Forensic Analysis of Paper

EQUIPMENT AND MATERIALS

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You'll need the following items to complete this lab session. (The standard kit for this book, available from *http://www.thehomescientist.com*, includes the items listed in the first group.)

MATERIALS FROM KIT

- Goggles
- Coverslips
- Forceps
- Herzberg's stain
- Jenk's stain
- Magnifier
- Pipette
- Slides, flat

MATERIALS YOU PROVIDE

- Gloves
- Camera with microscope adapter (optional)
- Desk lamp or other incident light source
- Microscope
- Paper towels
- Scissors
- Ultraviolet light source (optional)
- Watch or clock with second hand
- Specimens, known and questioned paper

WARNING

Herzberg's stain and Jenk's stain are toxic and corrosive. Wear splash goggles, gloves, and protective clothing. Read the MSDS for each chemical you use and follow the recommended safety precautions.

http://www.thehomescientist.com/kits/FK01/fk01-main.html

BACKGROUND

Although it is made up primarily of cellulose fibers, paper is not the simple material that most people think it is. The basic material may be new or reprocessed fibers of cotton, linen, straw, or another natural fiber, or it may be chemically pulped or mechanically pulped wood fibers. Paper may be *laid* (produced on a patterned, directional screen; now used almost exclusively for handmade and art papers) or *wove* (produced on a fine, nondirectional screen; now used for 99% of all paper production). Various coatings, binders, and fillers are used in different papers, as are visible dyes (to change the tint) and ultraviolet brighteners. Better quality papers are often watermarked, which is a process that introduces a mark that is invisible by reflected light but visible by transmitted light.

> Most people are aware that high-quality papers often contain high percentages of cotton or linen (so-called "rag") fibers. In fact, high-quality paper is often marketed using such terms as "100% cotton bond." But the original source of the fibers actually has little bearing on the quality of paper produced from them. How those fibers, regardless of their source, are extracted and treated determines the quality of the paper.
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> Most paper is produced using primarily or exclusively wood pulp, which is the least expensive source of cellulose fibers. Wood pulped mechanically, which essentially means grinding it up, gives high yields—one kilogram of wood yields about one kilogram of paper—but the quality of the paper is low because the lignin content is high. Such mechanical pulp papers are used primarily for newsprint, paperback books, and similar purposes, where paper longevity is not an issue. Wood pulped chemically—by treatment with concentrated sulfite or soda solutions—has most of the lignin removed. The downside of that is lower yields—as little as 50%—and accordingly higher costs, but the upside is that paper produced from chemical pulp is of excellent quality, as good as that produced from rag.

It is sometimes important forensically to determine if two paper specimens are closely similar or have distinguishable differences. For example, a question may arise as to whether a page was added to a contract or will. If the paper of the questioned page appears identical to the accepted pages under forensic examination, the questioned page may or may not be a part of the original document, because the person who added the questioned page may simply have used paper from the same stock as was used for the original document. But if the questioned page uses different paper from the rest of the document, it may reasonably be assumed that the questioned page was added later and was not a part of the original document.

A thorough forensic examination of a questioned paper specimen is typically done in four phases:

Visual examination

A preliminary visual examination of the gross physical characteristics of the paper specimen—color, weight, texture, transparency, watermarking, and so on—is occasionally sufficient to establish that the questioned specimen differs from the accepted specimen.

Microscopic examination

If the gross visual examination is inconclusive, the next step is to examine the questioned paper specimen microscopically by reflected and transmitted light. Two specimens that appear identical on gross visual examination may appear very different at 40X to 100X magnification. Differences in the type, length, or structure of the fibers, the size and shape of sizing or coating particles, or the dye absorption pattern of a colored paper are often sufficient to establish that two specimens differ.

Differential staining

It may be difficult or impossible to discriminate among the types of fibers present in the paper specimen using only visual and microscopic examination. Using *differential stains*—those that dye different types of fibers different colors—allows the examiner to determine which types of fibers are present in the specimen and in what relative numbers. We'll use two differential paper stains in this lab session, called *Herzberg's stain* and *Jenk's stain*.

Instrumental analysis

If all other methods are inconclusive—and if the matter is sufficiently important to justify it—instrumental analysis may be used to compare the questioned paper specimen against an accepted specimen. The most common techniques used for this purpose are electron microscopy and neutron-activation analysis, both used to compare the minor constituents of the paper specimen, such as sizing and coating particles.

In this lab session, we'll use the first three methods to compare various paper samples and discriminate one from another.

For your known and questioned paper specimens, obtain examples of as many similar types of white paper as possible. That is, try to obtain paper specimens that have no gross differences that would make it easy to discriminate between them at first glance. Specimens about 5 cm square are ideal, because they're large enough to allow visual comparison but not so large as to include gross differentiators such as watermarks. Try to obtain specimens ranging from inexpensive copy paper to high-quality bond paper with differing rag percentages. We got our specimens from sources ranging from junk mail to old letters to old hardback books that were destined for the discard pile. For your questioned specimen, ask a friend or relative to choose one of the sources of your known specimens and produce the questioned specimen from it.

FORMULARY

If you don't have the FK01 Forensic Science Kit, you can purchase Herzberg's stain and Jenk's stain from a law enforcement forensics supply vendor or make them up yourself. Wear gloves and safety goggles while making up and using these stains. Both are hazardous, particularly Herzberg's stain, which is extremely corrosive.

It's particularly important to use accurate weights and measures in making up Herzberg's and Jenk's stains, because small differences in the ratios between the components can cause major differences in staining behavior. For that reason, specimens stained with one batch of one of these stains cannot be compared to specimens stained with a different batch.

Herzberg's stain and Jenk's stain are reasonably stable in tightly capped containers stored in a cool, dark place. We've used samples of both that were five or more years old, and they worked properly. Note, however, that paper tests done years apart with the same batch of stain cannot be compared because the stains do gradually age and change properties.

Herzberg's stain Dissolve solid zinc chloride in 25 mL of distilled or deionized water to produce a saturated solution. (Caution: Corrosive!) Zinc chloride is hygroscopic and available in several hydration forms. It is also extremely soluble in water. Continue adding solid zinc chloride to the solution until undissolved crystals remain.

Separately dissolve 5.25 g of potassium iodide in about 5 mL of distilled or deionized water. Add 0.25 g of iodine crystals to the iodide solution and swirl until the iodine dissolves. Make up the iodide/iodine solution to 12.5 mL with distilled or deionized water.

Add 25 mL of the saturated zinc chloride solution to the 12.5 mL of the iodide/iodine solution with swirling to mix the solutions. Allow the mixed solutions to sit undisturbed overnight and then carefully decant off the red liquid, leaving any sediment present behind. Label the storage container Herzberg's stain and date it.

Jenk's stain Dissolve magnesium chloride in 50 mL of distilled or deionized water to produce a saturated solution. Continue adding solid magnesium chloride to the solution until undissolved crystals remain.

Separately dissolve 2 g of potassium iodide in about 4 mL of distilled or deionized water. Add 1.15 g of iodine crystals to the iodide solution and swirl until the iodine dissolves. Make up the iodide/iodine solution to 20 mL with distilled or deionized water.

While swirling to mix the solutions, add 2.5 mL of the iodide/iodine solution to 50 mL of the saturated magnesium chloride solution. Allow the mixed solutions to sit undisturbed overnight and then carefully decant off the liquid, leaving any sediment present behind. Label the storage container Jenk's stain and date it.

EXAMINE PAPER SPECIMENS VISUALLY

1. Examine the questioned specimen visually by transmitted light and by reflected flat and oblique light, both with the naked eye and using your magnifier. Note all visible characteristics, including color, texture, pattern, or weave, finish, and so on. If you have an ultraviolet light source, repeat your observations by ultraviolet light. Record your observations in your lab notebook.
2. Repeat step 1 for each of your known specimens, and attempt to determine if the questioned specimen is consistent visually with one or more of the known specimens.

EXAMINE PAPER SPECIMENS MICROSCOPICALLY

1. Cut a small sliver of the questioned specimen, place it on a slide and cover it with a coverslip. Do not use any water or other mountant; we'll examine the specimen first using a dry mount.
2. Examine the questioned specimen at 40X and 100X by incident (reflected) light. If solid particles are visible at 100X, they are probably coating or filler material. Increase magnification to 400X and try to determine the size, shape, color, and other characteristics of the solid particles. Record your observations in your lab notebook. If you are equipped to do so, shoot images of the specimen.
3. Turn off the incident light source and add one drop of water under the cover slip (or more, if necessary to wet the specimen thoroughly).
4. Examine the questioned specimen again at 40X and 100X by transmitted light. Record your observations in your lab notebook. If you are equipped to do so, shoot images of the specimen.
5. Repeat steps 1 through 4 for each of your known specimens, and attempt to determine whether the questioned specimen is consistent microscopically with one or more of the known specimens.

EXAMINE PAPER SPECIMENS BY DIFFERENTIAL STAINING

1. If you have not already done so, put on your splash goggles, gloves, and protective clothing. (Jenk's stain and particularly Herzberg's stain are hazardous; although you will use them dropwise, you still need to protect your eyes, hands, and clothing.)
2. Label one microscope slide "Q-H" for your questioned specimen that is to be stained with Herzberg's stain, and a second slide "Q-J" for the questioned specimen to be stained with Jenk's stain.
3. Position a small sliver of your questioned specimen centered on each of the two slides.
4. Note the time, and put one drop of Herzberg's stain on the "Q-H" slide and one drop of Jenk's stain on the "Q-J" slide. (Use more stain if necessary to thoroughly wet each specimen.)
5. Allow the stains to work for one minute by the clock.
6. Use the corner of a paper towel to wick up the excess stain from each slide.

7. Using the disposable pipette, apply several drops of distilled water to each specimen to rinse out excess stain. Use a clean corner of a paper towel to wick off the rinse water. Repeat as necessary until all excess dye has been rinsed out.

8. Place a coverslip over the specimen and observe it at 40X magnification. Note your observations in your lab notebook. If you are equipped to do so, shoot images of the specimen.

9. Repeat steps 2 through 8 for each of your known specimens, and attempt to match the questioned specimen to one or more of the known specimens. Figure X-3-1 shows a typical differentially stained specimen at 40X. This specimen, stained with Jenk's stain, reveals a combination of chemical wood pulp and chemical straw pulp fibers.

The effect of these two stains depends on their exact composition as well as the particular types of fibers being stained.

Herzberg's stain dyes mechanical (ground) wood pulp—as well as other wood-like fibers that contain significant lignin, including jute, flax, and hemp—a yellow shade that may vary from lemon yellow to egg-yolk yellow. Chemical (sulfite or soda) straw or wood pulp and similar wood-like fibers from which most or all of the lignin has been removed are dyed a blue shade, from sky blue to navy blue. Cotton and linen fibers (from high-rag content bond papers) are dyed a wine-red color.

Jenk's stain dyes mechanical wood pulp fibers yellow, chemical wood pulp fibers anything from colorless to deep red, chemical straw pulp fibers blue to violet, and cotton or linen fibers brown.

Figure X-3-1: *A differentially stained paper specimen at 40X*

REVIEW QUESTIONS

Q1: Which, if any, of your tests allowed you to determine that a possible match existed between your questioned specimen and one or more of your known specimens? How?

Q2: You have treated a paper specimen with Herzberg's stain and Jenk's stain and found that both stains dye all of the fibers in the specimen a yellow color. What do you conclude about the fiber type and general quality of the specimen?

Q3: You have a paper specimen thought to be a high-quality bond paper with 50% rag content. What results would you expect if you treated this paper with Herzberg's stain and Jenk's stain?