

## Questioned Documents Lab Document Analysis Parts 1-5

### Introduction

Crime scene investigators use many kinds of evidence when trying to solve a crime. Often the evidence includes the analysis of written or printed documents. This may include analysis of the paper, the writing, the inks, or other properties of the documents. Analyzing documents is an important aspect of forensic science and scientific analysis of documents often used as evidence in a court of law.

### Concepts

- Forensic science
- Microscopic observation
- Chromatography
- Acid-base indicators

### Background

The analysis of documents from a crime scene can take many different paths and can employ many different analysis techniques. The document experts of a crime lab will make use of chemistry, physics, microscopy, chromatography, photography, handwriting analysis, and various other specialized techniques. Most of the work in the crime lab emphasizes comparison of materials and writing with known standards. This often allows the crime scene investigator to trace the materials back to a certain location or to a certain manufacturer. Documents at crime scenes can include wills, checks, handwritten notes, typed materials, printed materials, photographic materials, and a variety of other paper documents. In addition to the actual documents, printing machines as well as an individual's handwriting can also be analyzed. Analysis of tampered documents is often important in criminal investigations.

Paper, in its simplest terms, is a very thin layer of bonded fibers. Paper has been made from many different fibers throughout history, but today most paper fiber is cotton, linen, wood or some combination of these. Because the formula and mixture of fibers for all papers is slightly different, various papers have distinctive looks and structures. The structure of a paper can help to identify its source or authenticity. Some manufacturers place a translucent mark, called a watermark, on the paper. These are specific to a certain manufacturer and are changed periodically. Watermarks can reveal information about the date and origin of a document.

What is written on paper is often more revealing than the paper itself. Secret messages may be written in code or with "invisible" chemicals that can be revealed later. Inks, pens, and pencils all have distinctive "trails" that are left behind on the paper. All of these things are analyzed carefully by the document specialists on a crime scene investigation team.

One common laboratory technique used to analyze the makeup of materials, such as inks, lipsticks and other markings on paper is called *paper chromatography*. There are many different types of chromatography but most work on the concept of absorbance. Two important components of any chromatography system are the absorbent and the eluent. A good absorbent is usually a solid material that will attract and absorb the materials to be separated. Paper, silica gel, or alumina are all very good absorbents. The eluent is the solvent that carries the materials to be separated through the absorbent.

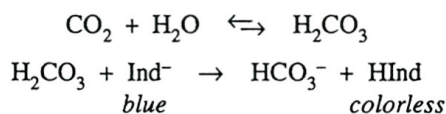
Chromatography works on the principle that the compounds to be separated are slightly soluble in the eluent and will spend some of the time in the eluent (or solvent) and some of the time on the absorbent. When the components of a mixture have different solubilities in the eluent, they can then be separated from one another. The polarity of the molecules to be separated and the polarity of the eluent are very important. Changing the polarity of the eluent will only slightly change the solubility of the molecules but will greatly change the degree to which they are held by the absorbent. This affinity for the eluent versus the absorbent is what separates the molecules in chromatography.

Paper chromatography is often used as a simple separation technique. In paper chromatography, the absorbent is the paper itself, while the eluent can be any number of solvents. The polarity of the eluent is very important in paper chromatography since

a small change in polarity will dramatically increase or decrease the solubility of some organic molecules. Many times, a mixture of a nonpolar solvent and a polar solvent is used to achieve an optimum polarity. When placed in a chromatography chamber as shown in Figure 1, the eluent moves up the paper strip, being drawn by capillary action. The organic molecules, which were “spotted” onto the paper chromatography strip, separate as they are carried with the eluent up the strip at different rates. Those molecules that have a polarity closest to the polarity of the eluent will be the most soluble, and will move up the strip the fastest.

The choice of the eluent or solvent is the most difficult task. Choosing the right polarity is critical because this determines the level of separation that will be achieved. Common solvents used in chromatography, in order of increasing polarity, are: petroleum ether or hexanes, cyclohexane, toluene, chloroform, ethyl ether, acetone, ethanol, methanol, and water. Sometimes mixtures of solvents are used to achieve the desired degree of polarity. Many inks are actually mixtures made up of several basic pigments. Each of these pigments has a different molecular structure and, usually, a different polarity. Many of these pigments can be separated using paper chromatography.

Disappearing ink used in this lab is a mixture of thymolphthalein indicator, ethyl alcohol, sodium hydroxide solution, and water at pH 11. When the blue ink is applied to paper, the blue color quickly vanishes. The disappearance of the blue ink color in air is due to the effect of carbon dioxide ( $\text{CO}_2$ ), which reacts with moisture in the air to form carbonic acid ( $\text{H}_2\text{CO}_3$ )—the pH change is enough to push the basic form of the indicator ( $\text{Ind}^-$ ) back to its colorless acidic form ( $\text{HInd}$ ).



Thymolphthalein is a weak organic acid that behaves as an acid–base indicator in the pH range 9.3 (colorless) to 10.5 (blue). It exists in two different forms—an acid form  $\text{HInd}$ , which is colorless, and a corresponding conjugate base form  $\text{Ind}^-$ , that is blue. The color transition range for an acid–base indicator depends on the strength of the weak acid  $\text{HInd}$ . The color change is due to the changing proportion of the indicator molecules in the acid or base form.

## Materials

Sodium hydroxide solution,  $\text{NaOH}$ , 0.1 M, 3–5 mL  
Thymolphthalein indicator solution, 3–5 mL  
Beaker, 50- or 100-mL  
Chromatography chambers, 6  
Chromatography paper, 6 strips  
Cotton-tipped applicator  
Filter paper  
Microscope (4X or stereoscope if available)

Paper, notebook, several sheets cut into fourths  
Paper samples, 5 (A–E)  
Pencils, 3 (#1, #2, #3)  
Pens, 6  
Spray bottle  
Tape, 6 strips ( $\frac{1}{2}$ " )  
UV lamp  
Wood splints, 6

## Safety Precautions

*Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Never look directly into a UV light source as eye damage can result. Wash hands with soap and water upon completion of laboratory work.*

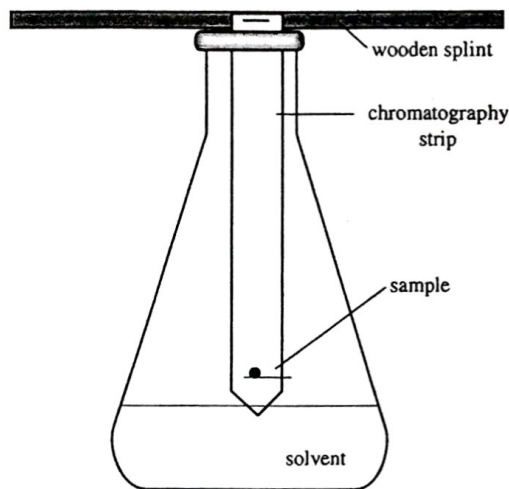


Figure 1.



## Procedure

### Part I. Paper Examination

1. Obtain paper samples labeled #A–#E.
2. Observe each paper type and record your observations of each paper on the Document Analysis Worksheet, Part I. Describe the color. Is it white, yellow-white, gray-white, blue-white, etc. Is it dull/shiny? Thick/thin? Smooth/rough?
3. Hold the paper up to a light or window. Does it have a watermark? What does the watermark look like? Are any of the marks the same?
4. Observe the papers under an ultraviolet light. (Use longwave ultraviolet light for best results). Shine the light on each paper and observe if the paper fluoresces or glows. What color is each paper under UV light?  
*Warning:* Do NOT look directly at a UV light; it may cause eye damage.
5. Try to rank the papers by weight or thickness. Rank them from thinnest (#1) to thickest (#5). If a centigram balance (0.01) is available, each paper should be weighed.
6. Use a microscope (on lowest power, or use a stereoscope, if available) to observe the fiber structure of each paper. Rank the fiber structure of each paper. Rank them from the smoothest (#1) to the roughest (#5).
7. Record all observations on the Document Analysis Worksheet.
8. Examine the ransom note provided by your instructor. Which paper type (A–E?) was used for writing the ransom note?

### Part II. Pencils/Erasers

1. Sharpen a #1, #2, and #3 pencil to the same degree.
2. Draw a 2 cm-long mark from each pencil on a sheet of notebook paper side by side so that they are relatively close together. Try to apply the same pressure on the pencil when making the lines. Label the lines 1, 2, and 3, respectively.
3. Examine each line using low power on a microscope. Focus on each line carefully and note its thickness, color intensity, sharpness, etc. Record your observations on Part II of the Document Analysis Worksheet.
4. Have a partner secretly select one of the three pencils and write a short sentence on another small piece of paper. Compare the writing with the three original marks. Examine the sentence writing using a microscope. Can you determine which pencil your partner used to write the sentence?
5. Have a partner make an “X” on a small piece of paper noting which line of the X was drawn first (bottom) and which line was drawn second (top). Use the microscope to determine which line is on top and which is on the bottom. (This technique could be important in trying to determine if one line has been written over the top of another.)
6. Use a pencil to write your name on another small piece of paper. Press firmly as you write. Erase part of a letter. Examine the eraser mark under the microscope. Shine the UV light on the eraser mark. Are the erasures easily detected?
7. Use a pencil to write a sentence on a piece of paper. Have a partner do the same. Erase a word in the sentence. Try to write the erased word again with the same pencil, being as careful to write right over the old writing as possible. (See if you are a good forger!) Swap the written sentences with your partner and use the microscope and UV light to track down the erased word. Were you able to detect the forgeries?
8. Write a summary of your observations about erasing pencil from paper. Do this on the Document Analysis Worksheet, Part II.

### Part III. Handwriting/Forgery

1. Use a ballpoint pen to firmly write your signature onto a small piece of paper. Label this paper “O.”
2. On a second sheet of paper write your signature again. Label this signature “A.”
3. Find another person. Give them paper “O” and instruct them to place a piece of paper over the signature and attempt to trace it. Have them label this paper “B.”
4. Find another person. Give them paper “A” and instruct them to forge the original signature by just looking at it and trying to make it look identical to “A.” Have them label this paper “C.”

5. Give papers "O," "A," "B," and "C" to a fellow crime scene investigator. Using a microscope, can the investigator determine which signatures, A, B, C are forgeries and which is an original like "O"?
6. Answer the question for Part III on the Document Analysis Worksheet.

#### Part IV. Inks/Chromatography

1. From a chromatography sheet, cut six strips, each 13 cm long  $\times$  2 cm wide.
2. Using a *pencil*, lightly draw a line across the width of each strip, 2 cm from one end (see Figure 2a).
3. Cut off the bottom corners of each strip to create a point, as shown in figure 2b. Staple or tape the strip to a wooden splint, as shown in Figure 2c. Repeat for all six strips.

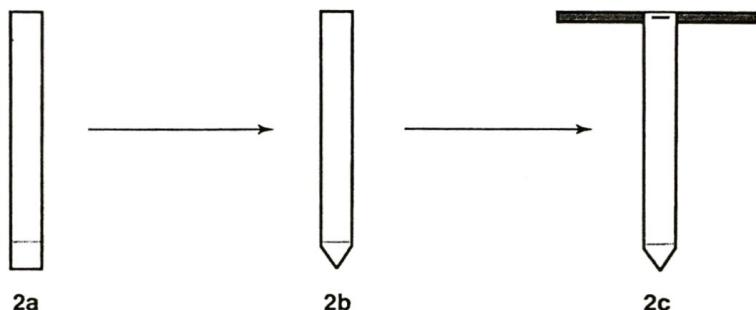


Figure 2.

4. Add 50 mL of water to six chromatography chambers.
5. Use a chromatography pen, place a *small* dot on the center of the drawn line on one chromatography strip. Repeat for each pen on a separate chromatography strip. Using a *pencil*, write the color of the pen on the top of the strip or on the wooden splint.
6. Slowly lower one chromatography strip into the chromatography chamber. The pointed end of the paper strip should just touch the solvent but the sample spot should remain above the solvent (the water). If the sample spot touches the solvent, it will simply dissolve in the solvent.
7. Repeat step 6 for each chromatography strip.
8. The solvent will be drawn up the chromatography strips by capillary action. As it is drawn up, it will carry the pigments in the ink samples up the strips at different rates depending on the characteristics of the individual components in the ink.
9. When the solvent front is within 0.5–1.0 cm of the top of the chromatography strip, stop the run by removing the strip from the flask.
10. It is a good idea to carefully mark the location of each of the pigment spots on the strips and the final solvent fronts, again using a pencil. This is done because some of the color and brightness of each of the spots may be lost over time. During this time, the residual water on the strips may continue to be drawn up the strip slightly by continued capillary action. If you have marked the location of the pigment spots in pencil, this is not a concern.
11. If a UV light is available, shine it on each of the strips in a darkened room.
12. Answer the Analysis Questions for Part IV of the Document Analysis Worksheet.

#### Part V. Secret Messages

1. Use a cotton-tipped applicator to "write" a message with the thymolphthalein "disappearing ink" solution on a large piece of chromatography or filter paper. The color will fade from blue to colorless almost immediately.
2. Allow the secret message to dry and disappear completely. If necessary, blow on the secret message to make the ink dry and disappear faster.
3. Once the ink has disappeared it can be "developed" (made to reappear) by spraying the message with a mist of 0.1 M NaOH solution from a spray bottle. The blue color will reappear almost instantly and will usually last 3–5 minutes before it fades again.
4. Answer the questions for Part V of the Document Analysis Worksheet.

Name: \_\_\_\_\_

# Document Analysis Worksheet

## Part I. Paper Examination

Paper	Color	Thickness	Watermark	Fiber Structure	UV	Other Observations
A						
B						
C						
D						
E						

## Part II. Pencils/Erasers

Pencil Lead	Observations
#1	
#2	
#3	

Pencil Eraser Observations:

## Part III. Handwriting/Forgery

Describe typical clues (especially microscopic evidence) in handwriting that might signal a forgery.



Name: \_\_\_\_\_

# Document Analysis Worksheet (Con't)

## Part IV. Inks/Chromatography

1. Draw representations of each of the strips including the pigment colors and locations. Label each drawing with the pen name and color.
2. Which inks appear to be made up of more than one pigment? Which inks appear to be a single pigment?
3. Do any of the inks appear to use common pigments?
4. Which pen was most likely used to write the ransom note provided by your instructor?

## Part V. Secret Messages

Describe a crime situation where disappearing ink or hidden messages might be useful to a criminal or to a secret agent.