that encompasses 1.46 to 1.64 in 0.01 increments, from Cargille Laboratories, 55 Commerce Road, Cedar Grove, NJ 07009, (973) 239-6633, but it is expensive. Alternatively, you can make up a set of such fluids using highly refined olive oil from a good ethnic foods store or Flinn, castor oil from a drugstore, and oil of cloves (pure) from an esoteric food store. Clove oil is also called eugenol. Refractive indices are:

n-butyl alcohol	1.402
olive oil	1.467
castor oil	1.482
clove oil	1.543

The refractive index varies linearly with the weight of each ingredient; thus, a 50:50 mixture of castor oil and clove oil would have a refractive index of 1.512.

Teacher Note

See Table 6.2 for refractive index of fibers.

Teacher Demonstration

A colorful teacher demonstration of fluorescence may be found in the TRCD as Blackline Master 6.8.

Observing Fluorescence in Fibers

Laboratory Activity 6.9

This activity will give you practice in observing the fluorescent properties of different fibers.

Observing fiber fluorescence of knowns in a UV light box

Materials

- fabric samples:

acrylic	acetate
nylon	olefin
polyester	wool
rayon	silk
linen	fiberglass
cotton	• UV lamp

Procedure

Do not write in your textbook. Take notes in your science notebook.

1. Look at your set of known fabric samples under short-wave and long-wave ultraviolet (UV) radiation.

   Caution: Ultraviolet light can damage your eyes. Do not look directly into any UV light source!
2. Describe the results in your notebook.
3. Take several fibers of the most fluorescent fabric and place them on the sleeve of a shirt or coat. Have your lab partner find them.
4. Observe various articles of clothing that people are wearing in class. Do many fluoresce? Do they fluoresce in same way?

Teacher Note

The black light (ultraviolet light) should have both short and long wavelengths. Lamps with both short and long wavelengths are available from a rock shop or places such as Cole Parmer or Flinn.

Laboratory Activity 6.9, *continued*

5. Work up statistics on the probability of being able to match a particular fluorescent fiber to a source in class.
6. Go back to the early part of this chapter and observe the material you collected on sticky tape. Does any of it fluoresce?
7. From your experience in the optical identification of fibers, can you guess what some of the material is?

Dyes

Look at the clothes your fellow students are wearing. Most are not white. What is the most common color?

Color is a good way to match fabrics or fibers, as in the statistical exercise on page 129 (Activity 6.2). Even the components that make up the dye itself can sometimes be separated and matched to an unknown.

In 1856, 18-year-old William Perkin made the first synthetic dye, quite by accident. He was trying to make quinine but didn't get the expected reaction. When he cleaned the flask with alcohol, it turned a beautiful deep purple color. Perkin seized the opportunity to commercialize the dye he had made. He called it mauve and made a fortune.

Different types of fabric react to dye molecules in different ways, depending on the chemical composition of the fiber, how its surface has been treated, the molecular makeup of the dye itself, and any subsequent chemical procedures it may experience.

Therefore, investigators can use the way a fabric accepts a particular dye to identify and compare samples.

Laboratory Activity 6.10

Dyeing Fabrics

You can use this activity to learn how different fabric samples react to dyes.

Materials

For each group:

- multifiber test fabric
- Testfabric Stain #1
- Testfabric Stain #2
- forceps

SAFETY ALERT! CHEMICALS USED

Always wear goggles and an apron when working in the labaratory

SAFETY NOTE *Disposable lab gloves are suggested to prevent staining of hands.*

Advance Preparation

Multifiber ribbon and the two Testfabric Stains are available from Educational Innovations, Inc. (www.teachersource.com). You can make the ribbon go further by cutting it up into 1-cm-wide pieces. A package of the two different dyes (stains) costs about $15 for five capsules of each color. The directions for dyeing come with the Testfabric Stains. One dye requires 1 ml of 10 percent acetic acid. You can also use commercially available

Procedure

Do not write in your textbook. Take notes in your science notebook.

1. To show how different types of fabric take a dye, soak a 1-cm-wide strip of multifiber ribbon in Testfabric Stain #1.
2. Repeat this process, soaking a new strip in Testfabric Stain #2.

The multifiber ribbon has 13 different fiber strips woven together into a single ribbon (see Figure 6.13). If you want to examine a particular fiber type, note that the warp, or lengthwise yarn, is a synthetic.

3. Fasten each strip of dyed ribbon and the fabric key in your notebook.
4. Dye about 1 square cm of each of your known fabric samples.
5. Rinse each sample thoroughly in hot tap water to set the dye, and blot it to dry. Compare your samples to the ribbons. Note which fabrics absorb the dye more readily than the others. Explain any differences you observe.

To use this procedure to identify colored textiles, you must first chemically remove (strip) the existing dye. This can be quite difficult for some dyes, as you will see in the next section.

Figure 6.13 Multifiber ribbon

Advance Preparation, *continued*

dyes such as RIT tints and dyes or natural products such as goldenrod or grape juice. The effect, however, is not as striking. Generally, when using dyes, immerse the fabric in simmering dye solution for 5 minutes, remove, rinse well in hot water, then dry. Sometimes the addition of salt helps fix the dye. You may wish to have the students design their own procedure for dyeing.

Chromatography

Dyes used to color fabric may be made up of different elements. Sometimes these can be separated by **chromatography**. Often the same color may be created by different combinations of dyes. Incredibly, there are more than 7,000 different dye formulations. Investigators may, therefore, compare the **chromatogram** of a dye taken from a colored fabric sample to others to find a match. This method is also used to analyze inks (Chapter 16, "Document and Handwriting Analysis"), metals (Chapter 9, "Trace Evidence"), drugs (Chapter 7, "Drugs"), and poisons (Chapter 8, "Toxicology: Poisons and Alcohol").

chromatography: a method of separating components of mixtures based on preferential adsorption or partitioning of components in a gas, liquid, or solution. In paper chromatography, the cellulose of the paper acts as the adsorbing medium. In thin-layer chromatography (TLC), the silica gel or alumina selectively adsorbs the components of the mixture. The word *chromatography* is derived from the Greek *chroma*, color, and *graphe*, writing.

chromatogram: the record of chromatographic separation

SCILINKS® NSTA

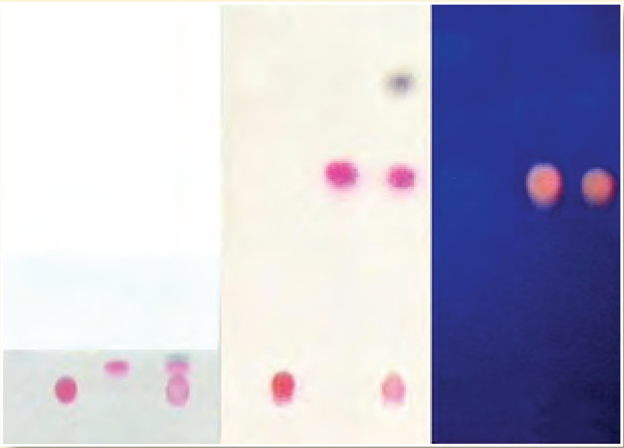
GO TO www.scilinks.org
TOPIC chromatography
CODE forensics2E153

On August 13, 2002, the FBI presented evidence at a press conference that conclusively linked Richard Evonitz to the abductions of Sofia Silva and Kristin and Kati Lisk. The most compelling evidence came from the trunk of Evonitz's 1992 Ford Taurus. The Latent Print Unit developed and identified two fingerprints of Kristin Lisk on the underside of the trunk lid, indicating that she had probably been locked inside the vehicle's trunk. Hairs consistent with hairs from Evonitz were found on Kristin and Kati Lisk's clothing, as well as on a rope used to bind Sofia Silva's body. Fibers found on all three victims were matched to carpets, including a pink bathroom rug, dark blue furry handcuffs, blankets, and other materials in Evonitz's home and vehicle. Newspaper clippings about the Lisk abductions and handwritten notes about the girls and the area where they lived were also found in Evonitz's apartment. Overwhelming and compelling physical evidence and cooperation among the FBI and multiple law enforcement agencies brought an end to the Lisk-Silva murder investigation.

—from the FBI 2002 Annual Report

Laboratory Activity 6.11 Thin-Layer Chromatography (TLC) of Dyes

You can use this activity to take dye from a fabric sample and analyze its components using chromatography.



TLC sequence: (l-r) just starting; chromatogram; same, uv light

Materials

For each lab group:

- TLC plates
- test tubes
- 0.5 M NaOH
- water bath and hot plate
- open-ended capillary tube
- 400-ml beaker and cover
- filter paper
- eluting solvent
- UV light

SAFETY ALERT! CHEMICALS USED

Always wear goggles and an apron when working in the labaratory

Procedure

Do not write in your textbook. Take notes in your science notebook.

Extract the dye from the fabric sample:

1. Cut a ½-cm-square piece from each colored fabric to be tested, or use an equivalent wad of thread or yarn.
2. Place in a 10 × 75-mm test tube.
3. Add 5 or 6 drops of 0.5 M NaOH. Be sure the fabric is immersed.
4. Place the test tube in a boiling water bath for 15 minutes. Record the color of the fabric and the color of the solution.
5. Gently draw a line, *in pencil,* across a 1 × 4-inch precut TLC strip 1 cm above one end. You can label the top end with a description of the sample.
6. Use an open-ended capillary tube to transfer extracted dye from the test tube in a water bath. Spot one drop on the center of the line. Be gentle so as not to dislodge the silica gel adsorbing medium. Keep the spot small.
7. Repeat this procedure ten times, allowing the drop to dry before each application.
8. Placing the strips on a hot plate on "low" will make the process quicker. The idea is to get as concentrated a spot as possible.
9. Set up the chromatographic developing chamber—a large beaker lined with filter paper (see Figure 6.14). Separation is quicker in a solvent atmosphere, which the soaked filter paper provides.
10. **Elute** the solvent and pour ½ cm in the bottom of the chamber, cover it with a watch glass or aluminum foil, and give it 15 minutes to equilibrate.
11. Lean the prepared and labeled TLC strips against the filter paper, spot side down. The solvent level should be well below the spotted dye. Replace the cover.

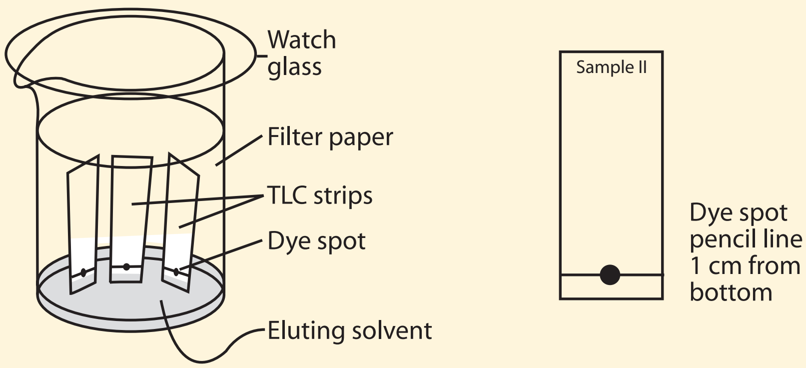


Figure 6.14 Thin-layer chromatography apparatus

Chromatography was developed by a Russian botanist named Tswett in 1906 to separate plant pigments. Various types of chromatography are now the most common means of separating and purifying components in mixtures.

The cotton in blue jeans is dyed with indigo, once extracted from plants. Termed a vat dye, indigo is introduced into the fiber in a colorless, soluble form, then "developed" into a colored, insoluble form that comes out of the solution on both the inside and the outside of the fiber. Indigo is one of the oldest dyes known.

elute: to extract one material from another, usually by means of a solvent

Advance Preparation

Blue is probably the most common fiber color. Collect as many different samples of blue cloth, yarn, and thread as you can. You may wish to dole out only a few for each group to analyze because the TLC plates are expensive. You should give each group or the whole class unknown blue fibers to match to the samples.

To obtain the TLC plates (silica gel on plastic or aluminum backing), check with your Kendall/Hunt representative, or you can order them from many scientific supply houses, such as Carolina Biological Supply Co. (www.carolina.com) or Flinn. Each 20-square-cm sheet can be cut with scissors to make 20 2 × 10-cm strips. Fluorescent

Advance Preparation, *continued*

TLC plates can also be purchased. If the developed spot is not fluorescent, it appears as a dark spot under UV illumination. Unfortunately, paper chromatography will not work for this experiment.

See Blackline Master 6.9 on the TRCD for procedure to prepare your own TLC plates.

To prepare the extracting solution, add 2 g NaOH to 100 ml water to make 0.5 N NaOH, or dilute the 6 M NaOH used earlier about 1:12. An eluting solvent system can be made of ethyl acetate (15 parts by volume), ethanol (7 parts), and water (6 parts). These two solvents are available from Flinn, for example, or can be purchased from your local hardware store. Another effective elution system is n-butyl alcohol (5 parts), acetone (5 parts), water (1 part), and NH_4OH (2 parts).

Sometimes you can make colorless spots on TLC plates (or filter paper) visible by placing the chromatogram in a covered beaker with a few crystals of iodine.

retention factor (R_f): a ratio used to characterize and compare components of samples in liquid chromatography

Laboratory Activity 6.11, *continued*

12. Watch the samples and pull the strips out when the solvent front is close to the top of the strip.
13. Lay the samples out on a paper towel to dry.
14. Draw each chromatogram in your lab book, labeling each spot.
15. Observe the spots under short-wave and long-wave ultraviolet radiation. Record your observations.
16. Compare the chromatograms of the different samples. Calculate R_f values.

You can quantitatively describe a chromatogram by calculating **retention factor (R_f)** for each separated component. R_f is simply the distance from the original spot to the center of the separated component of the dye (spot) divided by the distance from the original spot to the solvent front. See Figure 6.15. It may help you to draw a pencil line 1 or 2 cm below the top of the chromatography strip and remove the strip when the solvent front reaches that point.

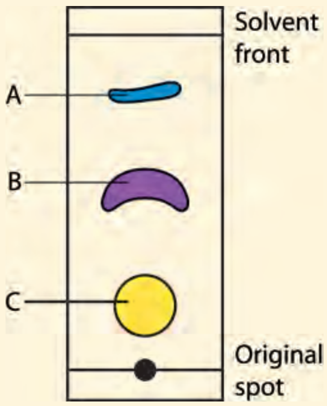


Figure 6.15 Calculation of R_f

$$R_f \text{ for A} = \frac{\text{distance from original spot to A}}{\text{distance from original spot to solvent front}}$$

17. What conclusions can you draw from your results? Note that you can also use the extraction observations in distinguishing samples. Even getting no results—that is, there is no dye extracted—this can be an important characteristic of a sample.

Activity 6.4 Matching Fibers from a Crime Scene

Procedure Notes

Collect a number of black fabric, yarn, or thread samples. Black was chosen because it shows up well microscopically, it is not too common a textile color, and black dyes are generally intense and separate well with TLC. If you can't find samples from which you can extract the dye, add different black inks from pens, markers, etc. Each group in the class can work with the same set of samples. This allows for some competition but also discussion of

Forensic scientists often need to identify and match textile fibers. In this case the partially decomposed body of a woman was found by two hunters in a tangled thicket of briars. Police immediately taped off the area and began a systematic search for evidence. Weathering had erased all footprints. A forensic entomologist determined that the corpse had been in the woods more than a month. A tuft of black fibers was found caught on some thorns about 5 feet off the ground. The woman was wearing black slacks, a white sheer blouse, a black headband, and white cotton socks and underclothing. Her shoes were never found.

Activity 6.4, *continued*

With the help of a forensic anthropologist and dental records, and knowing the approximate time of disappearance, investigators were able to identify the body. Police then questioned a number of people who knew the woman. Her boyfriend immediately became a suspect because of past abuse. Searching his apartment, investigators came up with an old, worn, black, hooded sweatshirt with a few tears and rips; a worn black-and-red checkered work shirt; and quite a few fibers that they vacuumed from his carpeting. Police found little in his car except a few black fibers on the sharp edge of a tire jack in the trunk. The woman's apartment had been cleaned and rented to a new tenant. Her belongings had all been picked up by her parents.

Your task, as a member of the fiber examination unit of the city crime lab, is to analyze the fiber evidence and come to a conclusion as to its probative value. Is there enough evidence to arrest the boyfriend? Will that evidence stand up in court? If so, will you be able, as an expert witness, to justify and defend your conclusions?

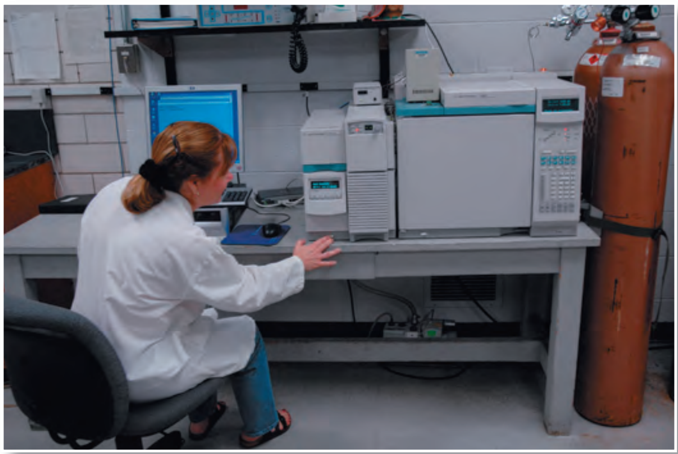
Procedure Notes, *continued*

the value of the evidence. You can ask for a written report, an oral presentation, or both. Either way, the evidence should be described with the source noted. An analytical protocol should be developed based on a limited sample size, using only those tests that are predicted to give the most unequivocal results, starting with the nondestructive analyses, such as ultraviolet observation. As with almost all forensic analysis of trace evidence, the objective is to get the most information from the least amount of material.

This problem can be made difficult or easy, depending on the amount of material each group is allowed to work with. In real situations similar to this activity, often only a few fibers are available, yet the same sort of tests can be performed using microtechniques and a stereomicroscope. For example, in the TLC separation of dyes, a single 1-cm length of fiber is poked up the neck of a capillary tube with a wire. The NaOH extracting reagent is drawn up by capillary action to immerse the fiber. The tube is then sealed off on both ends in a burner flame and incubated in a boiling water bath for 15 minutes. One end of the capillary is then scored with a file and snapped off. The solution can be spotted directly onto the TLC strip. The success of this process, of course, depends on the dye and type

Other Tests for Fiber Evidence

In a modern crime laboratory, there are additional, more sophisticated tools to analyze fibers as trace evidence. Fourier Transform Infrared analysis (FTIR) is a very powerful way to identify fiber type. FTIR is based on the selective absorption of wavelengths of light by particular functional groups in a polymer. This test is nondestructive, and investigators can perform it on single fibers. Optical microscopy is still the most important method of fiber identification and matching, using polarizing light and comparison microscopes. Pyrolysis gas chromatography–mass spectrometry (PGC-MS) burns a sample under controlled conditions and separates and analyzes each combustion product. Forensic scientists can match the resulting chromatogram and product analysis to known standards. They can use this method for short lengths of single fibers, but it is destructive.



Scientist at a PGC-MS

Procedure Notes, Activity 6.4, *continued*

of material. Black wool, nylon, and polyester will probably yield the best results.

SAMPLES

- **A.** slacks of the deceased, known origin
- **B.** headband of victim, known origin
- **C.** tuft of black fibers from thicket, unknown or questioned origin
- **D.** fabric from suspect's black sweatshirt, known origin
- **E.** fabric from suspect's red-and-black shirt, known origin
- **F.** accumulation of fibers vacuumed from suspect's apartment, unknown
- **G.** fibers from suspect's car trunk, unknown

A. Can match some of the black fibers from F. Their presence is logical if the suspect ever entertained his girlfriend in his apartment. They do not match C.

B. No other matches.

C. Should match those from D, sweatshirt that the suspect was wearing when carrying the body into the woods. It got caught, shoulder-high (5 ft off the ground), on some thorns. This puts the suspect at the site where the body was found.

D. Matches C. You also can include some of these fibers in F, which, logically, need not be analyzed.

E. No other matches.

F. Includes fibers from A and D, as well as some white cotton fibers and other material not germane to the case. Analysis on this sample need not be performed because it has no probative value.

G. Should match those from A, the slacks of the victim. This is very strong evidence: The victim was in the trunk of the suspect's car. No other explanation is apparent other than that he put her body in the trunk of his car to transport it into the woods.

IN THEIR JEANS

By Philip Cohen, San Francisco

The FBI has found an ingenious way to catch crooks—by looking at their jeans. Scientists from the bureau reported at last week's meeting of the American Academy of Forensic Sciences in San Francisco that every pair of blue jeans has a unique wear pattern. The FBI has already used this "bar code" to place a suspect at the scene of a crime.

Richard Vorder Bruegge, a forensic scientist at the FBI laboratory in Washington, D.C., and his colleagues developed the technique while helping to identify suspects who were robbing banks and setting off bombs in Spokane, Washington. In April 1996 one of the gang was caught on film. He was wearing a mask, but part of his trousers was visible.

When the photograph was enlarged, Vorder Bruegge noticed light and dark lines running across the seam of the man's jeans. His team found that the pattern originated from slight imperfections introduced when the trousers were made. Workers sew the seams by pushing the fabric through a machine, and the irregularity of that motion stretches and bunches the fabric. The dyed layer of cotton in the raised portion is worn away, creating white bands.

The patches are more striking on jeans than other types of trousers because they are often allowed to become extremely worn. "People just keep wearing them," says Vorder Bruegge.

The FBI analyzed the jeans of suspects in the Spokane case. One pair had a pattern with more than two dozen features that matched the jeans Vorder Bruegge's team photographed. At the trial, the defense called in a used jeans exporter as an expert witness who claimed the patterns were common to all jeans. He showed the court 34 similar pairs, but in each case the FBI could distinguish them from the accused's. The suspect was convicted.

—*from* New Scientist, *February 21, 1998*

Fiber Transfer and Persistence

Fiber can be used as trace evidence because of fiber transfer—no transfer, no evidence. In the case described in Activity 6.3, a transfer was clearly evident, yet it was not so clear what was transferred, what it was transferred to, and how that transfer occurred.

How easily are fibers transferred in a violent crime, say, from a blanket used to wrap a body to the victim's clothing? What influences transferability? How long do transferred fibers remain on the victim? Fibers that are transferred but lost before evidence is collected have no value. Make a list of things that could affect transfer of fibers and also whether and how long they might stay at their new site. Note that garments have labels describing the composition of the fabric. A label that reads "50% cotton and 50% polyester" means that half the material, by weight, is cotton (see Activity 6.3, page 139). These proportions may not be obvious under the stereomicroscope because of differences in density, compaction, twist, and the like. Would you expect fiber transfer to correspond to the makeup of a blended fabric? Explain.

O. J. Simpson's Civil Trial

In the celebrated O. J. Simpson murder trial in June 1995, certain compelling fiber evidence was ruled inadmissible by Judge Ito because the prosecution had not given the defense a complete report by the FBI's hair and fiber expert, Doug Deedrick. However, the following evidence was introduced in the civil case against Simpson in November 1996:

1. Fibers from the blue knit ski cap allegedly worn by Simpson on the night of the murders of his wife Nicole and Ronald Goldman were found to be similar to those on Goldman's shirt.
2. Blue-black cotton fibers from Goldman's shirt, the knit cap, socks found in Simpson's bedroom, and the infamous gloves were consistent with a common origin, which was suggested to be a blue-black sweatshirt that was allegedly worn by Simpson on the night of the crime, but was never found.
3. Rose-beige fibers stuck to the ski cap found at the crime scene and on the bloody glove found behind Simpson's house were similar to the carpet fibers in Simpson's white Ford Bronco. This carpeting was used by Ford for only two years, in 72,000 vehicles.

The defense countered this evidence by stating that fiber comparison is not an "exact science" like fingerprints, and by suggesting contamination and transference of fibers.

Procedure Notes, Activity 6.4, *continued*

Note that there is a cross-transfer of fibers from victim to vehicle, from suspect to crime scene. A defense attorney can argue that the black fiber found in the car trunk could have come from any of that particular type of fabric that had black in it. Have the class explore probabilities here: What sort of textile would innocently be in contact with a car jack? A blanket seems most likely. In an investigation such as this, samples of black fiber from blankets would be obtained and analyzed to compare with the evidence. Statistics could then be generated. The same reasoning would apply to the tuft of black fibers found in the woods: Anyone wearing or carrying something black could have left it there anytime before the body was dumped.

6.1: Wayne Williams Case

CASE STUDY

Read about the Wayne Williams case. Prepare a one-page summary. Suggested sources include:

www.crimelibrary.com/serial/atlanta

www.carpenoctem.tv/killers/williams.html

Wayne Williams

6.2: Amanda Davies Case

CASE STUDY

A woman was found knifed to death under a bridge. She was wearing a blue-and-gray acrylic sweater. There were no fingerprints, no witnesses, and no weapon. She had recently returned from a vacation and had some photographs of herself in some provocative poses with strange people. On the night she died, she told her parents she was going to see her boyfriend and show him the pictures to "rekindle his interest."

After the body was found, her boyfriend was interviewed. He said that he had seen her and that she had been in his truck, but that he had dropped her off "downtown." After obtaining a search warrant, investigators searched his truck and found a yellow polyester blanket.

When the crime laboratory examined the sweater, they found about 40 yellow polyester fibers similar to those from the blanket. Likewise, examination of the blanket revealed more than 30 blue and gray acrylic fibers that matched the sweater. No attempts were made to determine how readily the sweater or blanket shed fibers or how well foreign fibers adhered to these textiles. It would not have been proper to put the actual articles in contact with each other, but the defense argued that similar garments could have been used to investigate the "sheddability" of the garments.

At the trial the prosecutor tried to establish that there was a "primary transfer" between the blanket and the sweater and that the large number of fibers mutually transferred showed that the transfer had been recent. In other words, the prosecutor argued that the blanket had been in direct contact with the victim's sweater right before she died. Because the blanket was found in the truck, this implied that she had been in the truck shortly before she died. This was virtually the only physical evidence available during the case. The other circumstances, such as those shown in the photographs, were used to establish a motive.

The defense argued that the number of fibers transferred may or may not indicate a primary transfer. If these garments shed easily, the victim could have deposited fibers from her sweater into the suspect's truck on a previous occasion. Because the accused kept the blanket in the truck at all times, sometimes in the front seat, the defense argued that the sweater could have picked up blanket fibers from the truck seat; in other words, the truck seat could have acted as an intermediary for a secondary transfer of fibers. Because the crime lab had not done any testing to determine how easily these garments shed, the prosecutor could not know if the 30 to 40 fibers was a "large number."

Faced with these arguments, the jury returned a verdict of "not guilty."

The Amanda Davies case illustrates a lack of preparedness by the prosecution where defense arguments were not anticipated. Had they been, primary and secondary transfer tests could have been devised and carried out.

Testing Fiber Transfer and Persistence

Laboratory Activity 6.12

A case like the previous one highlights the difficulties investigators have in interpreting the presence of foreign fibers, even when there seems to have been a two-way transfer. The Davies case also shows how important information on fiber transfer and persistence may be in a criminal case. This activity will give you practice in testing fiber transfer and persistence.

Materials

For each lab group:

- different fabrics
- UV lamp
- magnifying glass
- graph paper

Procedure

Do not write in your textbook. Take notes in your science notebook.

Design and perform an experiment to determine the transfer and persistence of different types of fibers.

Select different pairs of fabric samples that contrast in some way; for example, fluorescent and nonfluorescent material or white and dark fabric. Test some of the variables you listed on page 158, measuring the number of fibers transferred for each unit area of contact. Remember to look at cross-transfer. Periodically count fibers left after different activities. If possible, construct a graph of fiber persistence versus time of wear. Comment on the number of fibers transferred, considering the defense's argument in the case described above. Will the number of fibers transferred convince a jury of primary transfer? Of secondary transfer?

Variables affecting transfer might include areas of contact; the amount of pressure exerted; any friction or side-to-side movement; the number of passes or contacts; what kind of clothing the donor and the recipient were wearing; the fiber type, length, and texture; and the history of the garment. Fiber persistence would be affected, most importantly, not only by the time of wear but also by the type of movement, whether anything is covering the fabric, type of activity, and weather conditions. Persistence of fibers has been found to decrease exponentially with time of wear.

Answers

1. A filament is a single strand of material; a fiber is many filaments twisted together; fabric is many fibers woven to make a textile.
2. They are natural. Rayon and acetate are two examples.
3. One out of ten: Not very favorable for conviction, but perhaps there is more circumstantial evidence.
4. Trap raccoons for a week or so. Paint their tails orange and set them free. Then take the ratio of the number of raccoons originally trapped to the number or orange-tailed raccoons trapped or seen subsequently over a longer period of time, and multiply by the total number of raccoons trapped or seen later. This will give an estimate of the total number of raccoons.
5. the smallest single unit of a polymer
6. amino acids, amino acids, glucose
7. nylon, polyester, spandex, acrylic
8. No. Textile fabrics are mass-produced. If something unique is associated with the textile, such as a particular fluorescence, then individualization may be possible.
9. Yes, a sample can be individualized when a piece of fabric torn from a garment can be matched directly to the tear pattern, like a piece in a jigsaw puzzle. This is termed a physical match. See Chapter 2, "Types of Evidence." Another way you can individualize a particular fabric sample would be if there is blood, paint, or something peculiar adsorbed to the garment.

Checkpoint Questions

Answer the following questions. Keep the answers in your notebook, to be turned in to your teacher at the end of the unit.

1. What are the different characteristics of a fiber, a filament, and fabric?
2. Are inorganic fibers natural or synthetic? Name two.
3. A gray cotton fiber was found on the red sweater of a victim. A gray cotton fiber was taken from a suspect's sweatshirt. After testing 280 gray sweatshirts, the lab found the fiber matched 28 of them. By chance alone, what is the probability that the crime scene fiber and the one from the suspect matched?
4. Design a sampling procedure to determine the number of raccoons in a particular habitat such as a local township or county.
5. What is the monomer of a polymer?
6. What is the monomer in wool? In silk? In cotton?
7. List four synthetic fibers.
8. Can a fiber be individualized to a particular textile fabric? Why or how?
9. Can a piece of fabric be individualized to a particular garment? Why or how?

10. Many fibers look very similar under the microscope, but there are major differences between synthetic and natural fibers. What would you look for in determining whether a particular fiber was synthetic or natural?

11. Develop flow charts for matching a questioned fabric to a known sample, for identification of a questioned fabric, and for the identification and matching of a single fiber 3 cm long.

12. If an unknown fiber is suspended in a solution with a density of 1.30, what might the fiber be?

13. If the same fiber in question 12 is found to have a refractive index of 1.53, what might it be?

14. Explain how an optical brightener may change how light reacts with fibers.

15. If a fiber disappears in castor oil, what might it be?

16. What is the most common natural fiber used in textiles? The most common synthetic fiber?

17. What is trace evidence? What is the common basis for analyzing trace evidence, that is, what are the goals?

18. During a trial, what are the primary concerns in analyzing and using extremely small bits of trace evidence?

Answers, *continued*

10. medullary index; also cuticle and diameter

11. Answers will vary.

12. wool, acetate, Dynel

13. Dynel

14. It may fluoresce.

15. acetate, with a refractive index of 1.48

16. cotton, polyester

17. Trace evidence is usually small objects. Because it is usually class evidence, the goal would be to find associations and links to everything involved with a crime. These connections can ultimately improve the strength of other circumstantial evidence.

18. Contamination, loss, and uncertainty in the analytical results are all issues because investigators can't run replicate tests or perform a full range of tests.

A new book on the JonBenet Ramsey murder reveals, for the first time, what went on behind closed doors in June of 1998 when police presented their mammoth investigation to prosecutors.

"Deliberately, it seemed, the police had organized the information to negate an intruder theory," author Lawrence Schiller wrote in his book, *Perfect Murder, Perfect Town*, detailing the police case against the slain six-year-old's parents.

Among the evidence Schiller claims police presented at their June 1–2 case overview:

Four fibers found on the duct tape covering JonBenet's mouth "were consistent with" the jacket Patsy Ramsey had worn to a party Christmas night—and also had on the next morning. Forensic expert Henry Lee and attorney Barry Scheck pointed out, though, that "fibers are fibers" and can't be matched like fingerprints.

What do you think?

Answers, *continued*

19. Repeat the test to verify the difference. Analyze the questioned and known samples at the same time to minimize experimental and observational uncertainties. If there is still a discrepancy, then there is no match, and no association among suspect, victim, and location based on the fiber evidence.

20. Refer to the pie chart on page 138. Polyester makes up 77 percent of the approximate 8 billion pounds of human-made fiber produced in the United States; acrylics account for 8 percent. Thus the probability of finding a random acrylic fiber is one-ninth that of finding an unassociated polyester fiber. Investigators can never say with any certainty that a fiber originated from a particular textile to the exclusion of all others; however, any factor that decreases the probability of an accidental association increases the significance of the findings.

Assessment Strategy

2. Have each student bring to class fibers from a blanket at his or her home. Compare all the samples using as few tests as possible (microscopic observation may be sufficient). You could expand the population base by having your students ask a friend for a sample from a blanket at his or her house.

19. **Out of seven analytical tests performed to match a questioned fiber to a known, you find one discrepancy; for example, the cross section is triangular rather than round. What do you do?**

20. **All else being equal, which fiber has more probative value, a polyester or an acrylic fiber? Why?**

Additional Projects

1. Rope is often used to tie victims up; less often, it is used to strangle a person. What is rope? What is it made of? Can it be classified? Individualized? How can it be used as evidence? A good starting reference is Chapter 7, "Fabric and Cordage," of *Forensic Fiber Exam Guidelines* (U.S. Department of Justice, FBI (1999); available online at www.fbi.gov/hq/lab/fsc/backissu/april1999/houckch7.htm).
2. How common are particular fibers? Here is an experiment in probabilities. Select enough fibers for different analyses from a blanket you have at home. If your fibers were found on both the victim and a suspect, what are the odds that this is a coincidence and there is not an association?
3. Expand Lab Activity 6.12 on primary transfer of fibers (p. 161) to include secondary transfers.
4. Prepare a half- to full-page summary of selected cases, noting the role fiber evidence played. For example:
   - **a.** Dr. Jeffrey MacDonald (the "Fatal Vision" murders; refer to Evans and Fisher on the next page)
   - **b.** Roger Payne (refer to Evans)
   - **c.** The Enrique Camarena case (refer to Saferstein)

References

Books and Articles

Doyle, M. P., and W. S. Mungall. Experiment 38 in *Experimental Organic Chemistry*. New York: John Wiley & Sons, 1980.

Evans, C, *The Casebook of Forensic Detection*. New York: John Wiley, 1996.

Fisher, D. Chapter 4 in *Hard Evidence*. New York: Dell, 1995.

"Industries, Textile." *The New Encyclopedia Britannica*, vol. 21, Macropedia (15th ed.). Chicago: Encyclopedia Britannica, 1993.

Manufactured Fiber Fact Book. Washington, D.C.: Education Department, American Fiber Manufacturers Association, 1988.

Meloan, C. E. "Fibers, Natural and Synthetic," *Chemistry*, vol. 51, April 1978.

Wood, C. G. "Natural Dyes," *ChemMatters*, December 1986, pp. 4–7.

Films and Videos

"The Common Thread: Multiple Forensics," video, Films for the Humanities & Sciences, P.O. Box 2053, Princeton, NJ 08543-2053; or available online at www.films.com.

Websites

www.fibersource.com/FiberWorld/fiber.html; lots of information on fibers

www.pslc.ws/macrog.htm; excellent overview on polymers and their uses

www.fbi.gov/hq/lab/fsc/backissu/july2000/deedric1.htm; forensic fiber evidence